Dead End Membrane Filtration

ENE 806

Laboratory Feasibility Studies in Environmental Engineering

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Report by

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Ahsan Munir

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I. Introduction

Membrane filtration can be a very efficient and economical way of separating components that are suspended or dissolved in a liquid. The membrane is a physical barrier that allows certain compounds to pass through, depending on their physical and/or chemical properties. Membranes commonly consist of a porous support layer with a thin dense layer on top that forms the actual membrane. Types of Membrane filtration based on membrane pore sizes are described below;

1.1 Types of Membrane Processes

Ultrafiltration

Ultrafiltration (UF) is the process of separating extremely small particles and dissolved molecules from fluids. The primary basis for separation is molecular size, although in all filtration applications, the permeability of a filter medium can be affected by the chemical, molecular or electrostatic properties of the sample. Ultra filtration can only separate molecules which differ by at least an order of magnitude in size. Molecules of similar size can not be separated by ultra filtration. Materials ranging in size from 1K to 1000K molecular weight (MW) are retained by certain ultrafiltration membranes, while salts and water will pass through. Colloidal and particulate matter can also be retained. Ultrafiltration membranes can be used both to purify material passing through the filter and also to collect material retained by the filter. Materials significantly smaller than the pore size rating pass through the filter and can be depyrogenated, clarified and separated from high molecular weight contaminants. Materials larger than the pore size rating are retained by the filter and can be concentrated or separated from low molecular weight contaminants. Ultrafiltration is typically used to separate proteins from buffer components for buffer exchange, desalting, or concentration. Ultrafilters are also ideal for removal or exchange of sugars, non-aqueous solvents, the separation of free from protein-bound ligands, the removal of materials of low molecular weight, or the rapid change of ionic and/or pH environment (see Figure 1.1). Depending on the protein to be retained, the most frequently used membranes have a nominal molecular weight limit (NMWL) of 3 kDa to 100 kDa. Ultrafiltration is far gentler to solutes than processes such as

precipitation. UF is more efficient because it can simultaneously concentrate and desalt solutes. It does not require a phase change, which often denatures labile species, and UF can be performed either at room temperature or in a cold room.

Microfiltration

Micro filtration (MF) is the process of removing particles or biological entities in the 0.025 μm to 10.0μm range from fluids by passage through a microporous medium such as a membrane filter. Although micron-sized particles can be removed by use of non-membrane or depth materials such as those found in fibrous media, only a membrane filter having a precisely defined pore size can ensure quantitative retention. Membrane filters can be used for final filtration or prefiltration, whereas a depth filter is generally used in clarifying applications where quantitative retention is not required or as a prefilter to prolong the life of a downstream membrane. Membrane and depth filters offer certain advantages and limitations. They can complement each other when used together in a microfiltration process system or fabricated device.The retention boundary defined by a membrane filter can also be used as an analytical tool to validate the integrity and efficiency of a system. For example, in addition to clarifying or sterilizing filtration, fluids containing bacteria can be filtered to trap the microorganisms on the membrane surface for subsequent culture and analysis. Microfiltration can also be used in sample preparation to remove intact cells and some cell debris from the lysate. Membrane pore size cut-offs used for these types of separation are typically in the range of 0.05 μm to 1.0 μm.

Reverse Osmosis

Reverse osmosis (RO) separates salts and small molecules from low molecular weight solutes (typically less than 100 daltons) at relatively high pressures using membranes with NMWLs of 1 kDa or lower. RO membranes are normally rated by their retention of sodium chloride while ultrafiltration membranes are characterized according to the molecular weight of retained solutes. Millipore water purification systems employ both reverse osmosis membranes as well as ultrafiltration membranes. Reverse osmosis systems are primarily used to purify tap water to purities that exceed distilled water

quality. Ultrafiltration systems ensure that ultrapure water is free from endotoxins as well as nucleases for critical biological research.
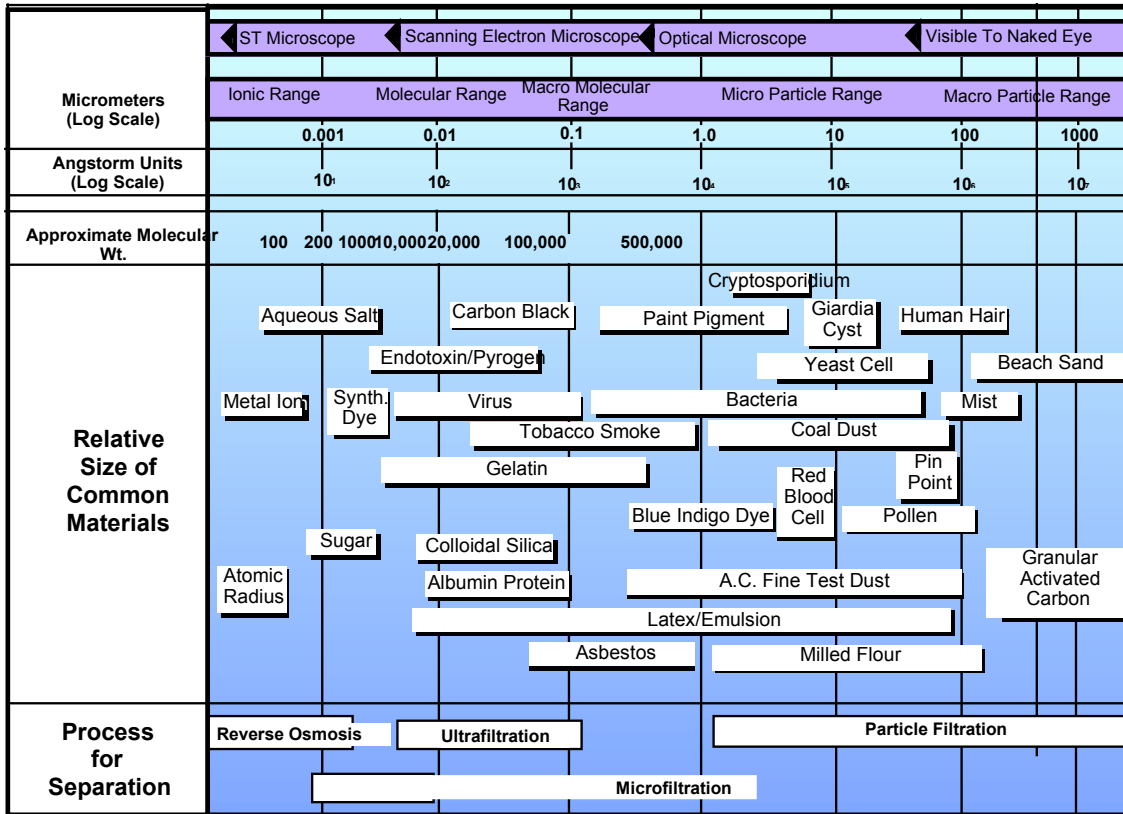
	ST Microscope	Scanning Electron Microscope	Optical Microscope		Visible To Naked Eye			
Micrometers (Log Scale)	Ionic Range	Molecular Range	Macro Molecular Range	Micro Particle Range		Macro Particle Range		
	0.001	0.01	0.1	1.0	10	100	1000	
Angstorm Units (Log Scale)	10^1	10^2	10^3	10^4	10^5	10^6	10^7	
Approximate Molecular Wt.	100 200 1000 10,000 20,000	100,000	500,000					

