

Ultrafiltration Membrane Separation of Coffee

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Abstract

This experiment was performed as part of the TKP4105/TKP4110 felleslab course at NTNU. The properties of an ultrafiltration membrane, and core principles of filtration processes was examined. A solution of coffee was filtrated, and flux, permeability and permeate concentration was determined. The flux was found to be $18.64 \text{ [L m}^{-2} \text{ h}^{-1}]$. Permeability was found to be $9.32 \text{ [L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}]$. The concentration of the permeate was 51.1%.

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1 Theory

The theory section of this document is generally based on the instruction script for the experiment.¹

The goal of all filtration processes is to separate two or more substances from each other. Ultrafiltration, microfiltration, nanofiltration or gas separation follow the same fundamental principles, the main difference is the size of retained molecules. Ultrafiltration membranes will typically remove substances with high molecular weights. Because of this, ultrafiltration achieves high flux rates at low applied pressure.

A membrane is a semi-permeable structure, which can be used for separation as it will only allow compounds of a certain size through itself. By choosing a membrane that is permeable for only one compound in a solution, and impenetrable for other compounds, very high levels of separation can be achieved. Ultrafiltration membranes are usually porous with pore diameter between 10 to 100 nm. A more common categorization of membranes are molecular-weight cut-off (MWCO), which is defined as a molecular size of dextran being 90% rejected by membrane.

Filtration processes are usually pressure-driven for increased effectivity. In this experiment a dead-end filtration will be used, where the feed flow will be directed perpendicularly to the membrane. Pressure will be applied from the top of the cell. Among the most common problems with ultrafiltration are concentration polarization and fouling, as both of these phenomena lead to flux decline. Flux decrease may also be caused by increased osmotic pressure, formation of a gel layer, solute adsorption on the membrane or pore plugging.

1.1 Measurements and parameters

Flux is one of the most important parameters for characterizing membranes. Instantaneous flux is defined in (1.1),

$$J_v = \frac{1}{A} \frac{\Delta V}{\Delta t} \quad (1.1)$$

where V is the filtrate volume, t is the time of filtration and A the surface area of the membrane. J_v has the unit $\text{L m}^{-2} \text{h}^{-1}$. When comparing membranes of equal surface area, it is sufficient to discuss the throughput given in L h^{-1} .

Permeability L_p with respect to the solvent is given by (1.2),

$$L_p = \frac{J_v}{\Delta P} \quad (1.2)$$

where J_v is the volumetric filtration flux, and ΔP the transmembrane pressure driving force.

The ratio of solute concentration in the filtrate (c_{pi}) to the solute concentration in the feed solution (c_{fi}) is commonly referred to as S_i , and is given by (1.3)

$$S_i = \frac{c_{pi}}{c_{fi}} = 1 - R \quad (1.3)$$

where R is defined as the rejection coefficient. By rearranging (1.3), we get

$$R = 1 - \frac{c_{pi}}{c_{fi}} \quad (1.4)$$

R is the apparent rejection calculated from concentration of component i in the feed and permeate. The true membrane rejection is higher due to concentration changes in the boundary layer. The data for concentration in the boundary layer are however inaccessible.

2 Experimental

The cell was assembled according to section 3.2 in the lab script.¹ A photograph of the assembled cell can be found in figure 2.1. All membranes were properly cleaned with deionized water before the filtration. All filtrations in the experiment was performed with a feed volume of 200 mL, stirring at 150 rpm, and an applied pressure of 2.0 bar.

Deionized water was first filtrated through the membrane to find permeability and pure water throughput for the membrane. This was done two times so any decline in membrane performance could be determined.

A sample of 1 wt% coffee was prepared, and 200 mL of this was filtrated through the same membrane as the water. After approximately 3 minutes a sample of the permeate was taken by holding a small glass beaker under the permeate exit tube.

The concentration of the permeate was determined by measuring the samples ionic activity. A series of dilutions of the original coffee solution was prepared in order to establish a benchmark curve for different ionic activities as a function of relative coffee-concentration. The activity of the permeate sample was then measured and the relative concentration was determined by comparison to the benchmark.

