Felleslab ST-7
Ultrafiltration
Group B-16

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October 21, 2013

Abstract

Contents

1 Introduction

This experiment has been conducted in order to obtain an understanding of the ultrafiltration-membrane process. To do so, the reduction of permeability of diluted milk has been measured and compared to that of deionized water.

2 Theory

The theory in this section is based on the curriculum [2] written by Georg Voss available on professor Heinz Preisig's Felleslab web page.

In its most basic form, ultrafiltration is a pressure-driven process designed to remove solvent (typically water) and small solutes (e.g., salts and sugars) from larger macromolecules weighing between 10^3 and 10^6 Da (e.g. proteins). A membrane used in ultrafiltration typically has pores with a diameter of somewhere between 10 and 100 nm. More often, membranes are categorized by the term molecular-weight-cut-of (MWCO), defined as the molecular weight of dextran being 90% rejected by the membrane, given by the unit Da.

The two most common set ups for filtration are cross-flow and dead end filtration. The first set up is mainly used for industrially scaled processes, while the latter is more common in lab scale experiments. In this experiment only the dead end filtration technique will be used.

The flux is one of the most important characteristics of a membrane for filtration. Instantaneous flux is given by equation (2.1):

$$J = \frac{1}{A}\frac{\Delta V}{\Delta t} \tag{2.1}$$

In equation (2.1) A is the membrane surface area, V is the filtration volume and t is the filtration time. When comparing membranes with the same surface area it is sufficient to discuss the throughput of the membranes.

Also of interest in an ultrafiltration is the permeability L_p of the solvent:

$$L_p = \frac{J_v}{\Delta P} \tag{2.2}$$

In equation (2.2) J_v is the volumetric filtration flux and ΔP is the transmembrane pressure driving force. Because water is the typical solvent L_p is often referrers to as the hydrodynamic permeability. The

data are often normalized by the solvent viscosity to account for the effects of temperature. Many different units are used for filtration flux and permeability. In this experiment the unit for filtration flux will be [$\mathrm{L\,m^{-2}\,h^{-1}}$] and the unit for permeability will be [$\mathrm{L\,m^{-2}\,h^{-1}\,bar^{-1}}$].

The mass transport through the membrane is dominated by convection. The rate of mass transport for both the product and for small impurities is proportional to the filtration flux and the corresponding solute sieving coefficients, S_i. S_i is equal to the ratio between the solute concentration in the filtrate over that in the feed solution. If R is the rejection coefficient the sieving coefficient is equal to 1-R. The apparent rejection for component i can be calculated from equation (2.3):

$$S_i = \frac{c_{pi}}{c_{fi}} = 1 - R \implies R = 1 - \frac{c_{pi}}{c_{fi}} \tag{2.3}$$

The apparent rejection of component i is calculated from the concentration of i in the feed (c_{fi}) and in the permeate (c_{pi}). Due to concentration changes in the boundary layer between the membrane and the feed, the true membrane rejection is higher. Data for the boundary layer cannot be obtained, however.

3 Experimental

3.1 Experimental Setup

The dead-end filtration was carried out using a Stirred Ultrafiltration Cell Model 8400 from Millipore with a total volume of 400 mL and a circular membrane with a total area of 41.8 $\mathrm{cm^2}$. The feed side was connected to a container of pressurised air with a pressure gauge mounted on the tube leading the air into the apparatus. The mass of the filtrate was measured and logged with the corresponding time by a computer with Labview software connected to the scale. The filtration apparatus stood atop a magnet stirrer in order to keep the feed in constant motion and well mixed.

Since ultrafiltration membranes can operate with low pressures as driving force, the transmembrane pressure for this experiment was set to 1 bar by inducing pressure with pressurised air at 2 bar at the feed side. During each filtration the rotation frequency of the stirrer was set to 140-150 rpm, and approximately 200 mL of feed was poured into the apparatus.