Figure 1.1: Types of Membrane filtration processes based on membrane pore sizes.

There are various configurations of operating a filtration process:

Dead-end Filtration

The most basic form of filtration is dead-end filtration. The complete feed flow is forced through the membrane and the filtered matter is accumulated on the surface of the membrane. The dead-end filtration is a batch process as accumulated matter on the filter decreases the filtration capacity, due to clogging. A next process step to remove the accumulated matter is required. Dead-end filtration can be a very useful technique for concentrating compounds.

Cross-flow Filtration

With cross-flow filtration a constant turbulent flow along the membrane surface prevents the accumulation of matter on the membrane surface. The membranes used in this process are commonly tubes with a membrane layer on the inside wall of the tube. The feed flow through the membrane tube has an elevated pressure as driving force for the filtration process and a high flow speed to create turbulent conditions. The process is referred to as "cross-flow", because the feed flow and filtration flow direction have a 90 degrees angle. Cross-flow filtration is an excellent way to filter liquids with a high concentration of filterable matter.

Hybrid-flow Filtration

The hybrid flow process combines the dead-end and the cross-flow principle. As in the cross-flow filtration tubular membranes are with the filtration layer on the inside wall are used. The filtration process has two phases: the production phase and the flushing phase. During the production phase, the tubes are closed on one side and a dead-end filtration is performed. During the flushing phase, the tube is open on both sides and the fraction that did not pass through the membranes is removed in order to clean the membrane surface as in cross-flow filtration. This filtration technique is especially suitable for treating water streams containing suspended solids in low concentrations (polishing).

Submerged Filtration

With submerged membrane filtration the membranes are submerged in the liquid that has to be filtered. The filtration is performed from the outside to the inside of the membrane (filtering layer is on the outer side of the tube or plate). Sheer forces along the membrane surface are created by a flow of air bubbles along the surface. In some cases the airflow also results in a liquid flow created by the airlift principle. The driving force is a vacuum applied on the inner side of the membrane.
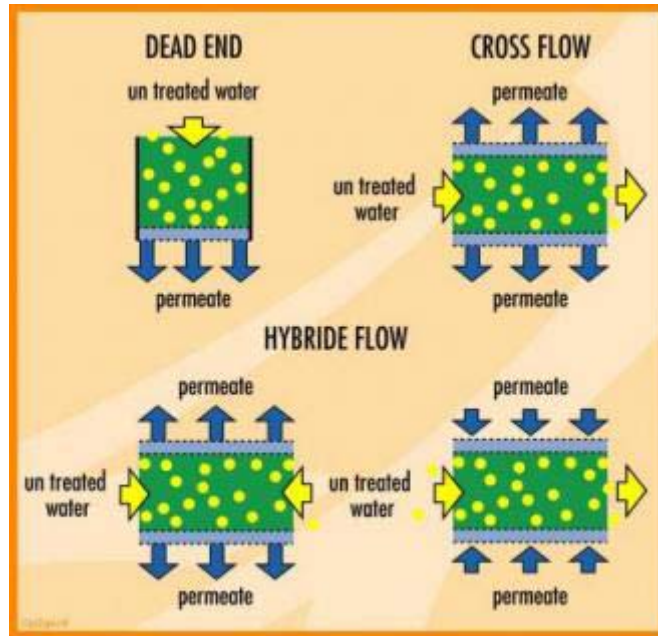
Figure 1.2: Diagrammatic representation of various configurations of operating a filtration process

1.2 Dead End Filtration: Theory & Equations

The dead end filtration is the one where the flow of water is perpendicular to the membrane surface. The water is pushed through the membrane by pressure. All the water that is introduced in the dead-end-cell passes through as the permeate, in other words there is no rejected water. In dead end filtration the retained particles build up with time one the membrane surface or within the membrane. In either case, the particle builds results in an increased resistance to filtration and causes the permeate flux to decline, as a result dead end filtration requires the stopping of filtration in order to clean or replace the membrane therefore this type of filtration is also called batch filtration.

There are two types of filtration which can be employed in a dead end cell unit; dead-end microfiltration with constant flux and dead end microfiltration with constant pressure drop. The dead end microfiltration with constant flux ensures that the permeate flux through the filter remains constant, this filtration can be achieved by positive displacement pump. As the cake build-up increases with time, the pressure drop must be increased to maintain constant flux. In dead end microfiltration with constant pressure, as the cake build-up with the time the permeate flux decreases.

