

Reverse Osmosis to Concentrate Ammonium Nitrate in Condensates, from Laboratory Studies to Industrial Scale Design and Operation

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Summary

In the manufacturing of ammonium nitrate, a condensate containing ammonia and ammonium nitrate is obtained. The concentrations of these species are in practically all cases too high for direct discharge into a water recipient. At some plants, some of the dilute ammonium nitrate solution can be spread out on adjacent farmland, as it is a fertilizer, but commonly, nitric acid is added to this stream to convert all the ammonia to ammonium nitrate, and this stream or part of it is concentrated in a separate evaporator, here called the low concentration evaporator. When the existing low concentration evaporator has reached its capacity limit, the manufacturing plant has to spend some capital to increase its condensate treatment capacity. Pre-concentrating the ammonium nitrate condensate with reverse osmosis (RO) is one way to accomplish this.

Extensive laboratory tests were conducted to determine the separation characteristics when using RO to concentrate ammonium nitrate solutions. The test conditions were 0.2-5.2 wt% ammonium nitrate feed at 25-54°C and gage pressures up to 62 bar (900 psi). The ammonium nitrate permeability at 25°C [B(25C)] increased with increasing ammonium nitrate concentration (c in wt%) according to $B(25C) = 1.12 \cdot 10^{-5} \cdot c^{0.27}$ cm/s. The temperature dependence of the ammonium nitrate permeability followed an Arrhenius equation with the activation energy 8500 cal/mol.

In subsequent pilot plant tests, there were problems with the ammonium nitrate concentration analyses, so the obtained performance data were not accurate. However, the pilot plant tests confirmed that the membrane fouling rate was very low when operating on the ammonium nitrate condensate, and convinced the plant personnel that RO can be used to concentrate the ammonium nitrate in the condensate, and obtain a permeate that can be reused within the plant.

At one fertilizer plant, the ammonium nitrate condensate flow rate is about 15 m³/h (67 US gpm) with a typical ammonium nitrate concentration after neutralization of 2 wt%. Using the water and ammonium nitrate permeability data from the laboratory tests, computer simulations predicted that a 3-pass RO system operating at 50°C would produce 11.4 m³/h (50 US gpm) final permeate with less than 50 mg/L ammonium nitrate from this condensate. The ammonium nitrate concentration in the concentrate would then be 8 wt%, which would be sent to the single effect evaporator. Each pass in the RO system would consist of 3 housings arranged in a 2-1 array, with 5 AD8040 elements per housing.

Even though the pay-off time of an RO system would be below 1.5 years, the end-user might be hesitant to make the investment because of the unfamiliarity with such systems. This was overcome in this case by contracting the supervision, maintenance, repair and consumables for the RO system to the seller of the system, GE Water & Process Technologies, for a fixed monthly cost. The RO system has not started up when this paper was written, but will very likely be in operation when this paper is presented.

Introduction

In the manufacturing of ammonium nitrate, a condensate containing ammonia and ammonium nitrate is obtained. The concentrations of these species are in practically all cases too high for direct discharge into a water recipient. At some plants, some of the dilute ammonium nitrate solution can be spread out on adjacent farmland, as it is a fertilizer, but commonly, nitric acid is added to this stream to convert all the ammonia to ammonium nitrate, and this stream or part of it is concentrated in a separate evaporator, here called low concentration evaporator. When the existing low concentration evaporator has reached its capacity limit, the manufacturing plant has to spend some capital to increase its condensate treatment capacity. Pre-concentrating the ammonium nitrate condensate with reverse osmosis (RO) is one way to accomplish this.

RO applications very seldom deal with a single solute dissolved in water with very low fouling tendency. The condensate from the ammonium nitrate evaporator is, however, such a case, which makes it possible to in detail analyze and model the separation process. This paper describes the physical properties of ammonium nitrate solutions that are important for the RO separation process, laboratory and pilot plant test data from using RO to concentrate an about 1 wt% ammonium nitrate solution up to 8 wt%, and some information about a full scale RO plant for this application that presently is being built.

Brief Description of Ammonium Nitrate Manufacturing

Ammonium nitrate is typically produced by letting anhydrous ammonia react with an aqueous solution of about 65 wt% nitric acid. Most of the water in the formed ammonium nitrate solution is removed in an evaporator, where the condensate containing ammonia and ammonium nitrate is generated. The ammonium nitrate concentrate goes to a spray dryer, while the condensate must be treated in some way. The condensate cannot be returned to the same evaporator, because if so, the evaporator would not remove any water from the ammonium nitrate feed solution, which is its sole purpose. Instead, the condensate frequently goes to the low concentration evaporator. The concentrate from this evaporator can be returned to the high efficiency evaporator, while the condensate can be reused within the plant or discharged.

The neutralized condensate from the first evaporator typically contains 1-2 wt% ammonium nitrate. RO can be used to concentrate this stream to about 8 wt% ammonium nitrate, thus decreasing the size of the ammonium nitrate containing stream by 75 percent or more. This could help in increasing the capacity and decrease the cost of the condensate treatment system.

Water and Salt Flux Model

Eqs.(1) and (2) were used to model the water and salt fluxes through the membrane.

$$J_w = A(25C) \cdot TCFA \cdot (\Delta P - \pi_m + \pi_p) \quad (1)$$

$$J_w \cdot c_p = B(25C) \cdot TCFB \cdot (c_m - c_p) \quad (2)$$

where

J_w is the permeate flux, (m/s)

$A(25C)$ is the water permeability at 25°C (also called A-value), (m/(s·Pa) or cm/(s·atm))

TCFA is the temperature correction factor for the water permeability

ΔP is the average hydraulic pressure difference between the feed and permeate sides of the membrane

c is the solute concentration in wt%

$B(25C)$ is the solute permeability (also called B-value) at 25°C, m/s or cm/s

TCFB is the temperature correction factor for the solute permeability

π is the osmotic pressure

the subscripts m and p denote the feed side/membrane interface, and permeate respectively.

The solute concentration at the interface between the membrane and the feed side was calculated with the equations shown in Appendix B.

The solute rejection is defined in Eq.(3)

$$R = 1 - c_p/c_m \quad (3)$$

Eqs.(2) and (3) give

$$\frac{1}{R} = 1 + B(25C) \cdot \frac{TCFB}{J_w} \quad (4)$$

Thus, Eq.(4) predicts that $B(25C)$ is the slope when plotting the inverse of solute rejection versus $TCFB/J_w$. For most combinations of RO and NF membranes and charged solutes, $B(25C)$ increases with the solute concentration to a power in the range 0.1-0.5, which is expressed in Eq.(5)

$$B(25C,c) = B(25C, c_{ref}) \cdot (c/c_{ref})^d \quad (5)$$

where

$B(25C,c)$ is the solute permeability at 25°C and solute concentration c at the membrane/feed side interface

c_{ref} is a reference concentration

d is a coefficient

Eq.(4) and (5) gives

$$\frac{1}{R} = 1 + B(25C, c_{ref}) \cdot \left(\frac{c}{c_{ref}} \right)^d \cdot \frac{TCFB}{J_w} \quad (6)$$

The temperature correction factors for both water and ammonium nitrate were assumed to follow Eq.(7).

$$TCFY = e^{\frac{E_Y}{R_g} \left(\frac{1}{298.15} - \frac{1}{273.15+T} \right)} \quad (7)$$

where

Y denotes A or B

E_A is an activation energy for water, which was set to 6100 cal/mol

E_B is an activation energy for the solute
 R_g is the molar gas constant, which is 1.9865 cal/(mol·K)
 T is the temperature in °C

Laboratory Tests

The flow schematic of the test unit that was used in the laboratory tests is shown in Fig.1. The feed pump was a Wanner M03 with maximum 11 L/minute (3 US gpm) flow rate. It was driven by a Baldor Inverter Drive® Motor IDM3587T, which has a 145TC NEMA frame. The shaft was turned down in the factory, so the frame was converted to NEMA 56C (except for a slightly longer shaft) to fit the Wanner M03 pump. To be able to vary the pump flow rate, a Baldor ID15H402-W Inverter was used, which had a NEMA 4X indoor enclosure.

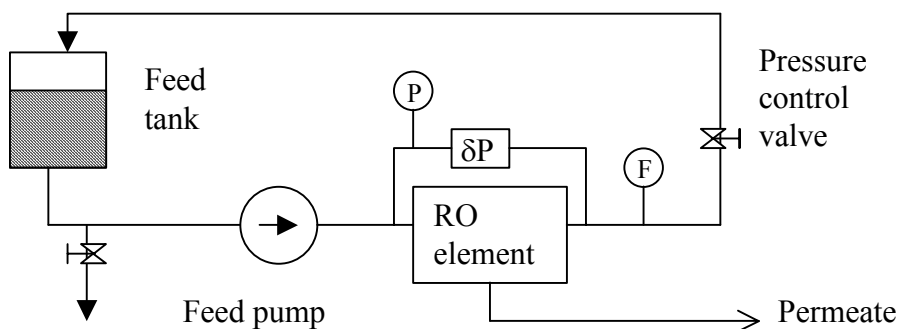


Figure 1. Flow schematic of the test unit. P is a pressure gage. δP is the differential pressure. F is a flowmeter. An AD1812C membrane element was used.

