

# **BT4110 - Downstream Processing Laboratory**

A report on the 2<sup>nd</sup> experiment, Filtration  
Group 6

## **Group 6**

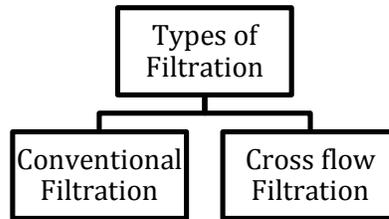
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## INTRODUCTION

Filtration is the process of separating suspended solid matter from a liquid, by causing the latter to pass through the pores of some substance, called a filter. The liquid which has passed through the filter is called the filtrate. The filter may be paper, cloth, cotton-wool, asbestos, slag- or glass-wool, unglazed earthenware, sand, or other porous material.



## CONVENTIONAL FILTRATION

### AIM & OBJECTIVE

To determine the specific cake resistance and the medium resistance

### THEORY

In conventional filtration, a solid suspension fluid, or filtrate, flows against a porous medium by application of a pressure gradient across the medium, where the solids in the suspension too large to pass through the medium become trapped on one side of the medium, building up in a layer called a cake, or filter cake. The flow of the liquid filtrate through the porous medium, which is a bed of solids, may be described by Darcy's Law written in the form:

$$\frac{1}{A} \frac{dV}{dt} = \frac{\Delta p}{\mu_0 R}$$

Where,

$A$  ( $m^2$ ) is the cross-sectional area of the medium through which the fluid flows,

$V$  ( $m^3$ ) is the volume of filtrate,  $t$  (sec) is the time,

$\Delta p$  (Pa) is the change in pressures across the medium and cake,

$\mu_0$  ( $kg/m*s$ ) is the is the viscosity of the filtrate,

$R$  ( $m^{-1}$ ) is the combined (in series) resistance of the filter medium (" $R_m$ ") and filter cake (" $R_c$ "), which is:

$$R = R_m + R_c.$$

The cake resistance may further be described in terms of the specific cake resistance,  $\alpha$  ( $m/kg$ ), in the following form:

$$R_c = \alpha \rho_c \left( \frac{V}{A} \right)$$

Where,  $\rho_c$  ( $kg/m^3$ ) is the mass of dry cake solids per unit volume of filtrate.

Darcy's Law for the flow of the liquid filtrate through the bed of solids porous filter medium and cake may therefore be rewritten as:

$$\frac{1}{A} \frac{dV}{dt} = \frac{\Delta p}{\mu_0 \left( \alpha \rho_c \left( \frac{V}{A} \right) + R_m \right)}$$

For constant pressure operation, this equation may be integrated with an initial condition of zero filtrate at time zero to yield:

$$\frac{t}{V} = \frac{\mu_0 \alpha \rho_c}{2 \Delta p} \left( \frac{V}{A} \right) + \frac{\mu_0 R_m}{\Delta p}$$

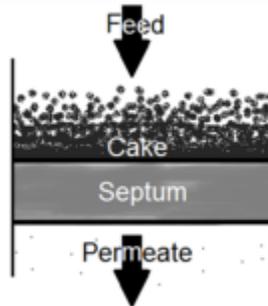


Figure A<sup>1</sup>: The process of conventional flow.

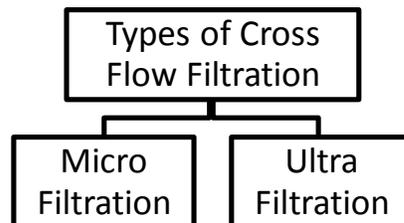
## CROSS FLOW FILTRATION: ULTRA FILTRATION

### AIM & OBJECTIVE

To determine the resistances due to the membrane (medium) and due to the cake.

### THEORY

In cross flow filtration, an incoming feed stream passes across the surface of a cross flow membrane, and two exiting streams are generated. The permeate stream is the portion of the fluid that passes through the membrane. This filtered fluid will contain some percentage of soluble and/or insoluble components from the initial feed stream that are smaller than the membrane removal rating. The remainder of the feed stream, which does not pass through the cross flow membrane, is known as the retentate stream.



<sup>1</sup> <http://en.citizendium.org/wiki/Filtration>

The principle of micro filtration and ultra-filtration is physical separation. The extent to which dissolved solids, turbidity and microorganisms are removed is determined by the size of the pores in the membranes. Substances that are larger than the pores in the membranes are fully removed. Substances that are smaller than the pores of the membranes are partially removed, depending on the construction of a refuse layer on the membrane. Micro filtration and ultra-filtration are pressure-dependent processes, which remove dissolved solids and other substances from water to a lesser extent than nano filtration and reverse osmosis. Membranes with a pore size of 0.1 – 10  $\mu\text{m}$  perform micro filtration. The pores of ultra-filtration membranes can remove particles of 0.001–0.1 $\mu\text{m}$  from fluids.