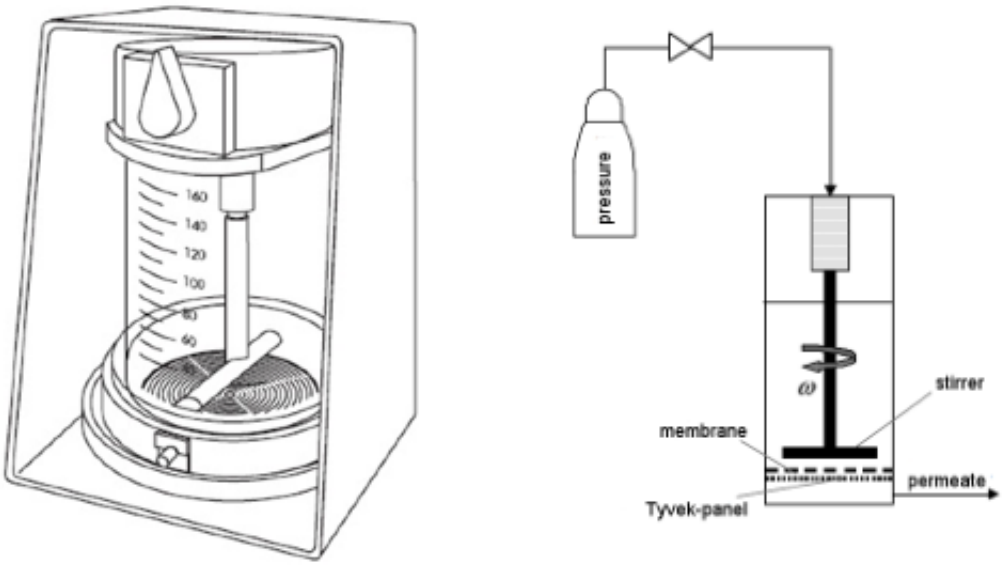

Figure 1: The apparatus used: A Stirred Ultrafiltration Cell Model 8400 from Millipore with a total volume of 400 mL and a circular membrane with a total area of 41.8 cm^2. Figure collected from the curriculum on the Felleslab web page [2].

3.2 Experimental procedure

Originally the experiment would have been performed using a membrane with a MWCO of 100 kDa, and three filtrations would be performed. To begin with, deionized water would be filtrated twice to examine wether there would be a decrease in the membrane's performance using a feed containing so few impurities, and then a solution consisting of milk diluted with water would have been filtrated for comparison. However, during the experimental procedure a few unexpected situations arose. Due to various reasons, the results attained with the 100 kDa MWCO membrane were deemed so highly dubious that it was decided to scrap the entire data collection and redo the experiment altogether with a different membrane, this time with a MCOW of 30 kDa.

When the experiment was restarted there was not much time left for the day and so the procedure had to be ended prematurely. There was enough time to perform both the filtrations with deionized water, but the data sampling from the filtration of the dilute milk had to be aborted after having time only to let slightly more than 10% of the feed to be filtrated.

3.3 Analysis

A portion of the filtrate was collected for analysis by UV/Vis-spectroscopy. As a basis for this analysis, ten solutions of milk diluted in water with concentrations as described in table 1 were prepared.

Table 1: Concentrations of milk diluted in water used for UV/Vis-spectroscopy.

Solution	Concentration [wt%]
1	0.065
2	0.26
3	0.38
4	0.65
5	0.85
6	1.37
7	1.8
8	2.0
9	5.1
10	9.6

4 Results

The amount of filtrate by mass as function of filtration time is plotted in figures 2 and 3. The permeate was almost colourless and did not resemble milk at all. By comparison to the prefabricated dilutions of milk in Table 1, it turned out to have a tint of light yellow/brown colour.