The temperature of the feed solution was measured with the conductivity meter simultaneously with the feed conductivity (taken from the feed tank). The conductivity meter was a Myron L Ultrameter™ 6P, which gave temperature compensated values to 25°C, using the temperature compensation factor that is valid for sodium chloride solutions. As described in Appendix A, the ammonium nitrate concentration was calculated from the conductivity reading, taking into account the error in the temperature compensation.

Test Solutions

The salt solutions were made up with deionized (DI) water that had a conductivity below 3 $\mu\text{S}/\text{cm}$ and Technical Grade ammonium nitrate from Gallade Chemical Inc.. To check for the purity of the ammonium nitrate and the correlation between conductivity and concentration of ammonium nitrate solutions, 20.0 g of ammonium nitrate was dissolved in 10.0 L DI water at about 25°C. This solution should have contained 0.200 wt% ammonium nitrate. The conductivity at 25°C of this solution was measured to 3276 $\mu\text{S}/\text{cm}$, which according to the equations in Appendix A corresponds to 0.199 wt% ammonium nitrate. The 0.5 percent discrepancy between the expected and calculated ammonium nitrate concentration was small enough to have no practical significance in the work reported here.

The pH of 0.2–5 wt% ammonium nitrate solutions was in the range 5.2-5.5.

Membrane Element

An AD1812C spiral wound membrane element was used for the tests. It was 0.30 m long, and had a nominal 0.79 mm (31 mil) thick diamond shape feed channel spacer, which

was measured to be 0.81 mm (32 mil) thick. The length of the feed channel spacer in the radial direction of the element was 0.89 m (35 inch), which gave a cross flow area for the feed of 0.00072 m². The active membrane area was measured after the test series to be 0.297 m² (3.2 ft²). It had been used in a previous test, and was considered stabilized with respect to pressure compaction.

The element was of a sanitary design, with a net-like cage as an outer wrap and a space between the element and the inner wall of the housing. To eliminate feed bypassing the element, an about 20 mm wide vinyl tape was wrapped around the outside of the element at the upstream end until it fit snugly inside the housing. After finishing the test, the author realized that a big part of the feed entering the element spiraled to the outside of the element and exited the element between the element and the wall of the housing. Thus, the fluid velocity inside the element was less than expected for most of the element length. The decrease in fluid velocity inside the element was not accounted for in the calculations, so the obtained feed side mass transfer coefficient was much smaller than initially expected.

Test Procedure

For each feed concentration of ammonium nitrate, the element performance was measured at different pressures, while both the concentrate and permeate were recirculated to the feed tank. No cooling device was available, so the temperature varied in the range 25-52°C. The ammonium nitrate concentrations were in consecutive order 2.4, 0.19, 0.6, 1.3, 2.8, 5.2 and 0.2 wt%. Appendix C describes the test procedure in more detail.

Each set of test data consisted of the temperature, feed pressure, concentrate flow rate, permeate flow rate, feed conductivity and permeate conductivity. The measured feed side pressure drop was never above 7 kPa (1 psi), so in the calculations, it was assumed that the average feed side pressure was 3.4 kPa (0.5 psi) lower than the feed pressure.

Test Data Evaluation and Results

The needed physical properties of ammonium nitrate solutions are listed in Appendix A. All the equations and test data were entered into an Excel spreadsheet. The constant "a" in the Sherwood number equation (Eq.(B-1)), and E_B (the activation energy for ammonium nitrate) in Eq.(7). where the only unknowns needed to calculate $A(25C)$ and $B(25C)$. E_B and the constant "a" were determined by the following trial and error method.

1. From the feed and permeate conductivities, calculate the corresponding ammonium nitrate concentrations, and through a mass balance the concentrate concentration. Calculate the average bulk concentration as the arithmetic average of that in the feed and the concentrate. This average bulk concentration (c_b) is assumed to be representative for the whole element, i.e. the same from the feed to the concentrate end of the element.
2. Calculate the density, viscosity and diffusivity of the average bulk solution inside the element. Use these values for the calculation of the Sh, Re and Sc numbers. It would probably be slightly better to use the physical properties at the membrane surface for these calculations, but that would greatly complicate the calculations.
3. Assume a value for the constant "a" in Eq.(B-1), and calculate the average mass transfer coefficient.
4. Calculate the average ammonium nitrate concentration at the membrane surface from Eq.(B-5).

5. Calculate $A(25C)$ from Eqs.(1), (7) and (A-6), and plot $A(25C)$ vs. average ΔP .
6. Go back to step 3, and assume a different value for the constant “a”. Chose the value of “a” that gives the best plots (straight lines) that are reasonable. For example, the element can be tested with pure water to determine $A(25C)$, and the value of $A(25C)$ obtained that way should agree with $A(25C)$ obtained from the tests with the solute.

After that the constant “a” was determined, E_B in Eq.(7) was determined by plotting $(1/R) - 1$ vs. $TCFB/J_w$ for different values of E_B . The value of E_B that gave the best linear curve fit, which extrapolated would intersect the origin, was assumed to be the correct value.

When the temperature is constant, or the expression for $TCFB$ is known, the solute permeability at the reference temperature and actual solute concentration is obtained from the slope of the plot $(1/R) - 1$ vs. $TCFB/J_w$ according to Eq.(4).

Because the temperature varied greatly during the test, and E_B was unknown, the constant “a” was determined from the best fit for the calculated water permeabilities. $a=0.18$ was chosen as the best fit, with the resulting water permeabilities shown in Fig.2. The data from the two first test days, Day 1 and Day 4, are not included, because until the end of the second test day, the calculated water permeabilities were up to 35 percent higher than those shown in Fig.2. Apparently, the element needed about 6 hours of operating time, which was the combined test time on the two first test days, to stabilize. As mentioned above, the value of the constant “a” was only about half of the expected one, and this was probably caused by a bad feed side flow distribution within the element.

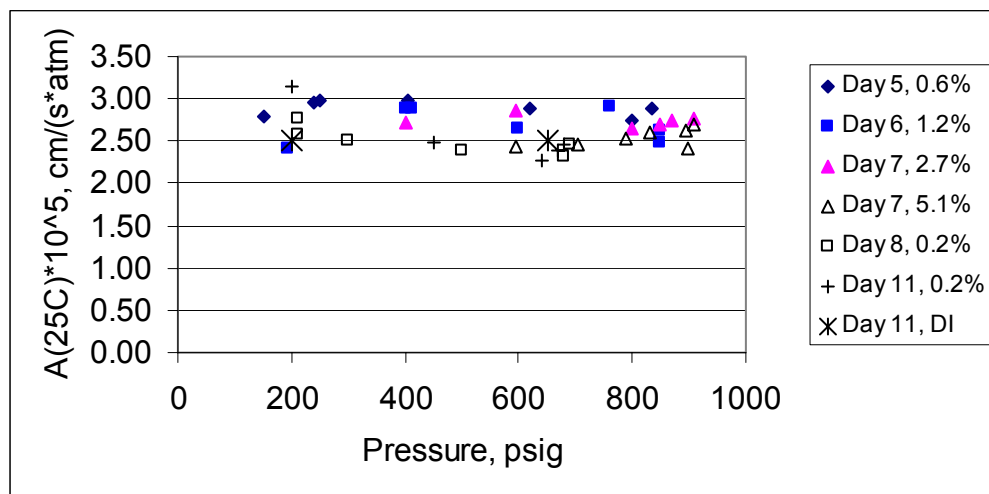


Figure 2. Calculated water permeabilities at 25°C for the AD1812 element with ammonium nitrate concentrations in the range 0.2-5.1 wt% at temperatures between 25 and 52°C, and at the end also for deionized water. The constant “a” in Eq.(B-1) was set to 0.18.

The water permeability decreased slightly with increasing feed pressure, which was caused by mostly reversible pressure compaction. It looks like the water permeability of the element decreased slightly from the beginning to the end of the test series. That could be from the membrane being slightly fouled during the tests.

With the constant “a” determined to be 0.18, E_B was determined from plots of Eq.(4). The test with the widest temperature range and about constant feed concentration was that on day 11 (27-50°C). Different E_B values were tried for the data from that test, and $E_B = 8500$ cal/mol gave the best fit, so that value was chosen. Fig.3 shows the graphs obtained from using $E_B = 8500$ and 6100 cal/mol.

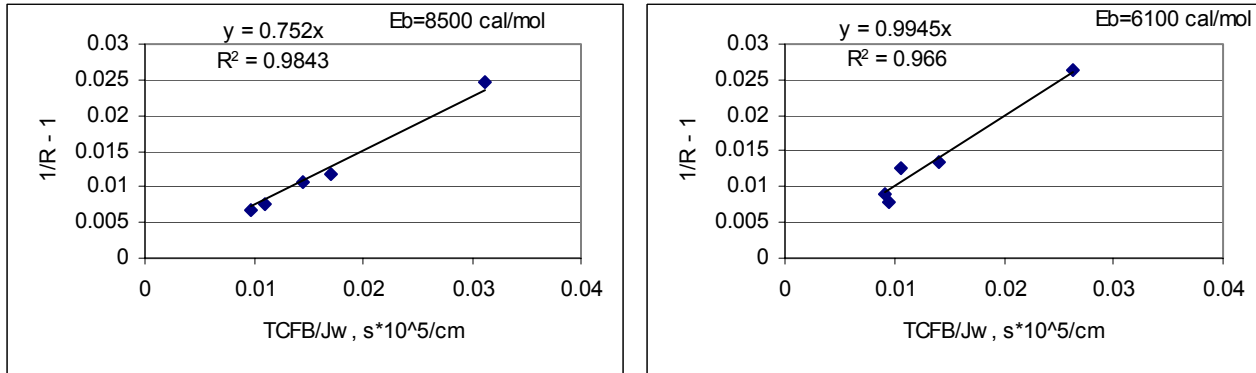


Figure 3. Ammonium nitrate rejection performance as a function of $TCFB/J_w$ at the tests on day 11. The feed ammonium nitrate concentration was 0.21-0.22 wt% and the temperature was 27-50°C with only two of the data points outside 38-40°C, the first (27°C) and third (50°C) data point from the left.