1.3 Membrane Transport Mechanism

Membranes provide absolute barrier to particles greater than their pore size. A membrane process requires two bulk phases physically separated by a third phase, the membrane. (figure 1.3).The membrane phase interposed between the two-bulk phases controls the exchange of mass between the two bulk phases in a membrane process. The process allows the selective and controlled transfer of a certain species from one bulk phase to another bulk phase separated by the membrane.
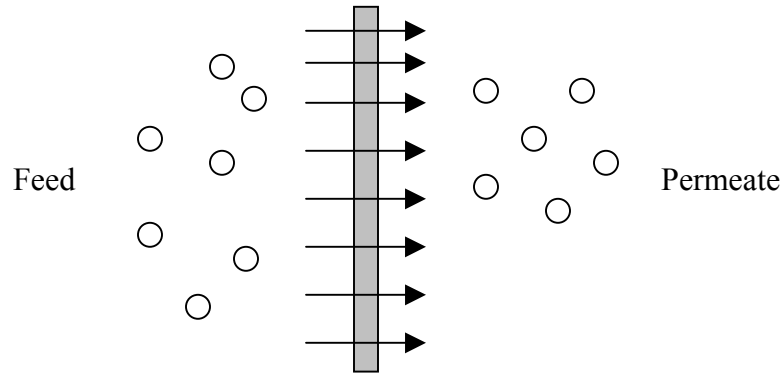
Figure 1.3: Schematic representation of the different phases in membrane separation

The flow of water through membranes can be modeled using empirical equations.The Hagen-Poiseuille or Carman-Kozeny models can be used to demonstrate this flow of water.The use of these equations depends on the shapes and sizes of the pores.

a) Hagen-Poiseuille Equation

The Hagen-Poiseuille equation considers that the membrane has a number of parallel cylindrical pores,which are parallel or oblique to the membrane surface.It assumes that the capillaries are uniform and cylindrical.

$$J = \left(\frac{\varepsilon_m \cdot r^2}{8\eta\tau} \right) \left(\Delta P / \Delta X_m \right)$$

Where,

J is the flux(L/m^2.hr)

ΔP is the pressure difference(N/m^2)

ΔX_m is the membrane thickness(m)

η is the viscosity of liquid(N.s/m^2)

ε_m is the surface porosity

b) Carman-Kozeny Equations

This equation is used for membranes which consist of closely packed spheres.

$$J = \left(\frac{\varepsilon_m^{\,3} \cdot r^2}{K \Delta X_m S_m^{\,2} (1 - \varepsilon_m)^2} \right) \left(\Delta P / \eta \right)$$

Where,

J is the flux(L/m^2.hr)

S_m is the pore internal surface area/unit volume(1/m)

ΔP is the pressure difference(N/m^2)

K is the Carman-Kozeny constant

ΔX_m is the membrane thickness(m)

η is the viscosity of liquid(N.s/m^2)

ε_m is the surface porosity
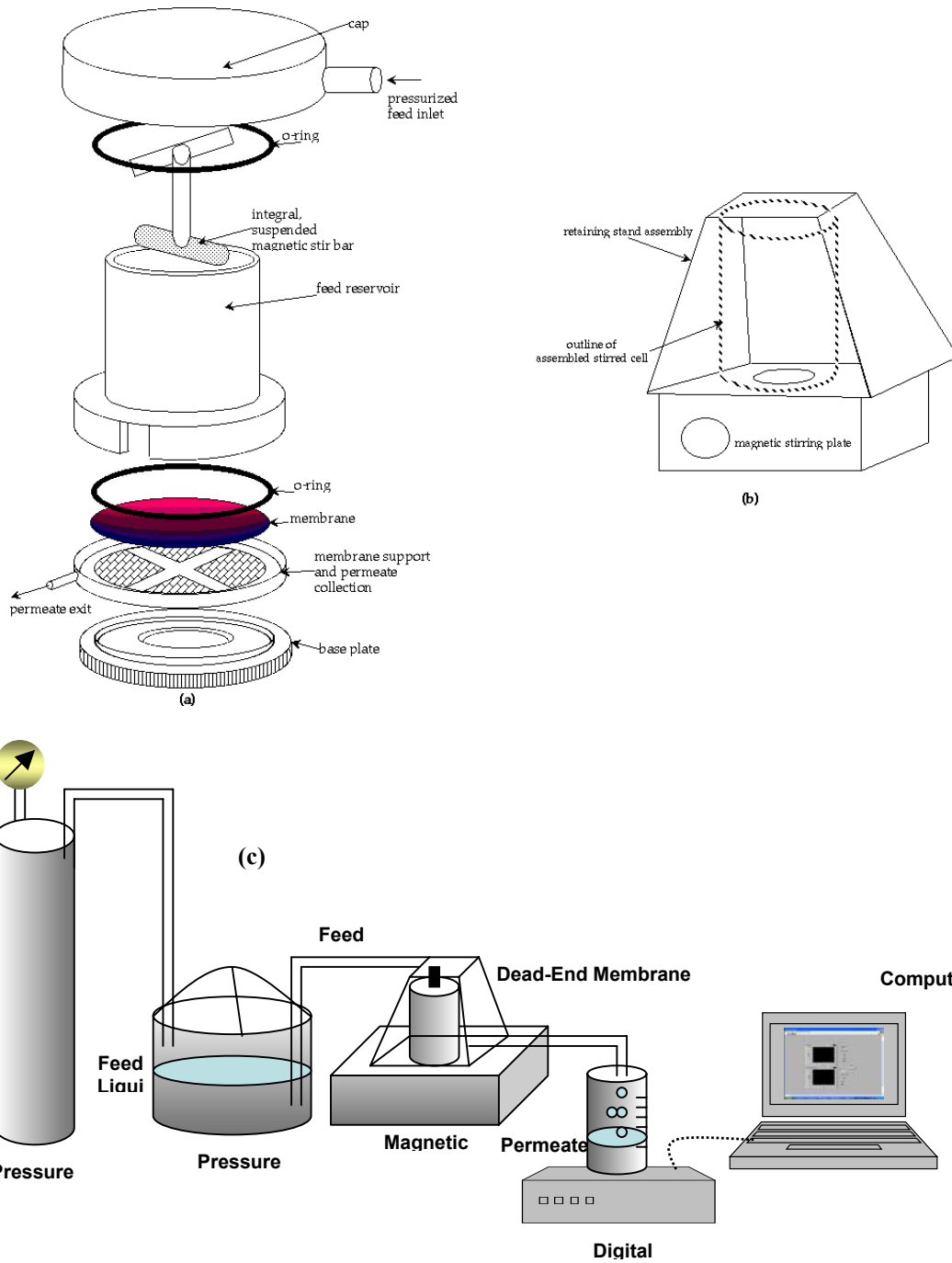
II. Experimental Setup

Figure 2.1 (a) Expanded-view schematic of a stirred-cell membrane filtration assembly. (b) An external housing provides the clamping pressure to keep assembly together on top of a magnetic stirrer plate. A cut-out in the external housing facilitates good coupling between the magnetic stirrer and the unit's internal stir bar. (c) A typical process configuration for batch filtration using an inert gas blanket on the feed reservoir.