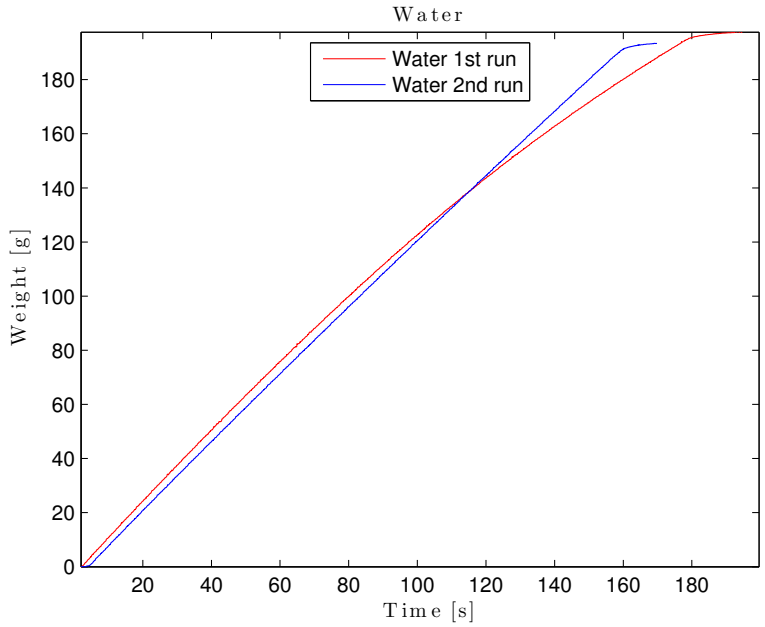

Figure 2: Plot of the amount of filtrate by mass as function of time during the filtrations of deionized water.

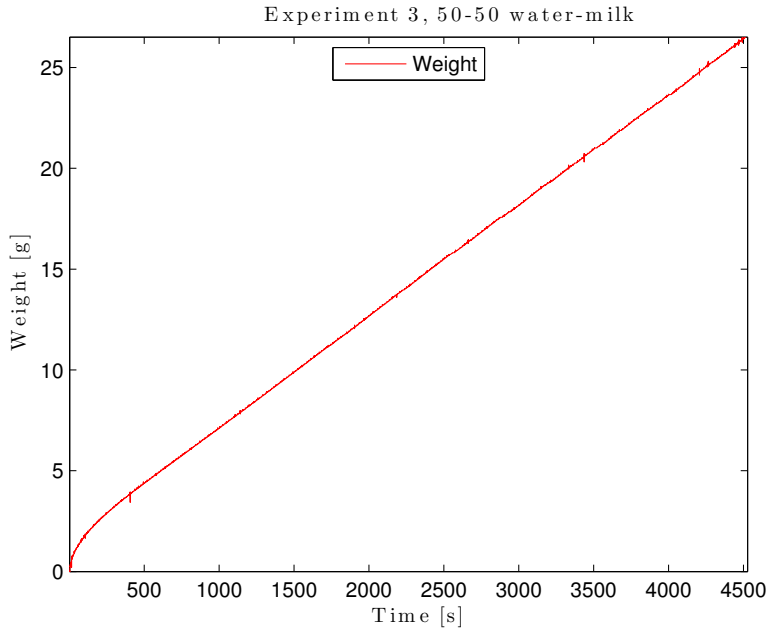

Figure 3: Plot of the amountof filtrate by mass as function of time during the filtration of a mixture of 50 wt% milk and 50 wt% water.

4.1 Flux and permeability

The flux and the permeability of the three filtration parallels were calculated using (2.1) for the flux and (2.2) for the permeability (Appendix A). The results are given in Table 2.

Table 2: Flux and permeability for the three filtration parallels. Note that the uncertainty in the permeability values is very high due to low precision in the pressure values.

Parallel	Flux $[\mathrm{mL\,s^{-1}\,cm^{-2}}]$	Permeability $[\mathrm{mL\,s^{-1}\,cm^{-2}\,bar^{-1}}]$
Water 1	0.27	0.14
Water 2	0.24	0.12
Water/Milk 50/50	$1.4 \cdot 10^{-3}$	$7.0 \cdot 10^{-4}$

It is important to note that the readings of the pressure used to calculate the permeability are unreliable. The pressure gauge normally read approximately 2 bar when pressure was applied, but it varied slightly throughout the filtrations. Rather than trying to constantly log the pressure value, it has been decided to set the value to 2 bar, which is a reasonable estimate of the mean value. However, this does mean that the calculated permeability values have within them a high degree of uncertainty and should be regarded merely as an indication of a trend.

4.2 UV/Vi-spectroscopy

5 Discussion

One thing that is very striking is that the flux through the membrane was higher the second time the deionized water was filtered through is than the first time. This was unexpected to say the least, as the membrane performance would normally be likely to sink after use. With deionized water, though, it would be natural to expect a so small change that the flux would turn out to be practically the same. What caused the flux to drop during the second filtration is not very clear, but it could simply be down to that pressure inside the container has been different. There was a breakage in the O-ring in the lid, and this could have caused the pressure to be lower the second time, resulting in a lower flux.

It is clear from Figure 3 that the mixture of 50% milk and 50% water went much slower through the membrane than the pure deionized water. This is a logical result since milk contains many big molecule substances, namely proteins, which would be likely to be held back by the membrane. The experiment on the diluted milk confirmed this, as a creamy white substance was detected on the membrane after the experimental procedure had been ended.