For reference, a permeate flux of 51 L/(m²·h) (30 gfd) at 25°C corresponds to $TCFB/J_w = 0.0072 \cdot 10^5$ cm/s, and a permeate flux of 8.5 L/(m²·h) (5 gfd) at 50°C corresponds to $TCFB/J_w = 0.10 \cdot 10^5$ cm/s. The abbreviation gfd stands for US gallons per ft² and day.

Fig.4 shows the same plots for all the tests with different ammonium nitrate concentrations and with $E_B = 8500$ cal/mol and $a=0.18$. The slopes of the lines in Fig.4 represent the ammonium nitrate permeabilities of the AD membrane at 25°C, $B(25C)$, at the respective concentration. The slopes are different at different concentrations, which means that the ammonium nitrate permeability is concentration dependent, in this case increasing with increasing concentration. For each feed concentration, the ammonium nitrate concentration at the membrane surface increased with increasing permeate flux, because of an increasing polarization modulus. Thus, the data points in Fig.4 should deviate slightly from the straight line, which makes it difficult to determine E_B with high accuracy.

The obtained $B(25C)$ from the two hours of testing on Day 1 was much higher than what was obtained from the other tests. The few hours test on the following test day (Day 4) gave a slightly higher $B(25C)$ than later tests at the same ammonium nitrate concentration. The reason for this is unknown. It is possible that it took a few hours for the membrane to stabilize in the ammonium nitrate solution. The data from these two days are not used when plotting the calculated $B(25C)$ versus the ammonium nitrate concentration at the feed side/membrane interface in Fig.5, which shows that $B(25C)$ is proportional to the ammonium nitrate concentration to the power 0.27.

At the end of test Day 1, feed pH was decreased from 5.2-5.5 to 3.7, and this did not increase the ammonium nitrate permeability of the membrane.

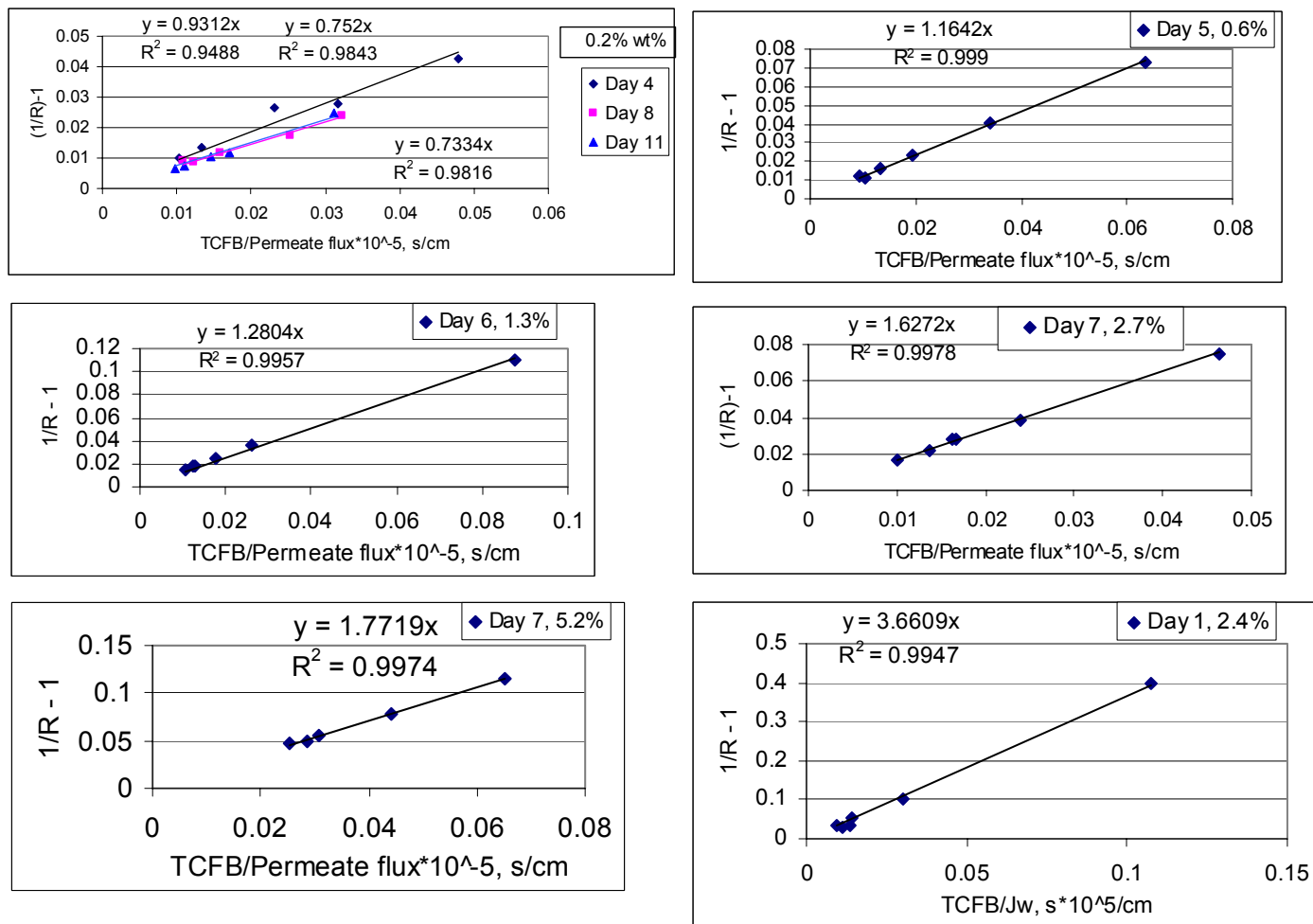


Figure 4. Ammonium nitrate rejection performance as a function of TCFB/ J_w for all the tests. $a=0.18$ and $E_B=8500$ cal/mol.

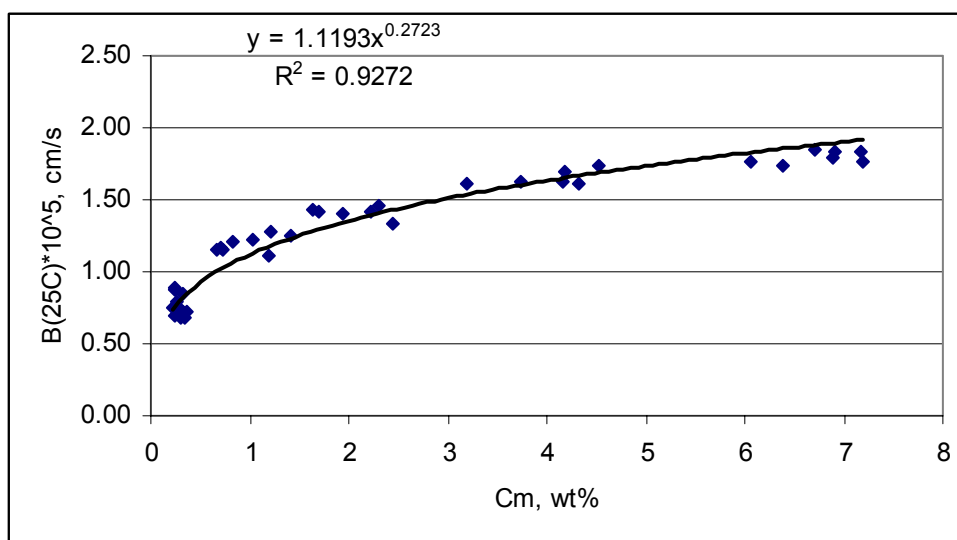


Figure 5. The calculated ammonium nitrate permeability at 25°C vs. ammonium nitrate concentration at the membrane surface. $a=0.18$ and $E_B=8500$ cal/mol.

To verify Fig.5, more than a year later, another test was carried out where 0.22 wt% ammonium nitrate was concentrated batchwise with another AD1812 element up to 1.6 wt% ammonium nitrate. At 0.2, 0.5 and 1.5 wt% concentrations, permeate was recycled back to the feed tank while the performance was measured at different pressures. The operating ranges were 14-56 bar (205-808 psi) for feed gage pressure and 20-30°C for temperature, except one data point each at 41 and 50°C. With the value of 0.27 for exponent d in Eq.(6), the ammonium nitrate rejection performance of that element is plotted in Fig.6, where c_{ref} was set to 1.00 wt% ammonium nitrate. All the data are fairly well correlated by a single line, which confirms that $B(25C)$ of the AD membrane for ammonium nitrate is proportional to the ammonium nitrate concentration to the power 0.27. The ammonium nitrate permeability at this second test was within a few percent of that obtained at the test series described in this report.