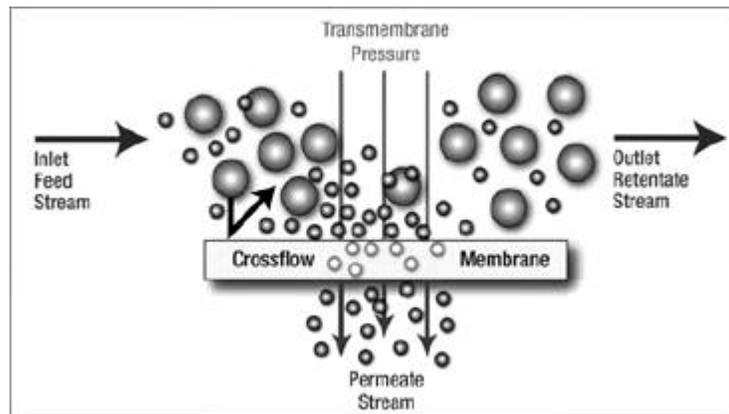


Figure B<sup>2</sup>: The process of cross flow filtration

## SESSION I

### Methodology

The protocol was followed as given in Lab Manual (G.K Suraish Kumar *et al.*)

### Results and Discussions:

#### Conventional Filtration:

We have used the parameters as:

$$1 \text{ psi} = 6894.75 \text{ Pa}$$

$$\mu = 8.90 \times 10^{-4} \text{ Pa.s}$$

Diameter of filter paper = 11.6cm

Weight of the dry membrane was found to be 0.62g. The cake was very well formed as observed in Figure 1.2.

<sup>2</sup> <http://www.pall.com/main/graphic-arts/how-crossflow-filtration-works.page>



Figure 1.2: Membrane with formed cake layer

The weight of membrane along with cake after drying in oven was found to be 2.82g. Hence, the weight of the cake was obtained as  $(2.82-0.62) \text{ g} = 2.2\text{g}$ .

The graph between  $At/V$  vs.  $V/A$  was plotted to calculate the value of specific cake resistance and filter resistance. The data was obtained as shown in Table 1.1 (refer appendix). Figure 1.1 is the required graph with R-square value of 0.9997. The slope and intercept of the curve is found to be  $34.8819 \text{ s/m}^2$  and  $0.1801 \text{ s/m}$  respectively.

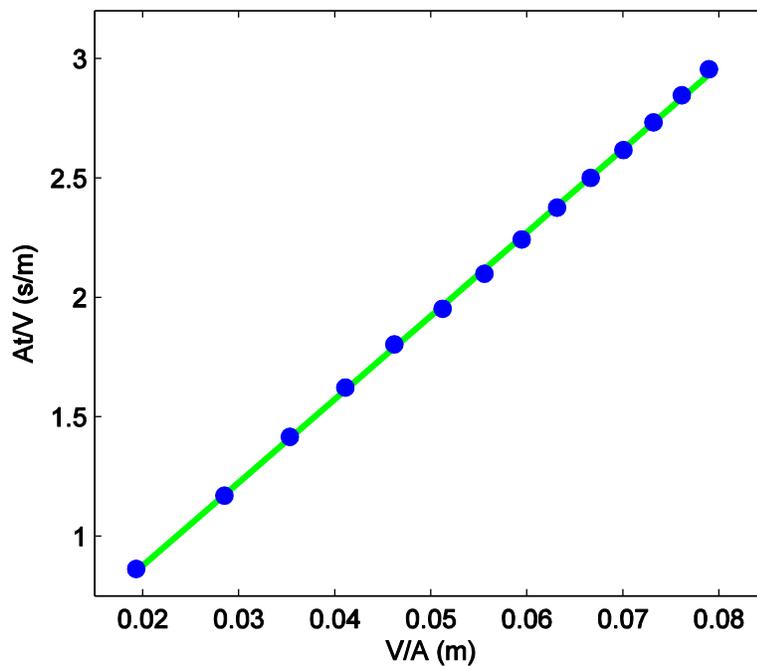


Figure 1.1: Graph between  $At/V$  vs.  $V/A$ .