The light yellow/brown tint in the permeate of the 50/50-filtration is probably is a bit difficult to explain, but it is perhaps possible that it could be due to pollution from the coffee that many other groups have performed the same filtration experiment with. The scientific assistant at the lab claimed this to be impossible, but it is hard to imagine a different explanation. Is seems logical that some colour might have stuck from the coffee experiments, especially since coffee is known to leave colour in f.ex. thermoses.

6 Conclution

Trondheim, October 21, 2013

Signatures: _____ _____

Symbols

Symbol	[Unit]	Explaination
A	cm^2	Area of membrane
J	$\mathrm{mL\,cm^{-2}\,s^{-1}}$	Flux
$J_{50/50}$	$\mathrm{mL\,cm^{-2}\,s^{-1}}$	Flux during filtration with a mixture of 50% milk and 50% water
J_{W1}	$\mathrm{mL\,cm^{-2}\,s^{-1}}$	Flux during first filtration with deionized water
J_{W2}	$\mathrm{mL\,cm^{-2}\,s^{-1}}$	Flux during second filtration with deionized water
L_p	$\mathrm{mL\,cm^{-2}\,s^{-1}\,bar^{-1}}$	Permeability
$L_{\mathrm{p}50/50}$	$\mathrm{mL\,cm^{-2}\,s^{-1}\,bar^{-1}}$	Permeability of the dilute milk
$L_{\mathrm{p\ W1}}$	$\mathrm{mL\,cm^{-2}\,s^{-1}\,bar^{-1}}$	Permeability of the deionized water in the first filtration
$L_{\mathrm{p\ W2}}$	$\mathrm{mL\,cm^{-2}\,s^{-1}\,bar^{-1}}$	Permeability of the deionized water in the second filtration
ΔP	bar	Transmembrane pressure
Δt	s	Filtration time
ΔV	mL	Filtration volume

References

[1] Etternavn, Fornavn *Verktittel*, x. utgave, Forlag, Land, **år**

[2] Curriculum by Georg Voss on professor Preisig's web page about the Felleslab, downloaded 22. September 2013.
`http://www.nt.ntnu.no/users/preisig/Repository/TKP_4110_Felles_Lab/experiment%20descriptions/Membrane_Ultrafiltration_Script.pdf`

A Calculation of flux and permeability

Equations (2.1) and (2.2) were used to calculate the flux and permeability of the three filtrations.

$$J_{\mathrm{W1}} = \frac{1}{41.8\,\mathrm{cm}^2} \cdot \frac{197.5\,\mathrm{mL}}{193.2\,\mathrm{s}} = 0.2446\,\mathrm{mL\,s^{-1}\,cm^{-2}}$$

$$J_{\mathrm{W2}} = \frac{1}{41.8\,\mathrm{cm}^2} \cdot \frac{193.4\,\mathrm{mL}}{169.0\,\mathrm{s}} = 0.2737\,\mathrm{mL\,s^{-1}\,cm^{-2}}$$

$$J_{50/50} = \frac{1}{41.8\,\mathrm{cm}^2} \cdot \frac{26.51\,\mathrm{mL}}{4527\,\mathrm{s}} = 1.401 \cdot 10^{-3}\mathrm{mL\,s^{-1}\,cm^{-2}}$$

$$L_{\mathrm{p\ W1}} = \frac{0.2446\,\mathrm{mL\,s^{-1}\,cm^{-2}}}{2\,\mathrm{bar}} = 0.1223\,\mathrm{mL\,s^{-1}\,cm^{-2}\,bar^{-1}}$$

$$L_{\mathrm{p\ W2}} = \frac{0.2737\,\mathrm{mL\,s^{-1}\,cm^{-2}}}{2\,\mathrm{bar}} = 0.1369\,\mathrm{mL\,s^{-1}\,cm^{-2}\,bar^{-1}}$$

$$L_{\mathrm{p\ 50/50}} = \frac{1.401 \cdot 10^{-3}\mathrm{mL\,s^{-1}\,cm^{-2}}}{2\,\mathrm{bar}} = 7.005 \cdot 10^{-4}\mathrm{mL\,s^{-1}\,cm^{-2}\,bar^{-1}}$$