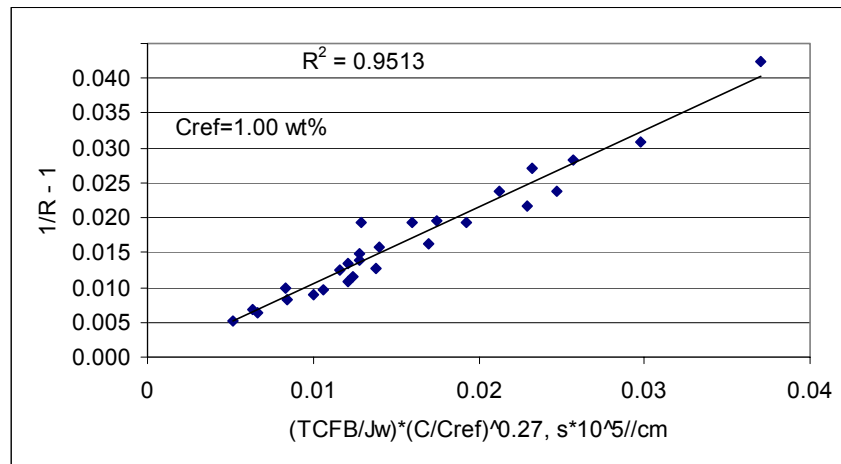


Figure 6. Ammonium nitrate rejection performance as a function of $c/c_{ref})^{0.27} \cdot TCFB/J_w$ at a later batchwise concentration test from 0.22-1.6 wt% ammonium nitrate. $c_{ref} = 1.00$ wt%.

Pilot Plant Testing

The laboratory tests determined the ammonium nitrate permeability as a function of temperature and ammonium nitrate concentration for the AD membrane, and confirmed that the water permeability is independent of the ammonium nitrate concentration. This is all the information that is required to make computer projections of full scale RO plant operation, by using Eqs.(1) and (2). However, before installing a full scale RO unit, it is highly recommended to run a pilot plant test at the actual site, to verify that no unknown factors will affect the RO membrane performance. In most cases, membrane fouling is the most critical factor, but it was not expected to be significant when operating on condensates from the ammonium nitrate evaporator. Pilot plant testing was conducted at two different ammonium nitrate manufacturing plants in North America, here denoted Plant 1 and Plant 2.

The flow schematic of the pilot plant RO unit is shown in Fig.7. It had two AD4040F elements in series, each 0.1 m (4") in diameter, 1 m (40") long and about 7.7 m² (83 ft²) active membrane area. At both plants, only batchwise runs were made. A feed tank was filled with the condensate, and nitric acid was added if needed to convert all ammonia to ammonium nitrate. Then the RO unit concentrated this solution by returning the concentrate to the feed tank while collecting the permeate in a second tank. A better configuration had been to operate in a continuous mode with both the permeate and concentrate leaving the RO system. This

can be done at high permeate recoveries by recirculating most of the concentrate back to the suction side of the booster pump. A continuous mode of operation was not practical at either plant, however.

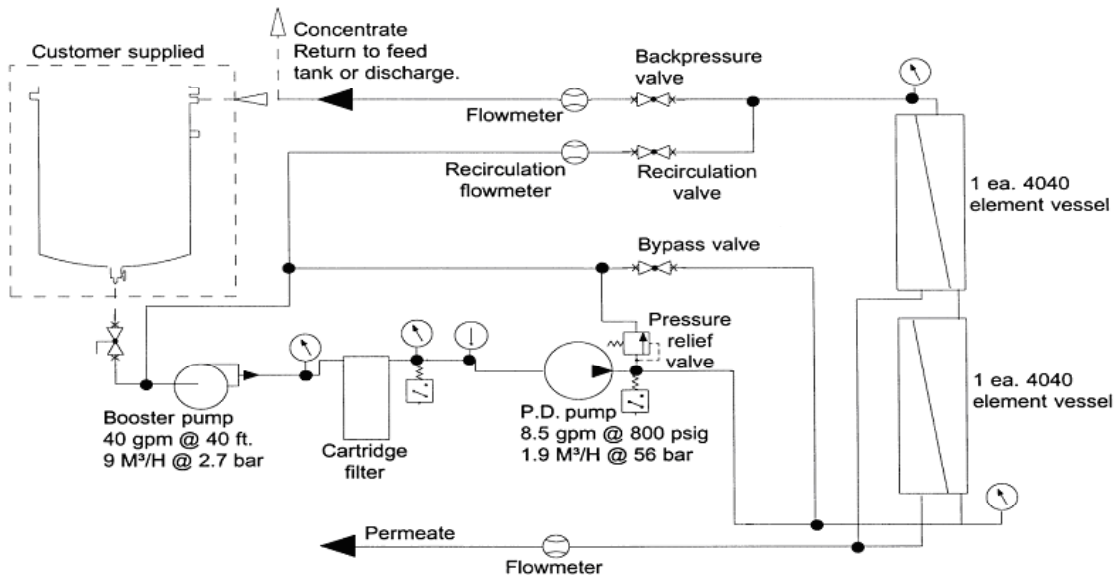


Figure 7. Flow schematic of the pilot plant RO unit used at two ammonium nitrate manufacturing plants.

Plant 1

Plant 1 operated the pilot plant unit for batchwise concentrations for a few months, but not every day. The ammonium nitrate concentrations were measured both with a conductivity meter and with the plant's standard laboratory analysis method, although the latter method was normally not used for the low ammonium nitrate concentrations that were present in the streams to and from the RO unit. The two different analyses methods resulted in widely different ammonium nitrate concentrations, and neither gave any result that made sense. In the last batchwise concentration (from 2 wt% to 12 wt% ammonium nitrate), samples of feed, permeate and concentrate were taken about every half hour, and these samples were shipped to Osmonics for conductivity measurements. The pH was measured in one of these samples, and it was 6.0. The data from that run are shown in Table 1. The ratio (conductivity measured at the plant)/(conductivity measured at Osmonics) for the same samples was in the range 0.35-0.9, and generally, the higher the conductivity, the lower was this ratio.

To calculate the water and ammonium nitrate permeabilities of the AD4040F elements, the constant "a" in Eq.(B-1) and E_B in Eq.(7) were set to 0.35 and 8500 cal/mol respectively. The results are shown in Figs.8-9. The water permeability was about at the AD element nominal value at the lowest ammonium nitrate concentration, but it decreased with increasing concentration. Further testing at the site might have revealed the reason for this decrease in the calculated water permeability, but further testing was not an option at that time. $a=0.35$ is a typical value for these elements, at least when operating on sodium chloride solutions. Other values for "a" were examined, but all values resulted in a water permeability that was strongly dependent on the ammonium nitrate concentration.

Table 1. Raw data from the last batchwise concentration of ammonium nitrate (NH₄NO₃) condensate at Plant 1. The conductivities were measured at Osmonics laboratory close to 25°C. The concentrate flow rate was 23-30 L/minute (6-8 US gpm). A permeate flow rate of 1 US gpm corresponds to a permeate flux of 14.7 L/(m²·h) (8.7 gfd).

| Day | Time | Temp | Feed Pressure | Permeate flow rate | Conductivity at 25°C, mS/cm | | Calculated NH ₄ NO ₃ concentration, wt% | | |
|-----|-------|------|---------------|--------------------|-----------------------------|------|---|----------------|--------|
| | | °C | psig | US gpm | Feed | Perm | Feed | c _m | Perm |
| 1 | 10:00 | 14 | 780 | 1.9 | 29.3 | 1.47 | 2.04 | 2.76 | 0.0866 |
| 1 | 10:15 | 14 | 780 | 1.9 | 29.5 | 1.4 | 2.05 | 2.78 | 0.0823 |
| 1 | 10:30 | 14 | 780 | 1.9 | 30.1 | 1.43 | 2.10 | 2.84 | 0.0842 |
| 1 | 10:45 | 14 | 780 | 1.875 | 30.9 | 1.49 | 2.16 | 2.91 | 0.0878 |
| 1 | 11:00 | 14 | 780 | 1.85 | 31.3 | 1.53 | 2.19 | 2.94 | 0.0902 |
| 1 | 11:30 | 15 | 780 | 1.8 | 33 | 1.65 | 2.32 | 3.09 | 0.0975 |
| 1 | 12:00 | 16 | 780 | 1.75 | 34.5 | 1.8 | 2.43 | 3.21 | 0.1067 |
| 1 | 12:30 | 16 | 780 | 1.7 | 37 | 2.02 | 2.62 | 3.43 | 0.1202 |
| 1 | 13:00 | 16 | 780 | 1.7 | 39.4 | 2.25 | 2.81 | 3.67 | 0.1345 |
| 1 | 13:30 | 17 | 780 | 1.675 | 40.2 | 2.36 | 2.87 | 3.72 | 0.1413 |
| 1 | 14:00 | 17 | 780 | 1.65 | 42.1 | 2.57 | 3.02 | 3.89 | 0.1545 |
| 1 | 14:30 | 18 | 780 | 1.6 | 45 | 2.96 | 3.25 | 4.14 | 0.1790 |
| 1 | 15:00 | 19 | 780 | 1.6 | 49.3 | 3.44 | 3.58 | 4.55 | 0.210 |
| 1 | 15:30 | 20 | 780 | 1.55 | 52.1 | 3.88 | 3.80 | 4.78 | 0.238 |
| 1 | 16:00 | 21 | 780 | 1.5 | 57 | 4.71 | 4.15 | 5.05 | 0.291 |
| 2 | 10:00 | 21 | 780 | 1.45 | 57.5 | 4.75 | 4.19 | 5.06 | 0.294 |
| 2 | 10:30 | 21 | 780 | 1.4 | 61.3 | 5.36 | 4.50 | 5.39 | 0.333 |
| 2 | 11:00 | 22 | 800 | 1.25 | 67.1 | 6.4 | 4.97 | 5.83 | 0.401 |
| 2 | 11:30 | 23 | 805 | 1.2 | 72.3 | 7.58 | 5.39 | 6.25 | 0.479 |
| 2 | 12:00 | 23 | 810 | 1.1 | 79.5 | 8.97 | 5.98 | 6.81 | 0.573 |
| 2 | 13:00 | 26 | 830 | 0.9 | 93.3 | 13.9 | 7.13 | 7.89 | 0.913 |
| 2 | 13:30 | 26 | 830 | 0.85 | 99.2 | 15.4 | 7.63 | 8.34 | 1.019 |
| 2 | 14:00 | 27 | 855 | 0.75 | 110 | 19.8 | 8.55 | 9.23 | 1.334 |
| 2 | 14:30 | 29 | 855 | 0.7 | 119.5 | 24 | 9.37 | 10.03 | 1.641 |
| 2 | 15:00 | 31 | 880 | 0.65 | 127.2 | 31.1 | 10.04 | 10.65 | 2.17 |
| 2 | 15:30 | 32 | 880 | 0.6 | 136 | 37 | 10.81 | 11.39 | 2.62 |
| 2 | 16:00 | 34 | 905 | 0.5 | 147 | 45.5 | 11.79 | 12.28 | 3.28 |
| 2 | 16:30 | 36 | 905 | 0.475 | 155 | 54.9 | 12.51 | 12.96 | 3.99 |