The experimental setup consisted of Amicon Model 8200, equipped with 180 mL filtration cells, this setup uses nitrogen gas tank to maintain the necessary pressure gradient to force the feed solution through the membrane. The magnetic stirrer provides a high but undefined shear force needed to reduce solid cake built up on the membrane surface. The installation procedures of each component in the setup is given below;

2.1 Equipment Installation

2.1.1 Dispensing Pressure Vessel

The dispensing pressure vessels are designed for dispensing fluids under pressure for filtration through any pressure-type filter holder. The volume of portable vessels used in our setup is 5 liter. The vent/relief valve (f) and the pressure gauge (g) must be used when operating the dispensing pressure vessel. Before initial use and after each use, disassemble all threaded fittings and rinse the vessel and fittings with laboratory-grade water. For a more thorough cleansing, wash the vessel with a mild, nonabrasive detergent and a soft brush or sponge before rinsing. Air dry all parts before reassembly. **CAUTION:** If salt solutions were used in the vessel, rinse thoroughly to prevent corrosion of the vessel and fittings. Figure 2.2 shows a typical dispensing vessel used in our setup.

Figure 2.2: Millipore Dispensing Vessel

2.1.2 Stirred Cell

This section explains how to install the membrane and stirrer assembly. To identify the parts of the stirred cell, refer to figure 2.3.
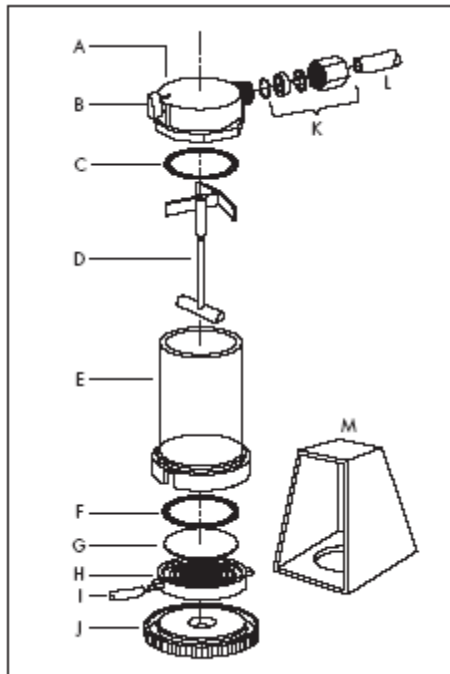
A Cap Assembly
B Pressure Relief Valve
C O-Ring D Stirrer Assembly
E Body
F O-Ring
G Membrane
H Membrane Holder
I Elastomeric Tubing 4 inch.
J Base
K Tube Fitting Assembly
L Tubing, Plastic 4 ft
M Stand Assembly

Figure 2.3.Parts of the stirred cell

2.1.3 Membrane

The ultracel membranes YM-10 were used in the setup,The hydrophilic, tight microstructure of Ultracel membranes assures the highest possible retention with the lowest possible adsorption of protein, DNA or other macromolecules.

Description	Ultrafiltration Discs, YM-10, Ultracel regenerated cellulose, 10 kDa NMWL, 63.5 mm
Brand Name	Ultracel
Filter Code	YM-10
Filtration Area, cm^2	28.7
Filter Diameter, mm	63.5
Filter Material	Regenerated cellulose
Qty/Pk	10
NMWL, kDa	10

Table 2.1: Specifications

Place the membrane into the holder, shiny side up. Place the O-ring on top of the membrane. Gently push the O-ring down, so that it contacts and seats the membrane evenly in the bottom of the holder. Fit the membrane holder into the cell body, aligning

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the tabs on the sides of the holder with the slots in the base of the cell body. Invert the cell body and membrane holder; screw the base firmly into the bottom of the cell body. Push the filtrate exit tubing onto the exit spout of the membrane holder.
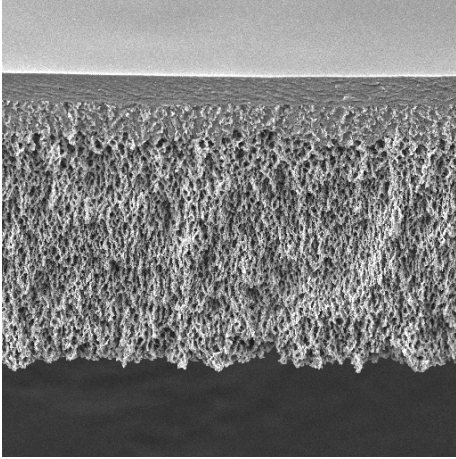

Figure 2.4. Ultracel regenerated cellulose ultra filtration membrane

2.1.4 Stirrer Assembly

Place the stirrer assembly into the cell body. When properly installed, the arms of the stirrer assembly will rest on the small ridge inside the top of the cell body. Pour the sample into the cell. Push the cap assembly down onto the cell body, using a twisting motion and orienting the gas inlet port on the cap opposite the filtrate exit port on the holder (NOTE: If the cap assembly does not slide easily, lubricate the O-ring lightly with water or petroleum jelly. Do not allow petroleum jelly to contact the membrane, to avoid clogging the membrane pores.) Set the pressure-relief valve knob to the horizontal (open) position. Slide the cell into the retaining stand, fitting the ring on the cell base into the hole in the stand. The flattened edge on the bottom flange of the cap ensures that the cell is inserted properly and prevents rotation of the cell once inside the stand. Turn the pressure-relief valve knob to the vertical (closed) position.

2.1.5 Gas Pressure Line

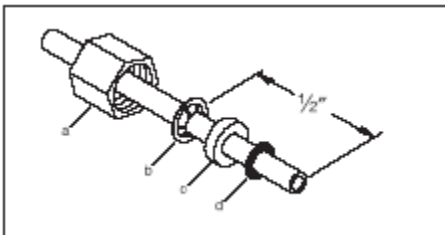Attach the gas pressure line to the tube fitting on the cap as follows:

Figure 2.5.Installation of gas pressure line

Unscrew the hexagonal nut (a) from the cap assembly and remove the O-ring (d).Slide the hex nut onto the end of gas tubing. Slide the metal grab ring (b) down one half-inch onto the tubing, with the flange facing away from the hex nut. Add the white spacer (c), positioning the countersunk surface toward the flange on the grab ring. Slide the O-ring (d) onto the end of the tubing. Insert the exposed tubing into the cap assembly and hand-tighten the hexagonal nut. Do not over tighten.