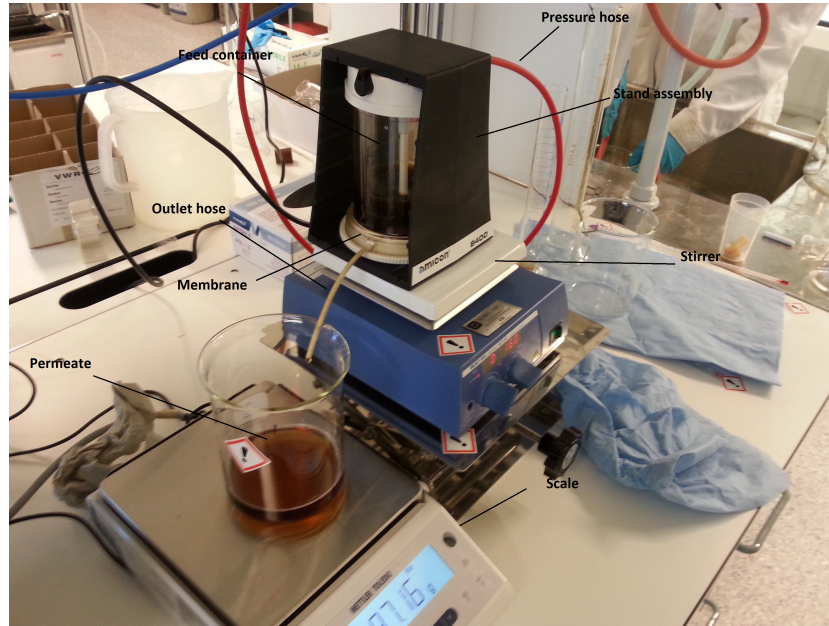


Figure 2.1: A photograph of the apparatus with vital components labeled.

3 Results

Flux and permeability for all filtrations was calculated using (1.1) and (1.2). Filtration area A was given as 41.8cm^2 .¹ The calculated values for flux and permeability can be found in table 3.1. For flux and permeability calculations it was assumed that the permeate from the coffee filtration had a density of 1 g cm^{-3} . An estimate for the density of the coffee solution can be found in equation (A.1).

Table 3.1: The table shows pure water flux and permeabilities for both filtrations of pure water.

Run #	ΔV [L]	Δt [h]	J_v [$\text{L m}^{-2} \text{h}^{-1}$]	L_p [$\text{L m}^{-2} \text{h}^{-1} \text{bar}^{-1}$]
1	0,19762	0,01758	2689,70	1344,85163
2	0,19735	0,01708	2763,55	1381,77412

An example calculation for J_v and L_p for the first filtration of pure water can be found in the equations (3.1) and (3.2), using (1.1) and (1.2).

$$J_v = \frac{0.19762\text{ L}}{0.00418\text{ m}^2 \cdot 0.01758\text{ h}} = 2689.70\text{ L m}^{-2} \text{h}^{-1} \quad (3.1)$$

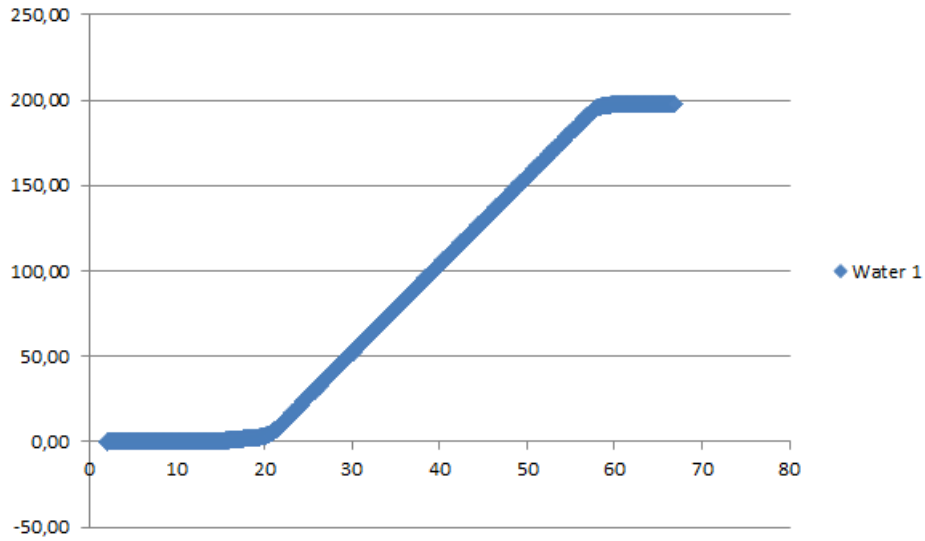


Figure 3.1: The figure shows a plot of volume against time for the first pure water filtration.