When the pilot plant unit was returned to Osmonics, the two AD elements was tested with a 3.4 wt% sodium chloride solution using the same pilot plant unit. The water permeability of the elements was then normal at $3 \cdot 10^{-5}$ cm/(s·atm), so the performance of the AD elements did not change irreversibly at the last batchwise concentration at Plant 1. The calculated sodium chloride permeability from the same 3.4 wt% sodium chloride test was about double as high as the nominal value for AD elements.

Compared to the AD1812 elements that were tested in the laboratory, the two field tested AD4040F elements had 2 times higher sodium chloride permeability but 4 times higher

ammonium nitrate permeability. The reason for this is unknown. It is possible that an increase in the sodium chloride permeability of a membrane is always followed by a greater increase in the ammonium nitrate permeability. For the AD membrane, the permeability and exponent d in Eq.(5) for ammonium nitrate are several times higher than those for sodium chloride.

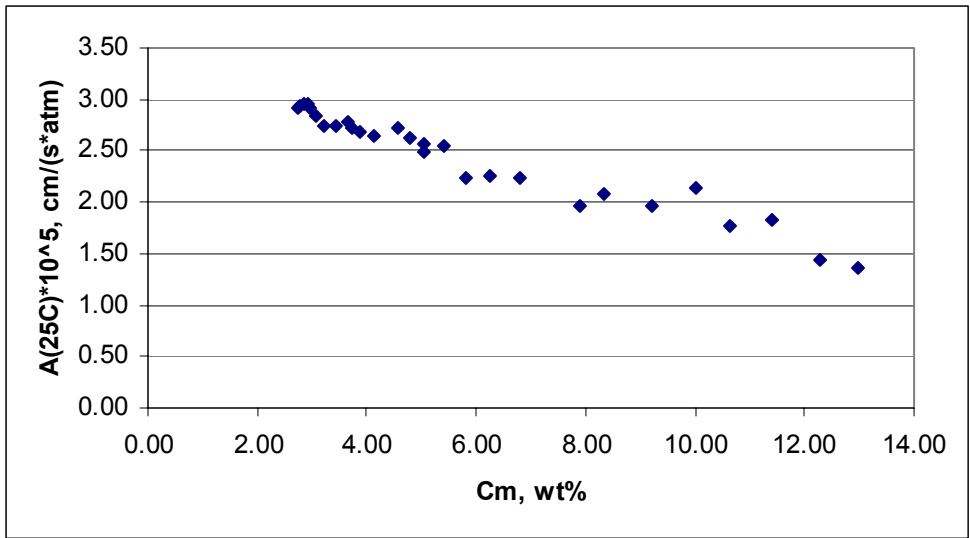


Figure 8. Calculated water permeability of the two AD4040F elements during the last batchwise concentration run at Plant 1.

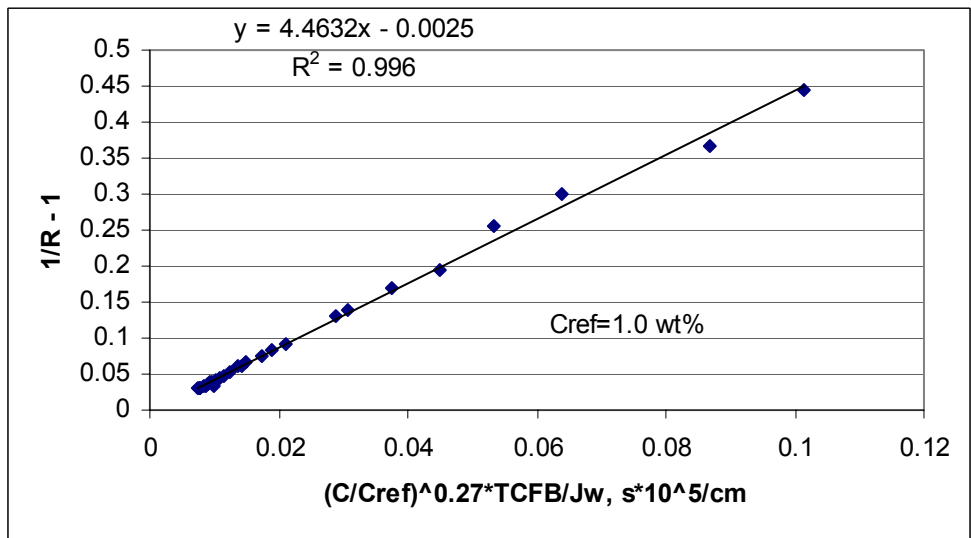


Figure 9. Calculated ammonium nitrate permeability of the two AD4040F elements during the last batchwise concentration run at Plant 1.

After the end of the pilot plant study, the ammonium nitrate manufacturing plant made a process change that decreased the amount of condensate that needed treatment, so there was no immediate need for an RO system.

Plant 2

Plant 2 had the same problem with analyzing the ammonium nitrate concentration as did Plant 1. No samples were analyzed at the Osmonics laboratory, so no accurate data are available from that test, which only lasted for a week, mostly at 43°C. The Plant 2 personnel learned, however, that RO could concentrate the dilute ammonium nitrate solution up to above 8 wt% ammonium nitrate, and that a 3-pass RO system could produce a permeate with less than 50 mg/L ammonium nitrate, which is low enough ammonium nitrate concentration to be reused within the plant.

Important Factors with Regards to Purchase of RO Plant

In Plant 2 the condensate went to a single effect evaporator, which is an energy intensive unit. The savings in costs of energy would give a fast pay-off time for an RO system to pre-concentrate the ammonium nitrate condensate before feeding it to the single effect evaporator. This is shown in Fig.10, which is based on the following numbers.

Final permeate flow rate (reduction of flow to the evaporator): 11.4 m³/h (50 US gpm)

Total cost for a 3-pass RO system, including tanks (buildings not included): \$ 1 million

Heat required to heat and evaporate the water: 2200 kJ/kg

Power to run the pumps in the RO plant: 208 kW**

Cost of electricity: 0.05 \$/kWh, which with 208 kW results in \$ 5,586 per month

Costs for supervision, maintenance, repair and consumables for RO plant: \$15,000 per month*

* This needs not to be the actual cost for the plant described in this paper. This cost is not critical to the overall economy at a natural gas price of \$5 per million BTU, because at that price, the savings in gas consumption represent \$ 85,000 per month.

** This is the combined effect rating for all the motors used in the RO system. In reality, the power consumption will be less.

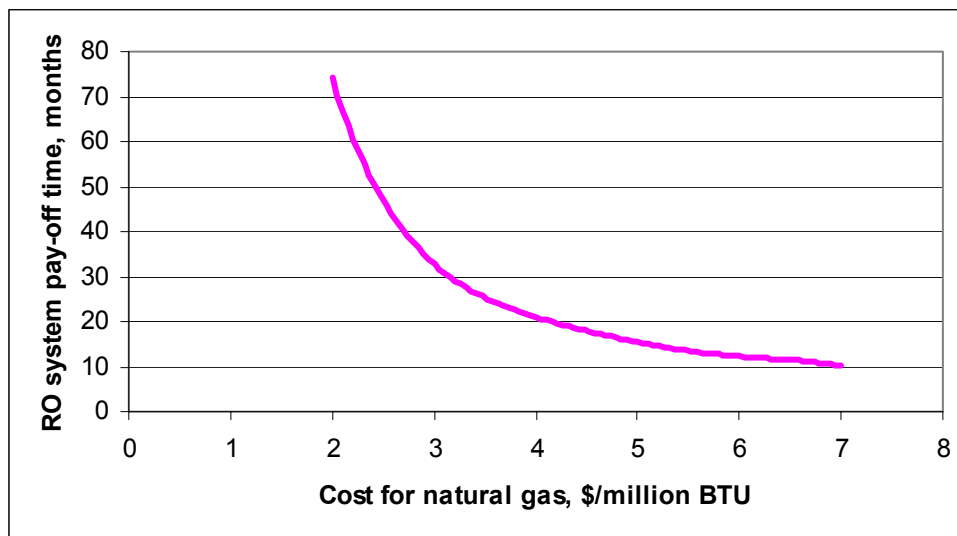


Figure 10. Pay-off time for the RO system as a function of the price for natural gas. \$5 per million BTU is equivalent to \$ 0.017 per kWh of heat.