Limitations

This section explains the limitations of the stirred ultrafiltration cell.

Pressure Limits

Do not exceed 75 psi (5.3 kg/cm2) nitrogen gas pressure. CAUTION: To avoid the cap popping off and splattering its contents during pressurization, never operate the cell without its retaining stand.

Temperature Limits

Although brief exposure to higher temperatures is possible, do not operate cell continuously above 85 °C (185 °F).

Chemical Resistance

Do not use the stirred cell with: Strong acids (pH < 2) or strong alkalies (pH > 10)Ketones (including acetone) Aromatic hydrocarbons (including toluene) CelloSolve® solvent Halogenated hydrocarbons DMF Aliphatic esters DMSO Polar aromatics NOTE: The spring in the pressure-relief valve is NOT compatible with 0.1 N NaOH. For other solvent compatibilities, consult a standard text or contact Millipore Technical Service. For the chemical resistance of disc membranes, see the instructions packed with the product.

2.1.6 Cell Operation

Connect the inlet line to a regulated gas pressure source. NOTE: Use nitrogen gas for pressurizing the cell. Using compressed air can cause large pH shifts, due to dissolution of carbon dioxide. With sensitive solutions, oxidation can occur also, potentially leading to other problems. Hold the cell steady on the laboratory bench and pressurize. Follow the instructions in the membrane package to determine optimal operating pressure. Do NOT exceed the cell pressure limit of 75 psi (5.3 kg/cm2). Once the system is pressurized, the cap assembly moves upward, forming a secure lock with the retaining

stand. Place the cell on the magnetic stirring table. To avoid membrane damage, before turning on the stirring table, make sure that the cell is pressurized.

▲WARNING: <u>When operating with hazardous or especially valuable materials, pressure-check the cell thoroughly, to ensure that all components have been properly assembled, before turning on the stirring table.</u>

Turn on the stirring table and adjust the stirring rate, until the vortex created is approximately one-third the depth of the liquid volume.

2.1.7 Digital Balance

The digital balance from Acculab was employed to measure the mass coming out as permeate,it was connected using the USB port to a computer where a data acquisition system called Lab view was employed to control the system as well as to continuously log data during filtration. Figure 2.6 shows the Acculab Digital balance with the specification.

Adapters: RS-232 and
USB data interfaces

Capacity	3100g
Readability	0.1g
Linearity	± 2d
Repeatability	± 1d

Figure 2.6: Acculab Digital balance

2.2 Cost of Equipments

In order to setup the unit most of the equipments where brought from Millipore and Cole-Palmer, They also provided good customer support for installation. The total cost of this unit was $ 2154.5.Table 2.2 shows the cost of each equipment which were used to complete the setup.

Items	Catalog Number	Company	Price
Dispensing			
Pressure Vessel(S.Steel), 5 L	XX6700P05	Millipore	779
Stirred Cell Model 8400	5123	Millipore	772
200 ml(Dead End Unit)			
Ultrafiltration Discs, YM-10	13632	Millipore	170
Ultracel regenerated cellulose			
10 kDa NMWL, 63.5 mm			
Tubing (Polypropylene Tubing, 3/16" X 1/4", 100'(package))	EW-95875-04	Cole Palmer	22.05
Quick Connectors	C-06372-00	Cole Palmer	15
	C-06372-30	Cole Palmer	23
	C-06372-20	Cole Palmer	26
	C-06372-50	Cole Palmer	5.25
	C-06378-10	Cole Palmer	52.25
Digital Balance,Vicon	EW-75211-15	Fischer	219.95
USB Connector(RS32)	PCM-2120-03	cables4computers.com	70
		Total	**2154.5**

Table 2.2: Cost of Equipments for the Dead-end membrane filtration unit

2.3 Data Acquisition

Once the set up is ready it is important to connect the digital balance to computer in order to acquire data with relative ease and with greater accuracy,this can be done using software called Lab VIEW. LabVIEW programs are called virtual instruments, or VIs, because their appearance and operation imitate physical instruments, such as oscilloscopes and multimeters. LabVIEW contains a comprehensive set of tools for acquiring, analyzing, displaying, and storing data, as well as tools to help you

troubleshoot code you write. It also helps in controlling the experiments from a computer. In this setup Lab VIEW is used to communicate with digital balance having RS232 port for acquiring the flowrate and than calculating and displaying the flux. Before running the experiment in LabVIEW environment one has to make sure that the digital balance used in the setup is communicating with the computer and than a simple LabVIEW code is used to acquire data and display it in real time. The following steps showed are followed in order to effectively connect digital balance to computer.

1) **Balance & USB Adapter Installation**

The USB interface adapter provided with the Digital balance is a solution to enable direct connection of the balance to a computer on the USB (Universal serial bus) port. The USB interface is implemented as a virtual serial interface over RS-232(virtual com port).For the application program running on the computer, there is no difference whether the serial interface is installed in the PC or enabled in the form of a virtual interface. The interface offers the same capabilities in either case.

The equipment supplied with the USB interface includes a CD containing the software drivers needed to set up a virtual interface on the computer. Follow the instructions needed to install the drivers for the USB interface using the supplied CD.In order to configure the USB interface, Switch on the computer and make sure the USB port is activated and functional. Plug the USB cable into the USB port on the computer and switch on the balance, Windows^R detects the USB device and runs the wizard for adding/removing hardware. At the end of the process, the virtual port should be ready to operate; Windows^R usually adds the virtual port in the position following highest-numbered COM port. (Port numbers are assigned under "port settings" in the Device Manager, opened from My Computer-Control Panel-System Properties-Hardware).Next we configure the balance and its data interface by setting following parameters in the balance. Please refer to the instructions manual provided with the acculab digital balance and set the following parameters;

Baud rate -1200 baud

Parity-odd

Handshake Mode- none.

Manual/automatic printing-Manual without stability

Now the Digital balance is ready to communicate with computer.

2) Communicating with the Digital Balance

There are two ways in which we can check whether our instrument is communicating with the computer, which are as follows;

a) **HyperTerminal**

HyperTerminal is a serial communication program provided in the Windows 98SE/ME/2000 and XP operating systems. We can use HyperTerminal to communicate with any balance equipped with an RS-232 serial interface or USB. HyperTerminal will receive the data output from the balance and display the information on the monitor screen of HyperTerminal program.