Using the above graph and above described parameters, the value of  $R_m$  and  $R_c$  was calculated using the formula explained earlier (refer theory of conventional filtration).

$$R_m = 22.32 \mu\text{m}^{-1}$$

$$R_c = 686.84 \mu\text{m}^{-1}$$

The value of  $R_c$  was found to be much higher than  $R_m$  which was in accordance with the results obtained.

#### Cross-flow Filtration:

The absorbance values for the standard curve were taken in triplicate as shown in Table 1.2 (refer appendix) and the standard curve was plotted. The standard curve (Figure 1.3) was found to have an  $R^2$  value of 0.9984 and slope as 0.5688 l/g.

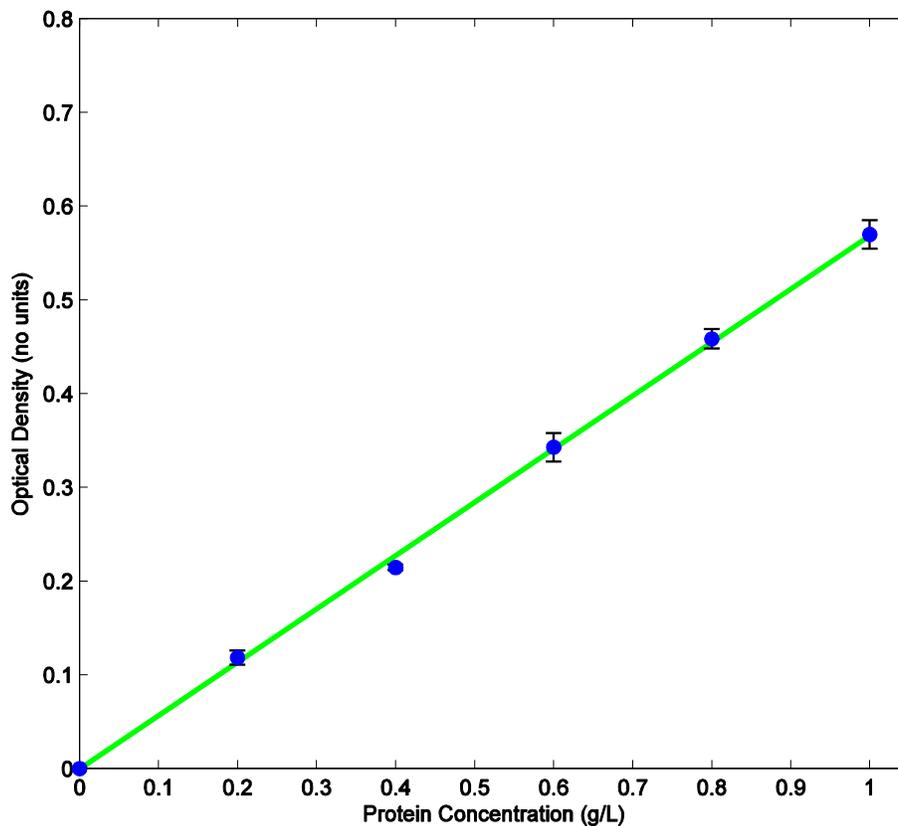


Figure 1.3: Standard Curve using known BSA concentration

Table 1.1 (a) & Table 1.1 (b) along with the data supplied in appendix (Table 1.3 & Table 1.4) were used to calculate the values of average flow rate with change in pressure. These data were used to plot curves (Figure 1.4 & Figure 1.5) for getting filter medium resistance and cake resistance.

Change in pressure (psi)	Volume (ml)	Flow Rate 1	Flow Rate 2	Average Flow rate (ml/sec)	Standard Deviation	Standard Error
10	5	0.041	0.034	0.037	0.0050	0.0035
20	5	0.074	0.065	0.069	0.0061	0.0043
30	5	0.109	0.102	0.105	0.0047	0.0033

Table 1.1 (a): Data used to calculate value of Rm.