With a natural gas price of \$5 per million BTU, the pay-off time would not exceed 16 months. The end-user is most times hesitant to a new technology, however, even when the

pay-off time is short. The plant personnel have no experience with the new technology, and do not know what problems could or could not arise. In this case, the seller, GE Water & Process Technologies (GEWPT), offered several options to minimize the risk for the end-user with regards to ownership and operation. The end-user chose to purchase the RO system, and pay a monthly fee for its supervision, maintenance, repair and consumables, including membrane elements. GEWPT personnel were already present at the site, servicing its water and wastewater needs, which made it easy to add servicing the RO system to the contract.

Full Scale RO System

The RO membrane does not reject ammonia, so the ammonia that might be present in the condensate must first be converted to ammonium nitrate by addition of nitric acid before entering the RO unit. After this neutralization at Plant 2, a typical value of the ammonium nitrate concentration in the condensate was 2 wt%, so this was chosen as the design value. The condensate flow rate was about 15 m³/h (67 US gpm). The RO system was designed to be able to produce 11.4 m³/h (50 US gpm) final permeate with less than 50 mg/L ammonium nitrate from this condensate. The ammonium nitrate concentration in the concentrate going to the single effect evaporator would then be 8 wt%.

To have the highest ammonium nitrate rejection, the temperature should be as low as possible. This plant, however, wanted to keep the concentrate and preferably also the final permeate at a high temperature, so the RO unit was designed to be able to operate up to 60°C. A heat exchanger is included in the design, so the temperature can be decreased, should there be a need to reduce the ammonium nitrate concentration in the final permeate.

The water and ammonium nitrate permeabilities obtained from the laboratory study, Figs.2 and 5, were used in a computer projection program for the design of the full scale RO system. A 3-pass RO system was needed, each pass consisted of 3 housings arranged in a 2-1 array, with 5 AD8040 elements per housing. A simplified flow schematic is shown in Fig.11. In case nitric acid is overdosed in the neutralization tank, a control system is in place to raise the pH to neutral in the equalization tank, which contains the feed water to the 2nd pass RO unit. Table 2 gives predicted data at design conditions, assuming an operating temperature of 50°C.

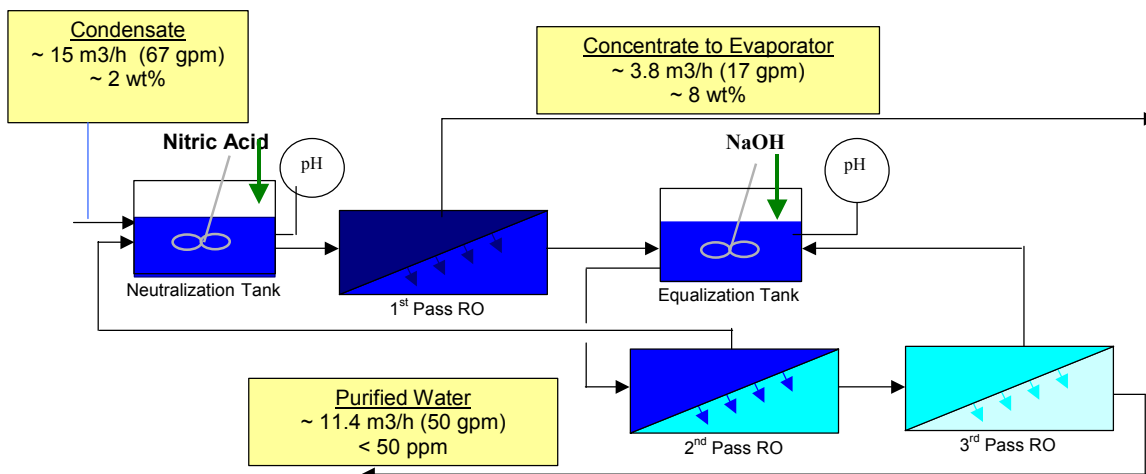


Figure 11. Simplified flow schematic of the full scale RO system at design conditions. The stated concentrations are those of ammonium nitrate after neutralization.

Table 2. Predicted operating performance for the 3-pass RO system at design conditions and 50°C. 1 wt% equals to 10⁴ ppm.

| | 1 st Pass | 2 nd Pass | 3 rd Pass |
|--|----------------------|----------------------|----------------------|
| Primary feed flow rate, m ³ /h (US gpm) | 15.1 (67) | 13.2 (58) | 13.6 (60) |
| Recirculated concentrate from upstream pass, m ³ /h (gpm) | 1.9 (8) | 2.3 (10) | 0 |
| Total pump flow rate, m ³ /h (US gpm) | 17 (75) | 15.5 (68) | 13.6 (60) |
| Required feed gage pressure, bar (psi) | 41 (600) | 22 (315) | 15 (210) |
| Feed ammonium nitrate concentration | 2.0 wt% | 0.32 wt% | 400 ppm |
| Permeate ammonium nitrate concentration | 0.32 wt% | 400 ppm | 26 ppm |
| Concentrate ammonium nitrate concentration | 7.9 wt% | 2.3 wt% | 0.23 wt% |

The RO system is controlled with an Allen Bradley PLC with many control loops, so the system can operate without operator assistance at a wide range of temperature, ammonium nitrate concentration and flow rate of the incoming condensate. The motors to the three RO feed pumps and one more pump in the RO system have variable frequency drives. The high pressure pumps are FEDCO multistage centrifugal pumps, with a maximum pressure of 62 bar (900 psi) in the 1st and 2nd pass, and 41 bar (600 psi) in the 3rd pass.

The RO system has not started up when this paper was written, but will very likely be in operation when this paper is presented.

Conclusions

Extensive laboratory tests were conducted to determine the separation characteristics when using RO to concentrate ammonium nitrate solutions. The ammonium nitrate permeability was determined to be proportional to its concentration to the power of 0.27. In subsequent pilot plant tests, there were problems with the ammonium nitrate concentration analyses, so the obtained performance data were not accurate. However, the pilot plant tests confirmed that the membrane fouling rate was very low when operating on the ammonium nitrate condensate, and convinced the plant personnel that RO can be used to concentrate the ammonium nitrate condensate, while obtaining a permeate that can be reused within the plant.

Even though the pay-off time of an RO system would be below 1.5 years, the end-user might hesitate in the investment because of unfamiliarity with such systems. This was overcome in this case by contracting the supervision, maintenance, repair and consumables for the RO system to the seller of the system, GE Water & Process Technologies, for a fixed monthly cost.

Acknowledgments

The author is thankful to Andrew McCreery, who took most of the readings on test Days 6 and later of the laboratory study, and Larry Lien, Dick Simonis and Robert Reitz, GE Water & Process Technologies, for information about the full scale RO system and pilot plant testing at Plant 2.

Appendix A: Physical Properties of Ammonium Nitrate Solutions

The ammonium nitrate condensates contain no significant amounts of impurities, so the physical properties of these are the same as for aqueous ammonium nitrate solutions, which are easy to find in commonly used reference books. The properties that are of importance for RO are, electrical conductivity (for concentration measurement), osmotic pressure, density, viscosity and diffusivity.

Density

The density of ammonium nitrate solutions was obtained from curve fitting data at 25°C from Perry (1997) Table 2-37, and assuming that the same correlation is valid at any temperature, provided the density of pure water at the actual temperature is inserted. The result is shown in Eqs.(A-1 a) and (A-1 b).

$$\rho = \rho_w \cdot (1 + 0.00408 \cdot c_{wt\%}) \quad c_{wt\%} < 8 \quad (\text{A-1 a})$$

$$\rho = \rho_w \cdot (1 + 0.00466 \cdot c_{wt\%}) \quad 8 < c_{wt\%} < 24 \quad (\text{A-1 b})$$

where

ρ is the density of the ammonium nitrate solution in kg/m^3

ρ_w is the density of pure water at the actual temperature

$c_{wt\%}$ is the ammonium nitrate concentration in wt%

The two equations cause a discontinuity at 8 wt% ammonium nitrate. Their resulting density values at that concentration differ by 0.4 percent, but this should not cause any practical problems. To get an estimate of the error at higher temperatures, Eq.(A-1 a) was used to calculate the density of 8 wt% NH_4NO_3 at 60°C. It predicts the density 1015.3 kg/m^3 , while Perry (1997) lists 1014.2 kg/m^3 . This small error in Eq.(A-1) is insignificant for this work.