Step 1

We start HyperTerminal by clicking Start, pointing to Programs, pointing to Accessories, pointing to Communications, and the click HyperTerminal.
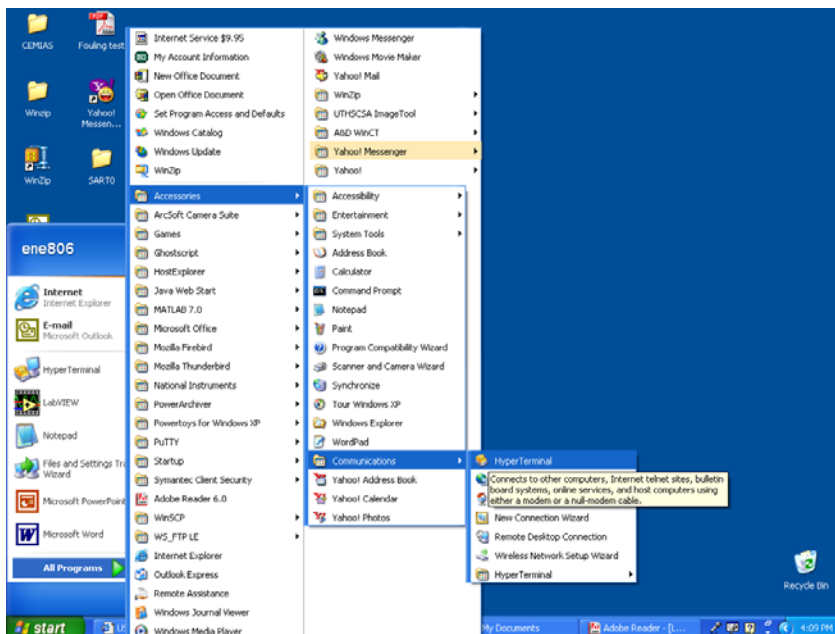
Step 2

After a few moments the Connection description window will appear. Enter a valid name, such as "Sartorius", into the Name: field, the Icon: field can remain at default. Click OK when finished.
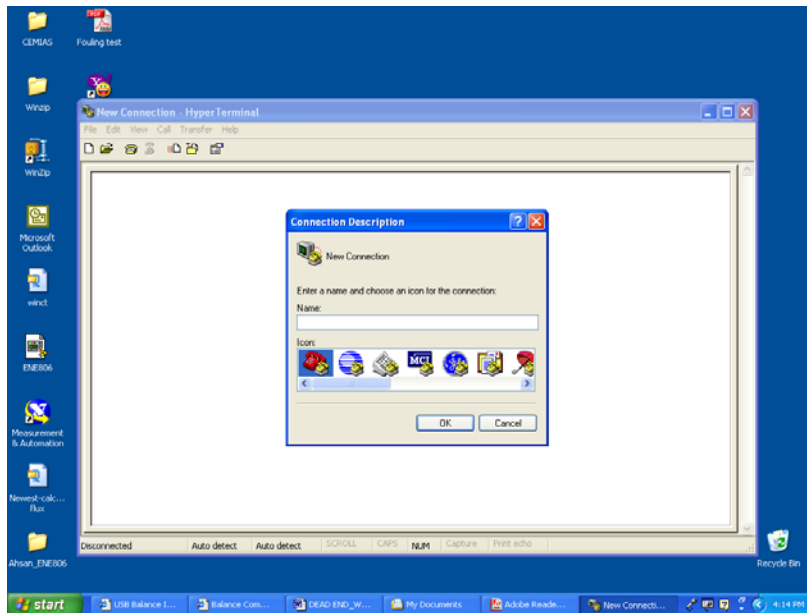
Step 3

The next screen is the Connect To window. Enter the COM port number you are using into the Connect using: field. The bottom pull down allows you select from Direct to Com1, Direct to Com2, Direct to Com3 or Direct to Com4. The most common selections are Direct to Com3, or Direct to Com4. The Country Code: Area code: and Phone Number: fields can remain at default. Click OK when finished. (Note: The most common problem resulting in data not transferring to HyperTerminal is selecting the wrong Com Port. If Com3 does not work then try Com4.)
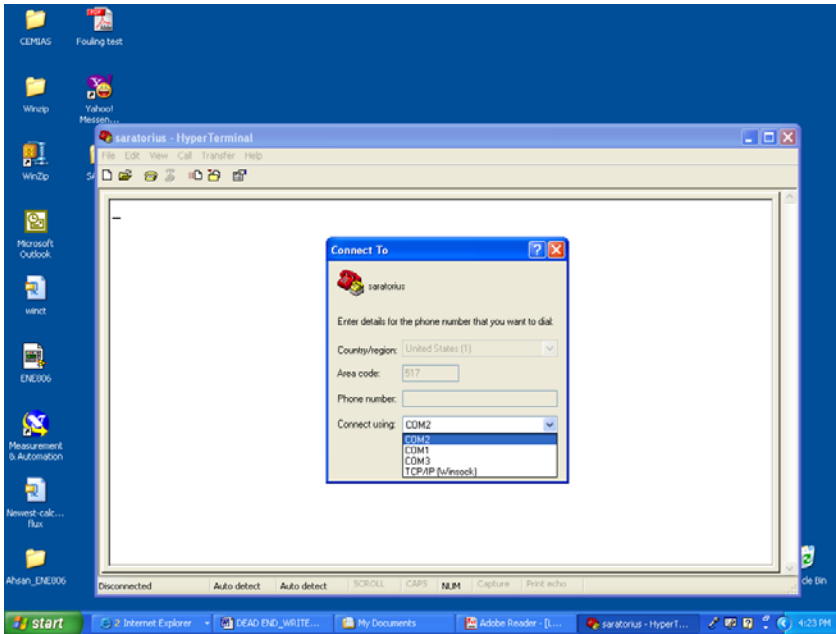
Step 4

The next screen is the Properties window. Enter the baud rate into the Bits per second: field. The top pull down typically allows you to select from the available bit rates. The Sartorius default format is 1200. The data bits: Odd, Stop bits: 1 and Flow control: None. Click OK when finished.