$$L_p = \frac{2689.70 \text{ L m}^{-2} \text{ h}^{-1}}{2.0 \text{ bar}} = 1344.85163 \text{ L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1} \quad (3.2)$$

Table 3.2: The table shows calculated flux and permeability for the coffee filtration, along with measured values for ΔV and Δt . It is assumed that the solution has a density of 1 g cm^{-3}

ΔV [L]	Δt [h]	J_v [$\text{L m}^{-2} \text{ h}^{-1}$]	L_p [$\text{L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}$]
0,1948	2,5004	18,6353	9,3176

The data in table 3.3 was plotted against each other to establish a benchmark curve with concentration as a function of activity. This plot can be found in figure 3.5. By using regression a second order polynom was found (3.3), and used to determine the concentration of the permeate.

$$y = 0.4X - 102.13X + 6529.1 \quad (3.3)$$

By inserting the measured activity of the permeate in (3.3), it was found that the permeate had a concentration of 51.1% relative to the original solution.

The rejection coefficient R is defined in (1.4). By applying the permeate

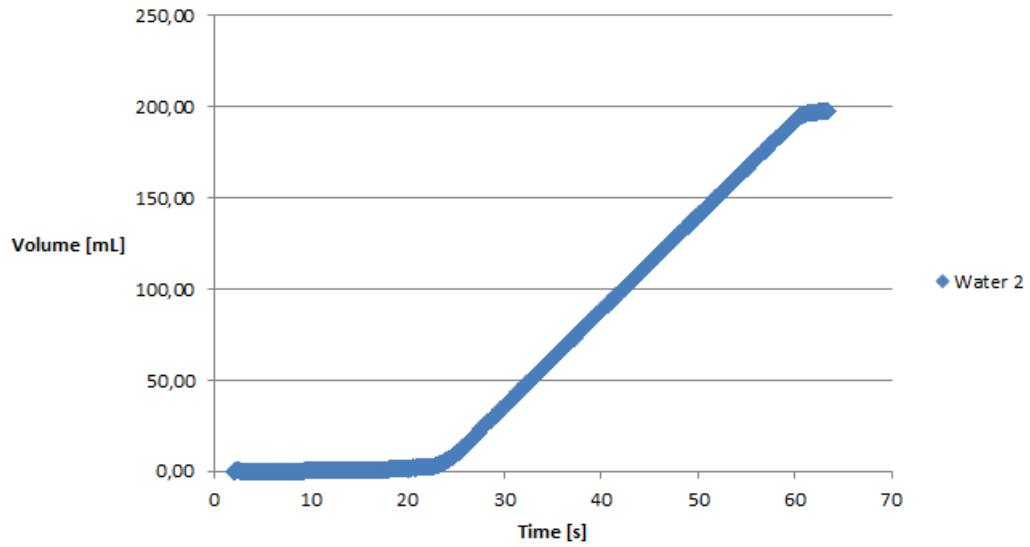


Figure 3.2: The figure shows a plot of volume against time for the second pure water filtration.