Viscosity

The viscosity of ammonium nitrate solutions was calculated using Eq.(A-2), which was generated from data in Robinson and Stokes (1968) pp. 304 and 516.

$$\mu_T = \mu_{w,T} \cdot [1 + 0.005 \cdot (M_i)^{0.5} + (0.0017 \cdot T - 0.0975) \cdot M_i] \quad (\text{A-2})$$

where

μ_T is the solution viscosity at the temperature $T^\circ\text{C}$

$\mu_{w,T}$ is the viscosity of pure water at the temperature $T^\circ\text{C}$

M_i is the molar concentration of ammonium nitrate

T is the temperature in $^\circ\text{C}$

Robinson and Stokes (1968) states that the general equation that was used to generate Eq.(A-2) usually is valid up to a few tenths molar salt concentration, which in this case means a few wt% ammonium nitrate. No attempt was made to estimate the error in Eq.(A-2) for higher salt concentrations.

Diffusivity

The diffusivity of ammonium nitrate in water was assumed to follow Eq.(A-3)

$$D_T = D_{25} \cdot [(T+273)/298] \cdot [\mu_{25}/\mu_T] \quad (\text{A-3})$$

where

D_T is the diffusivity of ammonium nitrate in water at $T^\circ\text{C}$, m^2/s

D_{25} is the diffusivity at 25°C , m^2/s

To calculate the diffusivity at 25°C for ammonium nitrate in water, the data up to 2 M (15 wt%) concentration in Robinson and Stokes (1968), Appendix 11.2 on p.515, were curve fitted to give Eq.(A-4). Fig.A-1 shows the data and the curve fit. The maximum deviation between the listed data and predicted from Eq.(A-4) is 0.1 percent.

$$D_{25} = (1.929 + 110.5098 \cdot M_i) / (1 + 63.224 \cdot M_i + 2.2789 \cdot M_i^2) \cdot 10^{-9} \quad (\text{A-4})$$

where

M_i is the molar concentration of ammonium nitrate

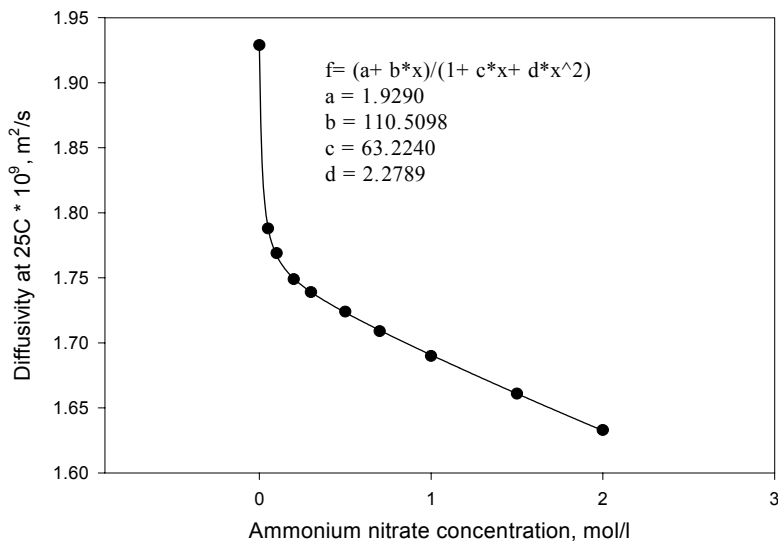


Figure A-1. Diffusivity at 25°C for ammonium nitrate in water according to Robinson and Stokes (1968), Appendix 11.2 on p.515

Osmotic Pressure

Robinson and Stokes (1968) in Appendix 8.10 Table 3, p. 485 lists the molal osmotic coefficient for ammonium nitrate from 0.1 to 6 molal. The data up to 2 molal, and adding that the molal osmotic coefficient is 1 at zero concentration, were curve fitted to give Eq.(A-5). The data and curve fit are shown in Fig.A-2. The maximum deviation between the listed data and predicted from Eq.(A-5) is 0.26 percent. The osmotic pressure is then calculated from Eq.(A-6).

$$\phi = (0.9998 + 13.1612 \cdot m_i) / (1 + 15.2671 \cdot m_i + 0.9267 \cdot m_i^2) \quad (\text{A-5})$$

$$\pi = \phi \cdot R \cdot (T + 273.15) \cdot \frac{MW_{\text{solv}}}{V} \cdot \sum m_i \quad (\text{A-6})$$

where

ϕ is the molal osmotic coefficient

m_i is the molality of ammonium nitrate (mol/kg solvent).

Σm_i is the total molality of dissolved species. One mol NH_4NO_3 in water gives two mol dissolved species, one mol of each ammonium ions and nitrate ions.

π is the osmotic pressure

R is the gas constant (8.314 J/(K·mol))

T is the temperature in °C

MW_{solv} is the molecular weight of the solvent (0.01802 kg/mol for water)

V is the partial molal volume of the solvent

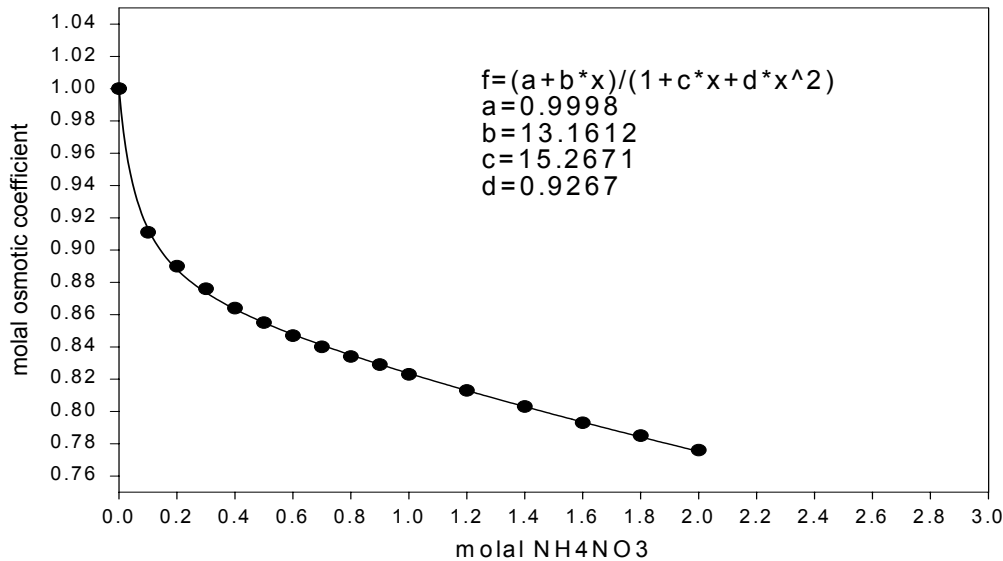


Figure A-2. Molal osmotic coefficient for ammonium nitrate in water according to Robinson and Stokes (1968), Appendix 8.10 Table 3, p. 485

The partial molal volume of water changes slightly with the salinity. Stoughton and Lietzke (1965) proposed Eq.(A-7) for the calculation of the partial molal volume of water in sodium chloride solutions at 25°C.

$$\frac{V}{MW_{\text{solv}}} = (1.00267 - 2.1045 \cdot 10^{-5} \cdot c_{\text{wt}\%}^2) \cdot 10^{-3} \quad (\text{A} - 7)$$

where

c is wt% NaCl, and V/MW_{solv} is expressed as m^3/kg

Eq.(A-7) was used to calculate V/MW_{solv} even for ammonium nitrate solutions at different temperatures. The error in doing so should be insignificant.

Conductivity

Conductivity data listed in ICT (1926) for up to 0.5 M (4 wt%) ammonium nitrate solutions at 25°C were curve fitted to give Eqs.(A-8 a) and (A-8 b). The maximum deviation between the listed conductivity data and predicted from Eqs.(A-8 a) and (A-8 b) is 1.3 percent for ammonium nitrate concentrations below 0.016 wt%, and 0.3 percent for ammonium nitrate concentrations above 0.04 wt%.