Note: If the Baud rate, Data bits, Parity or Stop bits are not set correctly then HyperTerminal will not work. Be careful and select the correct setting.
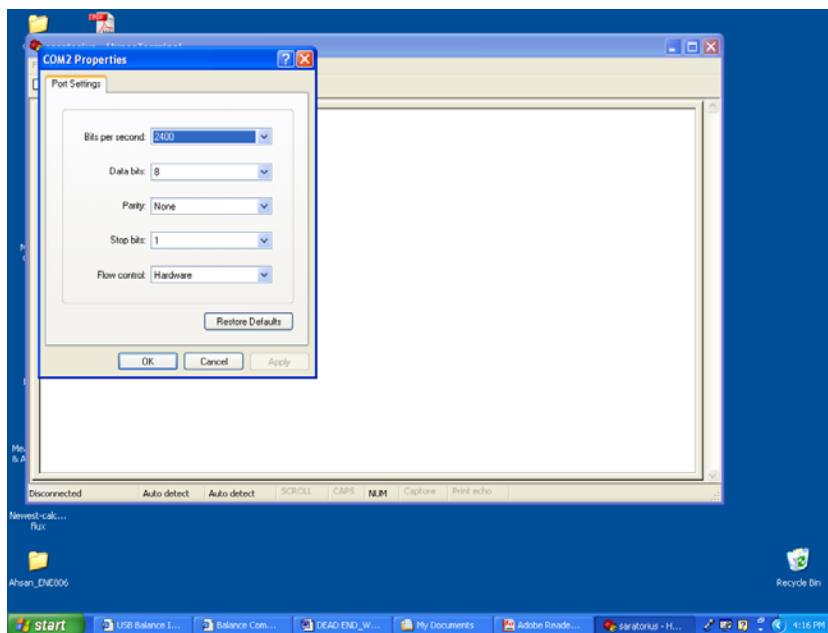
Step 5

In the following HyperTerminal window click on Files then on Properties then on Settings then on ASCII setup. Put a check mark in the box marked Append line feeds to incoming line ends. Click OK when finished and then Click OK again so you are back in the HyperTerminal window.

Then Close the HyperTerminal window and say Yes to disconnect now and Yes to Save HyperTerminal session when prompted to Sartorius balance.

Now you're ready to use HyperTerminal.

1.   Start your data acquisition by clicking Start, pointing to Programs, pointing to Accessories, pointing to Communications, clicking HyperTerminal, and then clicking on the Sartorius balance icon in our example.

2.   Connect the Sartorius Balance to your PC's USB Port with our USB to Serial Adapter cable and then apply power to the balance.

3.   Press the "Print" key on the Sartorius balance and weight shown on the display of the balance also shows in the HyperTerminal screen.

4.   For information about how to use HyperTerminal, click the Help menu in HyperTerminal.
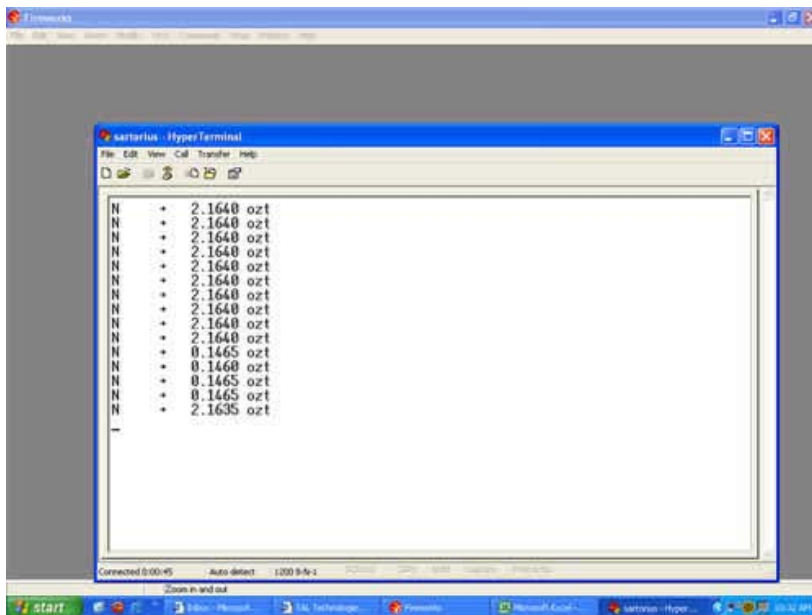
b) Communicating using LabVIEW (Instrument I/O Assistant Express VI)

Instrument drivers simplify instrument control and reduce test program development time by eliminating the need to learn the programming protocol for each instrument. An instrument driver is a set of software routines that control a programmable instrument. Each routine corresponds to a programmatic operation such as configuring, reading from, writing to, and triggering the instrument. Use an instrument driver for instrument control when possible. National Instruments provides thousands of instrument drivers for a wide variety of instruments.Use the NI Instrument Driver Finder to search for and install instrument drivers without leaving the LabVIEW development environment. Select Help»Find Instrument Drivers to launch the Instrument Driver Finder. You also can visit the NI Instrument Driver Network at ni.com/idnet to find a driver for an instrument. If a driver is not available for an instrument, you can use the Instrument I/O Assistant Express VI to communicate with the instrument.Before starting to work with LabVIEW download the Visa from ni.com and install it on the computer
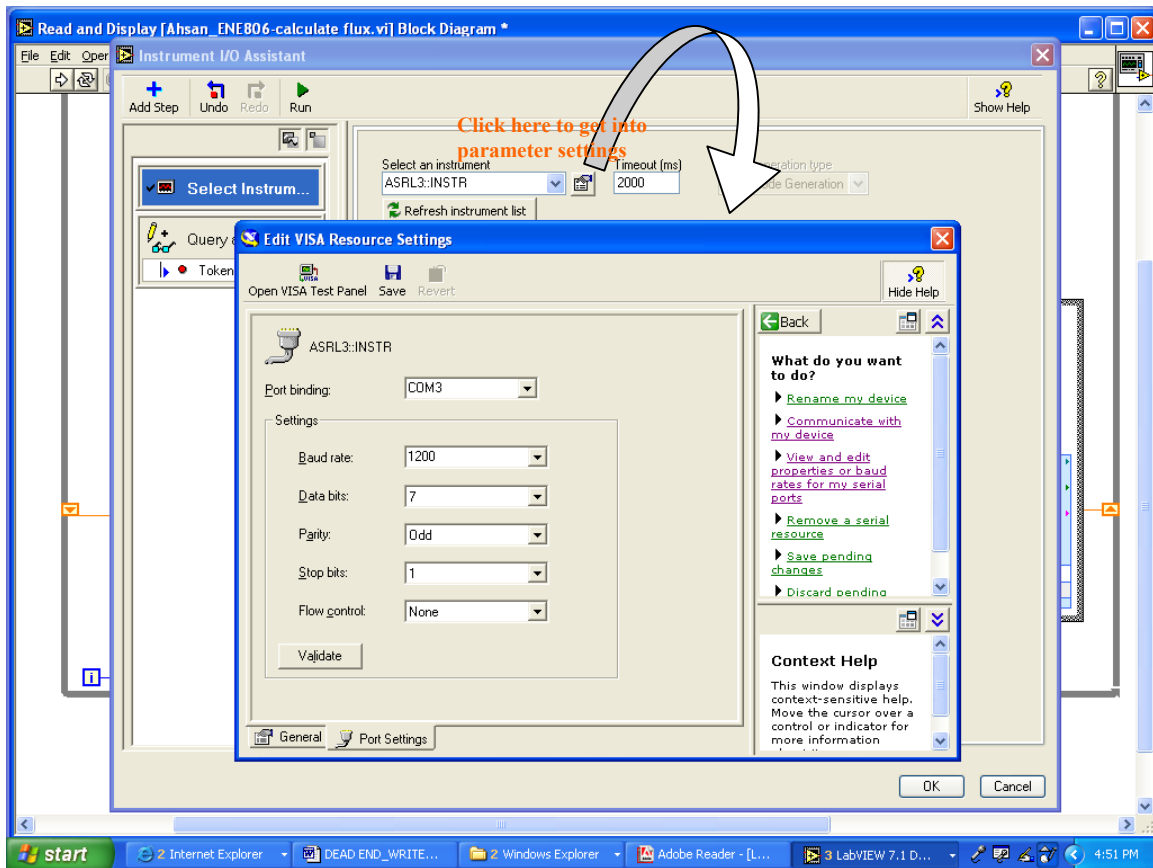
Selecting an Instrument