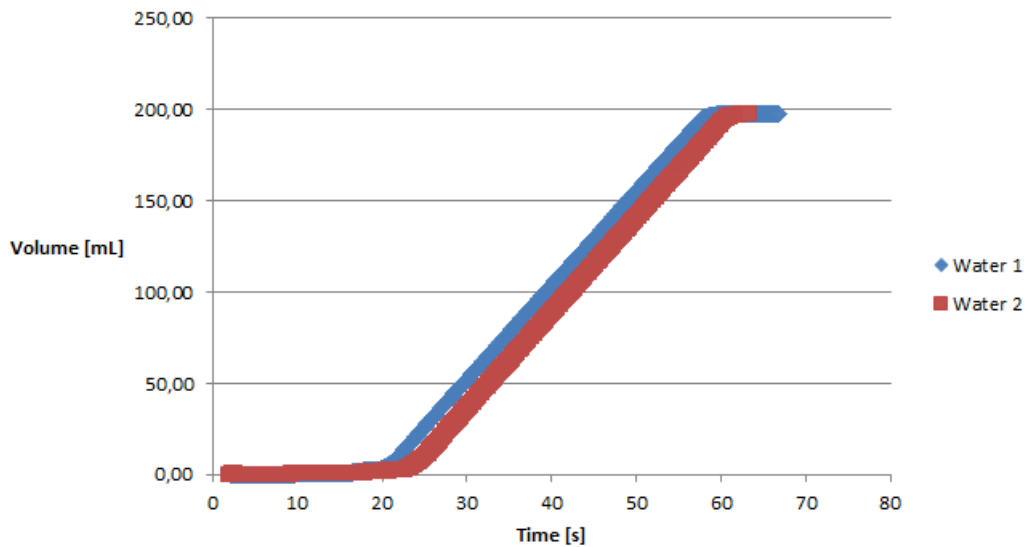


Figure 3.3: Comparison of the throughputs of the two filtrations with deionized water.

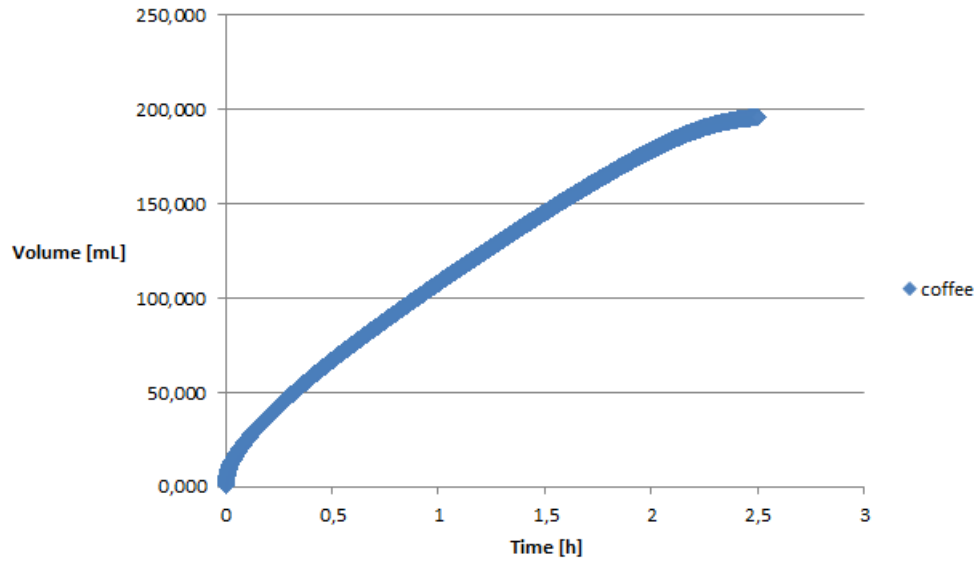


Figure 3.4: The figure shows the measured throughput of the coffee filtration as a function of time.

Table 3.3: The table shows the measured activity for the diluted coffee solutions. The concentration of the standards are given as percentages of the original solution to be filtered. The last entry is the measured activity of the permeability sample.

Activity [mV]	Concentration [%]
39,4	0
130,6	10
132,7	20
133,4	30
135,5	40
138,7	50
139	60
139,7	70
141,2	80
141,8	90
142	100
137,8	-