Lange (1979) lists conductivity data for ammonium nitrate solutions at 18°C up to 5 M. At the four concentrations in common between 0.01 and 0.5 M, the ratio [(Lange's data at 18°C)/(ICT's data at 25°C)] is in the range 0.859-0.875, with the average 0.867. There was no consistent trend in this ratio, but the highest value (0.875) occurred for the highest concentration. It was assumed that the ratio was 0.870 at all concentrations higher than 0.5 M. Then, the Lange's temperature compensated to 25°C conductivity data up to 2 M (15 wt%) ammonium nitrate were curve fitted to yield Eq.(A-8 c). The deviation between the temperature compensated listed conductivity data and predicted from Eq.(A-8 c) is below 1 relative percent for ammonium nitrate concentrations in the range 4-15 wt%.

$$\text{For } \kappa(25\text{C}) < 6400 \\ c_{\text{wt}\%} = -89.0442 \cdot 10^{-15} \cdot \kappa(25\text{C})^3 + 1.45506 \cdot 10^{-9} \cdot \kappa(25\text{C})^2 + 56.9528 \cdot 10^{-6} \cdot \kappa(25\text{C}) \quad (\text{A-8 a})$$

$$\text{For } 6400 < \kappa(25\text{C}) < 54000 \\ c_{\text{wt}\%} = -1.9078 \cdot 10^{-15} \cdot \kappa(25\text{C})^3 + 2.9770 \cdot 10^{-10} \cdot \kappa(25\text{C})^2 + 6.2894 \cdot 10^{-5} \cdot \kappa(25\text{C}) - 0.0135 \quad (\text{A-8 b})$$

$$\text{For } 54000 < \kappa(25\text{C}) < 196000 \\ c_{\text{wt}\%} = 5.165 \cdot 10^{-12} \cdot \kappa(25\text{C})^2 + 7.658 \cdot 10^{-5} \cdot \kappa(25\text{C}) - 0.1634 \quad (\text{A-8 c})$$

where

$c_{\text{wt}\%}$ is weight percent ammonium nitrate
 $\kappa(25\text{C})$ is the conductivity at 25°C in $\mu\text{S}/\text{cm}$

Many conductivity meters will temperature compensate the conductivity assuming the temperature compensation factor is the same as for a sodium chloride solution. In such cases, the real temperature compensated conductivity value for an ammonium nitrate solution is obtained from Eq.(A-9 a).

$$\kappa(25\text{C})_{\text{AN}} = \kappa_{\text{read}} \cdot [\text{CTCF}(T)_{\text{NaCl}}/\text{CTCF}(T)_{\text{AN}}] \quad (\text{A-9 a})$$

where

κ_{read} is the temperature compensated conductivity reading of the instrument
 CTCF(T) is the conductivity temperature compensation factor $\{\kappa(T)/\kappa(25\text{C})\}$ of the solution at T°C, where $\kappa(T)$ is the conductivity at the actual temperature T at the reading
 subscript AN and NaCl refer to ammonium nitrate and sodium chloride respectively

The temperature dependence of the conductivity of ammonium nitrate solutions differs slightly from that of sodium chloride solutions. ICT (1926) lists the conductivities of ammonium nitrate and sodium chloride solutions in the concentration range 1 mmol/l – 0.5 mol/l at both 25 and 50°C. The ratio (conductivity at 50°C)/(conductivity at 25°C) is 1.7 to 3.3 percent lower for ammonium nitrate solutions than for sodium chloride solution, with the average value 2.61 percent. It is assumed that the temperature compensation factor for ammonium nitrate solutions follows Eq.(A-9 b).

$$\text{CTCF}(T)_{\text{AN}} = [1 - 0.0261 \cdot (T-25)/25] \cdot \text{CTCF}(T)_{\text{NaCl}} \quad (\text{A-9 b})$$

Eqs.(A-9 a) and (A-9 b) give

$$\kappa(25C)_{AN} = \frac{\kappa_{read}}{1 - 0.0261 \cdot \frac{T - 25}{25}} \quad (A - 9c)$$

The obtained equations were checked with ammonium nitrate solutions made by weighing technical grade ammonium nitrate and added deionized water. The same conductivity meter that was used in the laboratory experiments was used to measure the conductivities of these prepared solutions. The measured and calculated conductivities agreed within 1 percent up to 67 mS/cm conductivity, but then they started to deviate from each other, to differ by 6 percent at 150 mS/cm conductivity. Eq.(A-10) was obtained from curve fitting the measured conductivities vs. the ammonium nitrate concentrations obtained from the weight measurements, and this equation was used instead of Eq.(A-8c) at high conductivities. The deviation might have been caused by the conductivity meter not being accurate at the high conductivities. The highest conductivity standard available in the laboratory at the moment was 64 mS/cm.

For $54000 < \kappa(25C) < 196000$

$$c_{wt\%} = 51.793 \cdot 10^{-12} \cdot \kappa(25C)^2 + 74.246 \cdot 10^{-6} \cdot \kappa(25C) - 0.24671 \quad (A-10)$$

References

ICT (1926). International Critical Tables, vol.6, published by McGraw Hill Book Company, New York, 1926.

Lange (1979). Lange's Handbook of Chemistry, 12th edition, edited by John A. Dean, McGraw-Hill Book Company, 1979

Perry (1997). Perry's Chemical Engineers' Handbook, seventh edition, edited by R.H. Perry and D. Green, McGraw-Hill, USA, 1997.

R. A. Robinson and R. H. Stokes (1968), "Electrolyte Solutions", reprinted and revised 2nd edition, Butterworths, London (1968).

R. W. Stoughton and M. H. Lietzke (1965), "Calculation of Some Thermodynamic Properties of Sea Salt Solutions at Elevated Temperatures from Data on NaCl solutions", Journal of Chemical and Engineering Data, vol. 10, no. 3 (1965).

Appendix B: Concentration Polarization

The solute concentration is higher at the membrane surface than in the bulk solution, because of concentration polarization. Eqs.(B-1)-(B-5) were used to calculate the solute concentration at the membrane surface.

$$Sh = a \cdot Re^{0.54} \cdot Sc^{0.33} \quad (B-1)$$

$$Sh = \frac{k_{avg} \cdot d_h}{D_T} \quad (B-2)$$

$$Re = \frac{v \cdot d_h \cdot \rho}{\mu_T} \quad (B-3)$$

$$Sc = \frac{\mu_T}{\rho \cdot D_T} \quad (B-4)$$

where

Sh is a Sherwood number

Re is a Reynolds number

Sc is a Schmidt number

a is a constant to be determined

k_{avg} is the average mass transfer coefficient in the channel, (m/s)

d_h is the characteristic length, (hydraulic diameter), which in this paper is defined as double the spacer thickness

D_T is the diffusivity of the solute at the actual temperature T, (m^2/s)

μ_T is the viscosity of the solute at the actual temperature T, ($kg/(m \cdot s)$)

ρ is the density of the feed solution, (kg/m^3)

v is the fluid superficial velocity, (m/s)

The superficial velocity is calculated assuming that the feed spacer occupies a zero volume in the feed channel.

With known flow rates, feed channel geometry, the constant a, and physical properties of the solution, Eqs.(B-1)-(B-4) give the average mass transfer coefficient. Eq.(B-5) is then used to calculate the average solute concentration at the membrane surface.

$$\frac{c_m - c_p}{c_b - c_p} = e^{\frac{J_w}{k_{avg}}} \quad (B-5)$$

where

J_w is the permeate flux, m/s

c is the solute concentration in wt%

the subscripts m, b and p denote the feed side/membrane interface, bulk solution, and permeate respectively.

The polarization modulus is defined as the ratio c_m/c_b .

The constant “a” is determined by a curve fit of the actual experimental data. A value in the range 0.3- 0.5 is expected for spiral wound elements with standard diamond shape feed channel spacers.

Appendix C: TEST PROCEDURE

The test series started on July 14, which is denoted day 1, and ended on July 24, which is denoted day 11. The day number is the date of the month minus 13.

The volume of the feed solution was about 10 L. The test mode was recirculation of permeate and concentrate all the time. There was no cooling available. To minimize the temperature variation during a test series, except for day 4, the initial pressure in the test series was the highest pressure. On a change in operating conditions (except for the temperature), readings were taken after 10-20 minutes on day 1, after about 30 minutes on day 4, and after about 45 minutes the following days, unless otherwise stated. The RO unit was always shut down overnight. Except between Day 1 and day 4, the used ammonium nitrate solution remained in the system overnight.

The feed flow rate was 4.7-5.7 L/minute (1.25-1.5 US gpm). The tests were as follows below, where the pressures are listed in consecutive order.

Day 1. 2.4 wt% ammonium nitrate at 37-43°C, and 790, 600, 400, 208 and 800 psig. Then pH was adjusted downward to 3.7, and readings were taken at 820 psig. Drain and fill up the system with DI water.

Day 4. 0.19 wt% ammonium nitrate at 25-46°C, and 221, 419, 625, 202 and 129 psig.

Day 5. Same feed solution as day 4 at 212 psig and 28°C. Then, ammonium nitrate was added to 0.6 wt%, and readings were taken at 37-50°C, and 835, 618, 405, 238, 150 and 800 psig.

Day 6. Same feed solution as day 5 at 250 psig and 27°C (reading was taken only 13 minutes after start). Then, changed to a fresh 1.3 wt% ammonium nitrate solution, and readings were taken at 42-52°C, and 850, 760, 600, 400, 195 and 850 psig.

Day 7. Same feed solution as day 6 at 411 psig and 25°C (reading was taken only 14 minutes after start). Then, ammonium nitrate was added to 2.8 wt%, and readings were taken at 40-51°C, and 910 (only 11 minutes from salt addition and at 32°C), 870, 800, 595, 400, 200 and 850 psig. Then, ammonium nitrate was added to 5.2 wt%, and readings were taken at 48-55°C, and 895 (only 9 minutes from salt addition), 910, 830, 705, 595 and 898 psig.

Day 8. Same feed solution as day 7 at 790 psig and 31°C (reading was taken only 19 minutes after start). Then, changed to a fresh 0.2 wt% ammonium nitrate solution, and readings were taken at 36-43°C, and 210 (reading was taken only 13 minutes after start and at 27°C), 690, 500, 300, 210 and 680 psig.

Day 11. Same feed solution as day 8 at 27-50°C, and 670 (reading was taken only 10 minutes after start, when the temperature was 27°C), 680 (39°C), 450 (40°C), 200 (38°C) and 640 (50°C) psig. Then the feed solution was DI water at 32-34°C at 650 and 200 psig, with readings taken 10-20 minutes after pressure change.