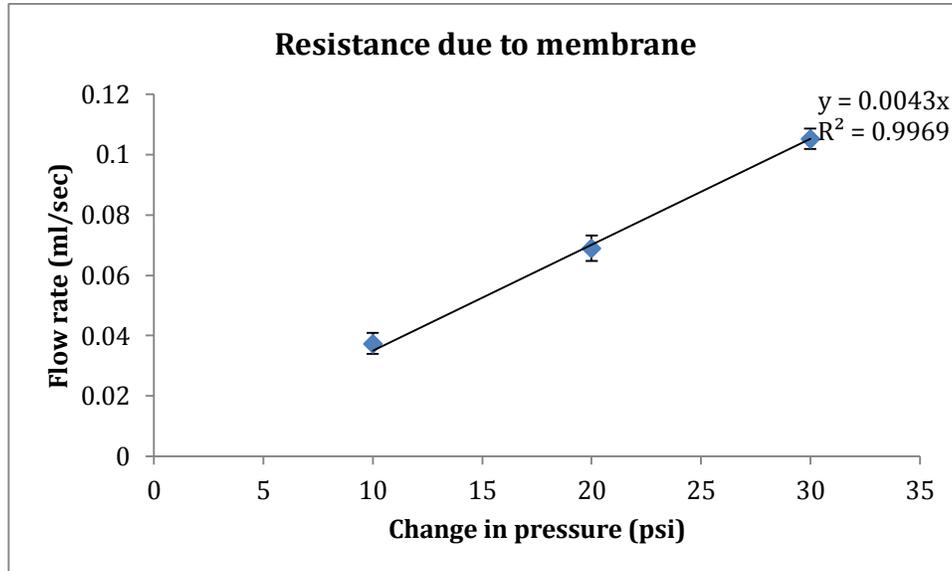


Figure 1.4: Curve used to calculate filter membrane resistance

Change in pressure	Volume (ml/sec)	Flow Rate 1	Flow Rate 2	Flow Rate 3	Flow Rate 4	Average Flow rate (ml/sec)	Standard Deviation	Standard Error
10	5	0.0521	0.044	0.0495	0.0424	0.047	0.0046	0.0023
20	5	0.0893	0.083	0.0893	0.0806	0.085	0.0044	0.0022
30	5	0.1389	0.119	0.1282	0.125	0.127	0.0083	0.0042

Table 1.1 (b): Data used to calculate value of Rm + Rc.

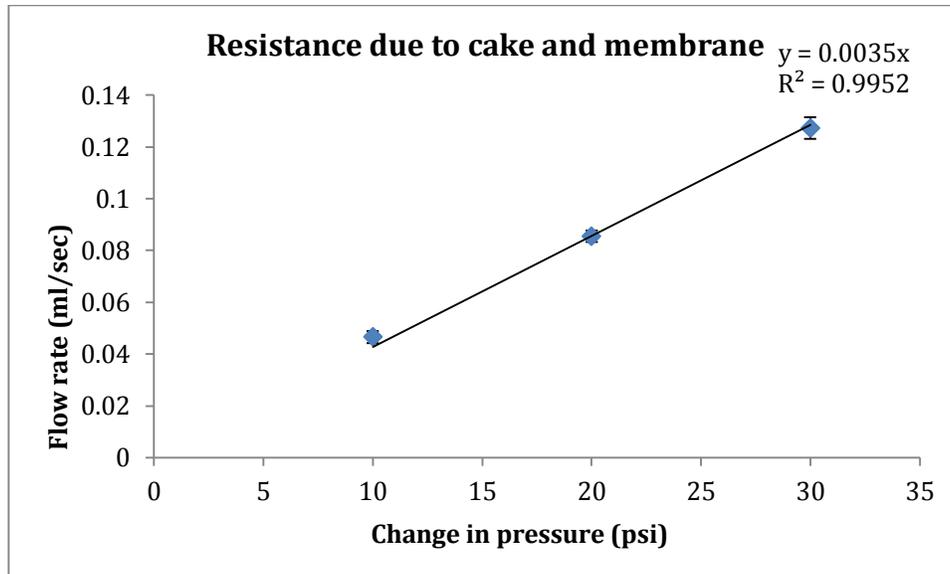


Figure 1.5: Curve used to calculate  $R_m + R_c$ .

The value of  $R_m$  and  $R_m + R_c$  was calculated using Figure 1.4 & 1.5. The area of the hollow cylinder in the case of cross-filtration is taken as  $A \text{ m}^2$ .