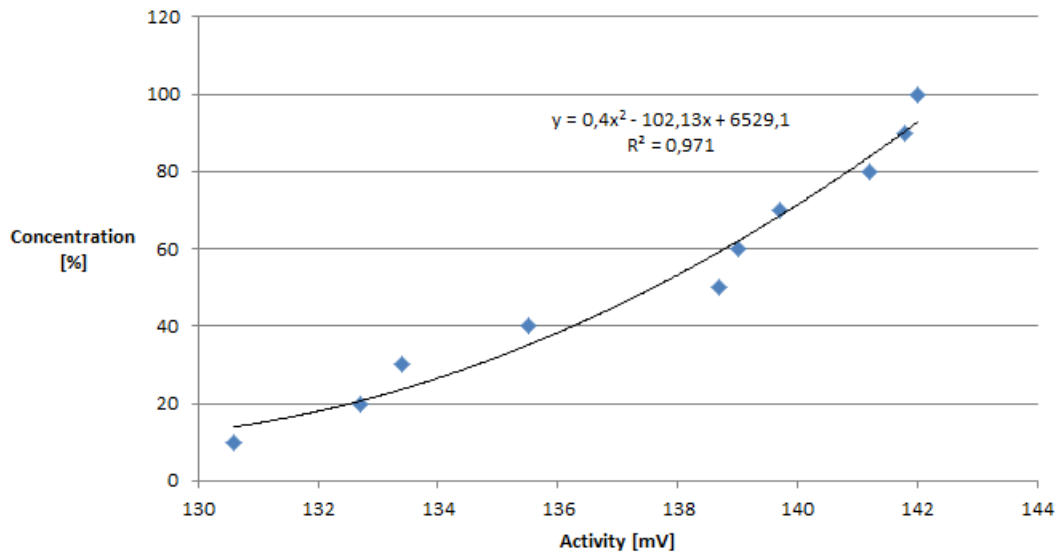


Figure 3.5: The figure shows a plot of relative concentration against activity for diluted coffee samples. A second order polynomial was found by regression. The regression curve is drawn in the plot.

concentration found from (3.3), The rejection coefficient is calculated:

$$R = 1 - \frac{51.1}{100} = 0.481 \quad (3.4)$$

4 Discussion

A comparison of throughputs of both water filtrations can be found in figure 3.3. In this plot, it is seen that the slope of both graphs are approximately the same. This is reflected in the calculations shown in table 3.1. These results indicate that the membrane performance does not decrease notably between the filtrations.

In order to calculate permeate volume, it was assumed that the density of the permeate was approximately the same as the density of pure water. Considering the estimation for the feed density made in (A.1) and that the concentration of coffee in the permeate is even lower, it seems unlikely that this assumption causes much error in the results.

The plot and regression curve which forms the basis for the permeate concentration are shown in figure 3.5. In this plot the point at $y = 50\%$ seems to deviate from the general trend, which can have affected the slope of the regression curve. This in turn may be cause for some error in the found

permeate concentration.

By comparing the colors of the standard solutions, made for the calibration curve 3.5, to the permeate sample, a rough estimate of concentration could be made. It was seen that the color of the permeate sample looked most like the 50 % sample.

5 Conclusion

The flux and permeability through the membrane for filtration of a 1 wt% solution of coffee was found as respectively $18.64 \text{ [L m}^{-2} \text{ h}^{-1}]$ and $9.32 \text{ [L m}^{-2} \text{ h}^{-1} \text{ bar}^{-1}]$. The concentration of the permeate was found to be 51.1% relative to the feed solution.

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References

- [1] *Felles Lab: Distillation Columns*, September, 2012, read 7th Oct. 2013. Available at http://www.nt.ntnu.no/users/preisig/Repository/TKP_4110_Felles_Lab/experiment%20descriptions/DistInstruct.pdf
- [2] Aylward, G. Findlay, T. *SI Chemical Data*, 6th ed.; John Wiley & Sons Ltd., 2008

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Symbol	Dimension	Description
A	m^2	Filtration area
c_{fi}	%	Concentration in the feed solution
c_{pi}	%	Concentration in the filtrate
J_v	g mol^{-1}	Volumetric filtration flux
L_p	mol	Permeability
R	Dimensionless	Rejection coefficient
S_i	Dimensionless	Solute ratio
ΔP	bar	Transmembrane pressure driving force
Δt	h	Filtration time
ΔV	L	Filtration volume

A Calculations

The 1 wt % coffee solution was prepared by diluting 2.5g freeze-dried coffee with 247.5g water. The final solutions mass was found to be 250.11g. The density of this solution can be estimated by (A.1)

$$\rho = \frac{250.11}{250} = 1.00044 \text{g cm}^{-3}. \quad (\text{A.1})$$