$$\text{Here, } \frac{R_m}{A} = 0.261 \text{ ml}^{-1}.$$

$$\frac{(R_m + R_c)}{A} = 0.321 \text{ ml}^{-1}$$

$$\frac{R_c}{A} = 0.06 \text{ ml}^{-1}$$

As expected, the value of  $R_c/A$  was comparatively lesser than the value of  $R_m/A$ . This suggested that there was a very little formation of cake due to the tangential flow of the filtrate. From Table 1.4 (refer appendix), it was also observed that at constant pressure there is not much change in flow rate suggesting lesser amount of cake formation and the ability of the process to last for longer duration. At 10 psi (Table 1.4), the amount of BSA recovered was calculated by allowing 35ml of filtrate to pass leaving out most of the bigger size (66.5kDa) BSA in 15ml retentate. Initial BSA concentration fed was 0.5g/l. The absorbance value obtained after filtration through 0.2 $\mu\text{m}$  membrane was calculated. From remaining 15ml, a small amount of sample was collected and its absorbance at 280nm was measured. This was done because, it was expected that BSA due to its bigger size will be left as retentate and wouldn't be present in filtrate. The absorbance was found to be 0.717 (taken in triplicate). Thus the concentration of protein was  $0.717/0.5688$  (slope was found from Figure 1.3) = 1.26g/l.

Now, the initial amount of BSA in 50ml of water =  $0.5 \text{ g/l} * 0.051 = 0.025 \text{ g}$

Final amount of BSA in 15ml of solution =  $0.015 * 1.26 = 0.0189 \text{ g}$

Hence, recovery of BSA =  $0.0189/0.025 = 0.756 \text{ g/g}$

Percentage recovery of BSA in the retentate was found to be 75.6%. Due to bigger size of BSA, most of it stayed in the retentate stream while some of them contributed in formation of the cake.

Thus, we were able to calculate the values of filter membrane resistance as well as cake resistance using both conventional and cross-flow filtration.

Conventional filtration, on one hand, is best when filtering micro-organisms, other contaminants like organic matter etc. Most of the contaminants are negatively charged, hence adding a coagulating agent (positively charged) can further enhance efficiency of the process. On other hand, apart from filtering contaminants, cross-flow filtration can be best employed while separating proteins of different sizes. Multiple size proteins can be separated depending on the size of filter used in multiple steps. Thus, cross-flow filtration, in particular finds a wide application in downstream processing of proteins.

While both of the methods find wide applications in various fields, but for most of the large scale purposes, we tend to use cross-flow or tangential filtration. This is because it offers an additional advantage of being more of a continuous process, unlike conventional filtration. In case of conventional filtration, there is continuous accumulation of filter cake because of which, its efficiency to run for longer time decreases, and makes it tedious and less profitable process on large scale. In case of cross-filtration the filter cake is substantially washed away during the process of filtration which leads to its longevity in operation.

Keeping the above disadvantage offered by conventional process in mind, we have tried to make it more of an efficient process in our next session. Along with that we have also provided a practical use of conventional filtration in day-to-day life.

## **SESSION II**

We have divided this session in two parts where we have tried to work on two important modules of conventional filtration i.e. efficiency and application.

### **PART 1:**

#### **Aim:**

*An approach to make conventional filtration more efficient process.*

#### **Approach:**

We wished to recalculate the amount of resistance ( $R_m$ ) offered by the filter paper we used during the first session, using a simpler procedure. During session 1, we used the design

equation for cake filters to find  $R_m$ . This entailed performing a very dynamic experiment where there were many avenues for error. E.g. the cake could have formed asymmetrically. Thus, for this part of session 2, we decided to directly use Darcy's law (eq. 1) to calculate  $R_m$ . In other words, we did not pass a suspension through the equipment. We used distilled water instead.

$$Q = \frac{\Delta P}{\mu R} \dots\dots\dots (1)$$

where,

- $Q$  = flow rate
- $\Delta P$  = pressure difference
- $\mu$  = viscosity of solution
- $R$  = resistance of membrane

The basic idea that our experiment hinged on is that when two exactly similar filter papers are placed on top of one another, the net resistance offered by both of them is double that offered by one of them. We decided to use this phenomenon to calculate the value of  $R_m$ . Instead of taking measurements of flow rate using a single filter paper, we would take measurements using different numbers of filter papers while keeping the pressure difference constant. Then, from the plot of flow rate versus the number of filter papers used, we would find the value of  $R_m$ . We modified the equation given as Darcy's Law to suit our purpose:

$$Q = \frac{\Delta P}{\mu(R_b + nR_m)} \dots\dots\dots (2)$$

Where,

- $R_b$  = Resistance offered by the equipment
- $n$  = Number of filter papers
- $R_m$  = Resistance offered by the membrane

**Procedure**

The same procedure for the conventional method that was used during session 1 was used. In this session, for each measurement of flow rate, we allowed distilled water to pass through the set up for 5 seconds.

**Results & Discussion**

$\Delta P = 2 \text{ psi} = 13,789.515 \text{ Pa}$   
 $\mu = 8.90 \times 10^{-4} \text{ Pa.s}$

N	Volume1 (mL)	Volume2 (mL)	Volume3 (mL)	Average Vol. (mL)	Q (mL/s)
0	226	265	250	247	49.4
1	70	98	55	74.33	14.86
2	13.5	15	13	13.83	2.766
3	13	18	22.5	17.83	3.566
4	5	7	6.5	6.166	1.233

Table 2: Data showing change in flow rate with number of filter papers

Using MATLAB, the above data points were fit to eq. 2.

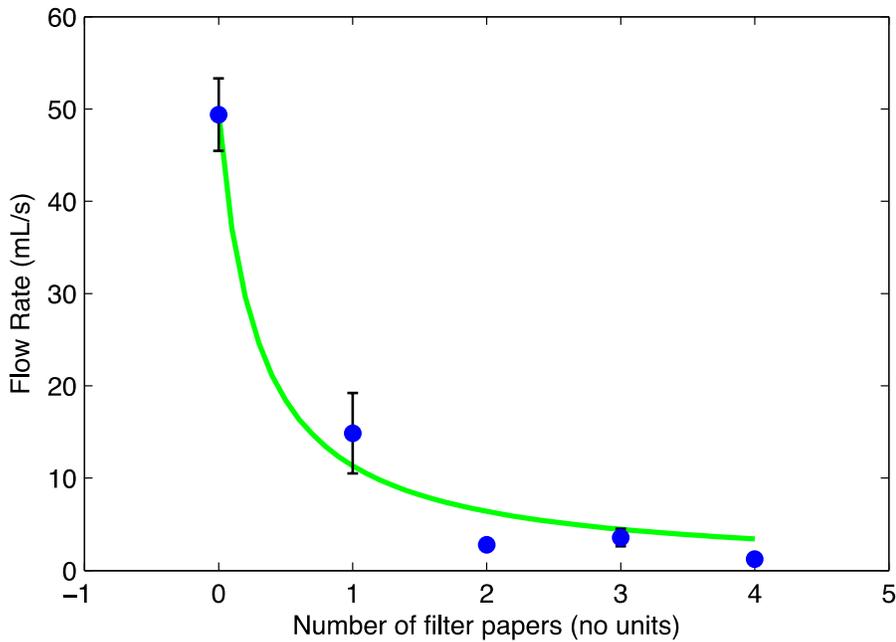


Figure 2.1: Curve b/n flow rate vs. number of filter papers

R-square value of the fit: 0.9834

$R_b = 313 \text{ (mL)}^{-1}$ ,  $R_m = 1053.7 \text{ (mL)}^{-1}$  was calculated using MATLAB.

Hence, we were able to successfully calculate the values of  $R_b$  and  $R_m$  (Figure 2.1).

The major thing we achieved by following our modified procedure is calculate the amount of resistance offered by a single filter paper. In the previous experiments we calculated the resistance of the filter paper combined with that offered by the equipment.

Our approach of exclusively finding the correct filter resistance finds application in designing membrane area for particular application based on temperature, membrane material, or other site- or system-specific factors (Steve Allgeier et al.). In the above applications, cake resistance is not taken into consideration while designing, hence it is totally dependent on the value of constant filter resistance provided by the manufacturer.

Thus, our process can be employed by membrane manufacturer to calculate the exact value of membrane filter resistance.

## **PART 2:**

### **Aim:**

*To test the purity of water from various sources inside Institute (IIT Madras) using conventional filtration.*

### **Procedure**

The same procedure for the conventional method that was used during session 1 was used.

### **Approach:**

Water was collected from different part of institute to test the amount of contaminants present and provide a comparative study at various levels.

Our experiment tested the amount of contaminants present at four different levels:

1. Tap water of hostels: It is mainly used for brushing teeth, bathing and other purposes.
2. Residential zone water: It is mainly used for dish washing, cloth washing, brushing teeth and other house hold purposes.



Figure 2.2: Water used for dish washing

3. Pump house water: It is used as drinking water by the people residing in residential zone.
4. Lake water: It was primarily taken to compare the amount of cake formed with that of water taken from other sources. Also, the presence of high amount of contaminants can be dangerous for the people residing nearby because of being prone to various water-borne diseases. Lake water was aseptically collected in bottle (Figure 2.3) for further use.



Figure 2.3: Place near Institute Stadium where Lake Water was collected

### Results and Discussions:

After collecting water from different places, we compared the amount of cake formed. We found that the amount of cake formed differed with different water.



Figure 2.4: Cake obtained with Hostel tap water

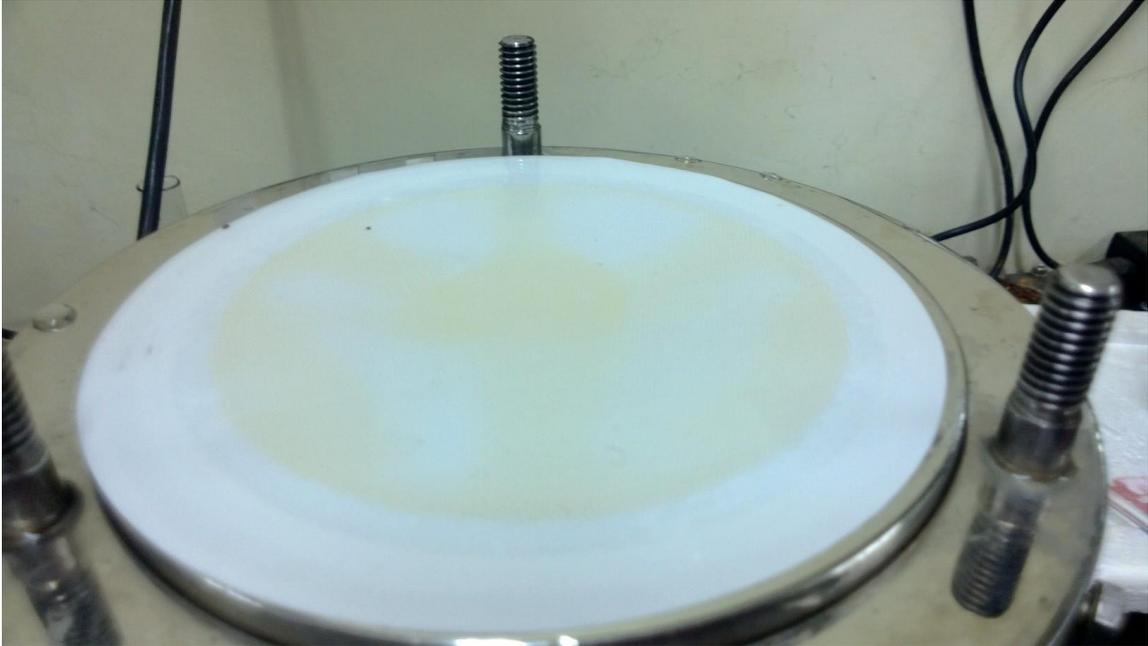


Figure 2.5: Cake obtained with water from Pump House



Figure 2.6: Cake obtained from residential zone water



Figure 2.7: Cake obtained from lake water

We observed that the cake formed by lake water (Figure 2.7) had thickest layer. The cake obtained by pump water (Figure 2.5) was less thicker as compared with the cake obtained by residential zone water (Figure 2.6) and hostel tap water (Figure 2.4) respectively. The hostel tap water and residential zone water showed almost identical pattern and similar intense cake was formed. While that of residential zone and pump house water showed a good all round distribution of cake, lake water and hostel tap water showed a round patches in the middle along with the distribution all around. This can be because of improper placing of the filter membrane. In future, care must be taken while placing and properly wiping of the membrane should be done for avoiding any unwanted bubble.

Despite the improper distribution of cake, it doesn't affected our experiments in this case and we were able to perform the comparative study for the four cases.

Along with this, the dry weights of the membrane along with the cake was compared.

The following observations were made:

- Weight of the membrane along with cake for hostel tap water (Figure 2.4) = 0.62g
- Weight of the membrane along with cake for pump house water (Figure 2.5) = 0.6g
- Weight of the membrane along with cake for residential zone (Figure 2.6) = 0.622g
- Weight of the membrane along with cake for lake water (Figure 2.7) = 0.644g

This was similar to what we observed from figure 2.4-2.7. It also suggested that the water which is used in hostel zone and residential zone had almost equal amount of contamination. As expected, lake water had maximum amount of cake formation while that of drinking water had the least. This indicated that pump house is least contaminated and indeed good for drinking.

Taking a closer look, we realized that the amount of cake formed for drinking water can still be dangerous for health. Apart from that, water used for dish cleaning and ultimately usage of dishes while eating is indirectly affecting us. Thus, further treatment of water need to be done before supplying to residential zone or hostel zone.

On literature survey (Ahsan Munir et al., Steve Allgeier et al.), we found one of the approach which can be employed to further treat the water is based on conventional filtration. A combination of coagulation, sedimentation, filtration and disinfection can be used as depicted in Figure 2.8.

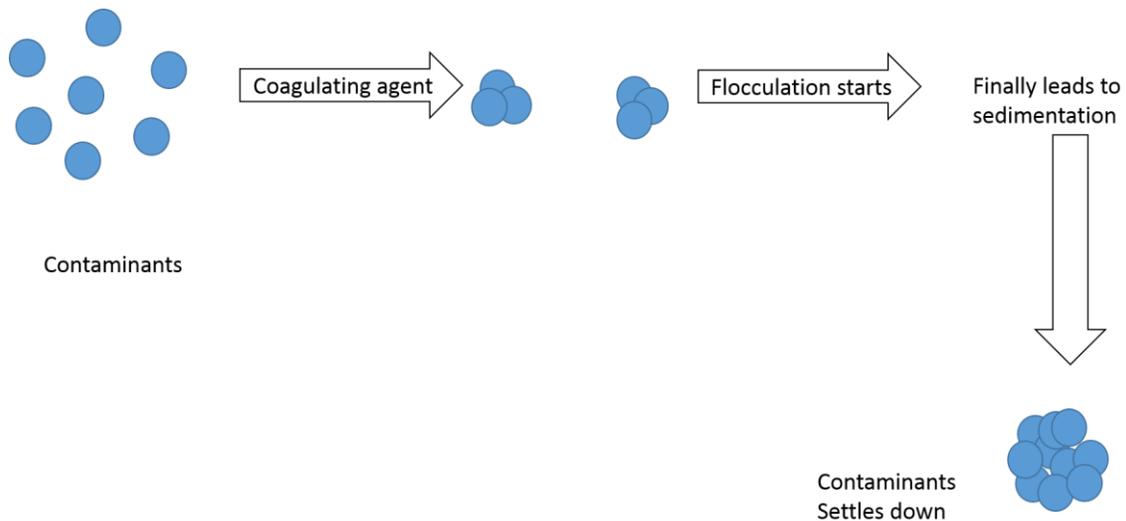


Figure 2.8: Flow chart explaining water treatment

Most of the contaminants are negatively charged, thus generally a positively charged coagulating agent is added which interacts with the contaminants present in the water. Due to its weight, the contaminant along with the coagulating agent settles down and finally it can be filtered to remove the sediment particles (Figure 2.8). The particles based on their sizes can be removed through various filtration methods with different filter pore size. One of the filtration method is slow sand filtration which removes contaminants as small as bacteria, protozoa and viruses. The water can be further disinfected as a precautionary measure and to finally get safe drinking water.

Thus, a comparative study was successfully performed for water from different sources. Our results demand a quick attention in this regard.

Altogether, we were able to improve the conventional flow technique as well as provide a practical application for the benefit of Institute and its residents.

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## APPENDIX

Time (min)	Volume (ml)
1	204
2	97
3	72
4	61
5	54
6	53
7	46
8	41
9	39
10	37
11	36
12	33
13	31
14	30

Table 1.1: Data obtained using conventional filtration

Conc (g/l)	Absorption @ 280nm			
	OD1	OD2	OD3	Average
0.2	0.117	0.12	0.118	0.118
0.4	0.215	0.214	0.214	0.214
0.6	0.34	0.346	0.342	0.343
0.8	0.456	0.459	0.46	0.458
1	0.573	0.567	0.569	0.570

Table 1.2: Standard Curve Data

Change(p)	Time (in sec)			Volume (ml)	Flow rate (ml/sec)
	Reading 1	Reading 2	Average		
10	121	146	133.5	5	0.0374
20	68	77	72.5	5	0.0689
30	46	49	47.5	5	0.1052

Table 1.3: Data obtained for calculating filter resistance

Volume (ml)	Time (sec)			Flow Rate (ml/sec)
	Reading 1	Reading 2	Average	
5	138	125	131.5	0.0380
10	263	252	257.5	0.0388
15	388	375	381.5	0.0393
20	518	500	509	0.0392
25	628	611	619.5	0.0403
30	752	745	748.5	0.0400
35	878	875	876.5	0.0399

Table 1.4: Flow rate data obtained at 10psi

Change (p)	Time (sec)					Volume (ml/sec)	Flow rate (ml/sec)
	Reading 1	Reading 2	Reading 3	Reading 4	Average		
10	96	114	101	118	107.25	5	0.0466
20	56	60	56	62	58.5	5	0.0854
30	36	42	39	40	39.25	5	0.1273

Table 1.5: Data obtained to calculate cake resistance