Before you communicate with an instrument, you must select the instrument with which you want to communicate. Complete the following steps to use the Instrument I/O Assistant Express VI to select an instrument.

1. Turn on the instrument you want to use. The instrument must be powered on to use the Instrument I/O Assistant Express VI.

2. Select the Instrument I/O Assistant Express VI on the Input palette and place it on the block diagram. The Instrument I/O Assistant dialog box appears.
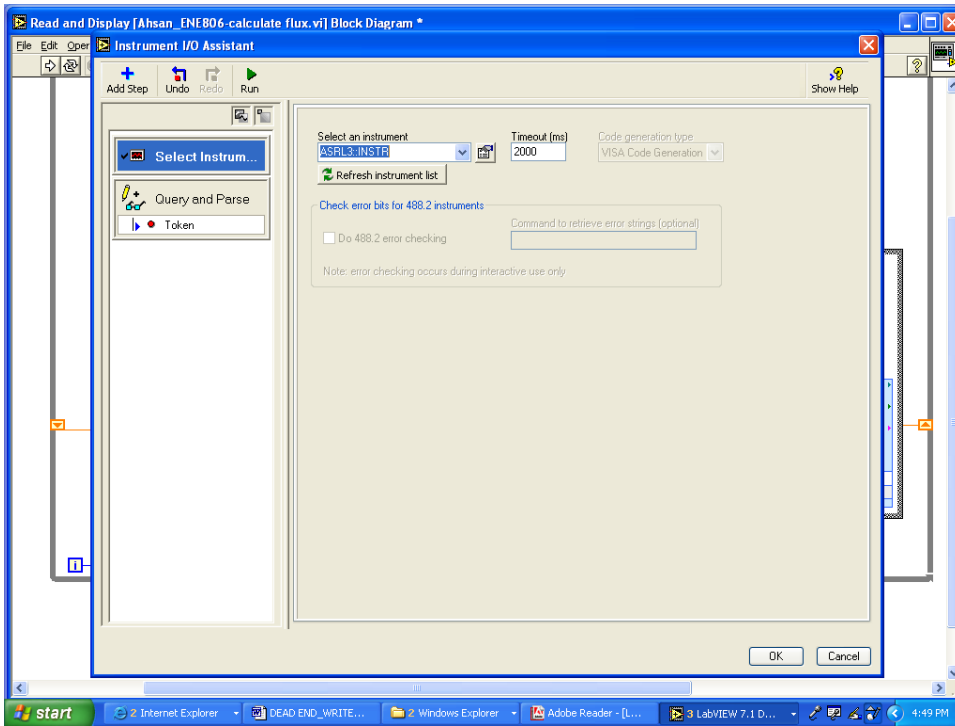
3. Click the Show Help button, shown at left, in the upper right corner of the Instrument I/O Assistant dialog box. The help appears to the right of the dialog box. The top help window contains how-to information about using the Instrument I/O Assistant. The bottom help window provides context-sensitive help about components in the dialog box.

4. Click the Select Instrument link in the top help window and follow the instructions in the help window to select the instrument with which you want to communicate.

5. Configure the properties of the instrument. This can be done by setting parameters in LabVIEW which should exactly match with the setting of digital balance.

6. If you want to minimize the help window, click the Hide Help button, shown at left, in the upper right corner of the Instrument I/O Assistant dialog box.

Acquiring and Parsing Information for an Instrument

After you select the instrument, you can send commands to the instrument to retrieve data. In this exercise, you will learn to use the Instrument I/O Assistant Express VI to acquire and parse identification information for an instrument. Complete the following steps to communicate with the instrument.

1. In the Instrument I/O Assistant dialog box, click the Add Step button and click the Query and Parse step.

2. Enter *SMP in the Enter a command text box.*SMP is a query that most instruments recognize.

3. Click the Run button, shown at left. The Instrument I/O Assistant sends the command to the instrument, and the instrument returns its identification information.

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4. Select ASCII only from the pull-down menu below the Byte index column of the response window to parse the instrument name as an ASCII string. You also can use the Instrument I/O Assistant to parse ASCII numbers and binary data.

5. Click the Parsing help button, shown at left, in the Instrument I/O Assistant dialog box to display information about parsing data.

6. In the ASCII representation column of the response window, click the value you want to parse.

7. Enter a name for the token or parsed data selection, in the Token name text box.

8. Click the OK button to save the current configuration and close the Instrument I/O Assistant dialog box. The name that you entered in the Token name text box is the output of the Instrument I/O Assistant Express VI, shown at left.
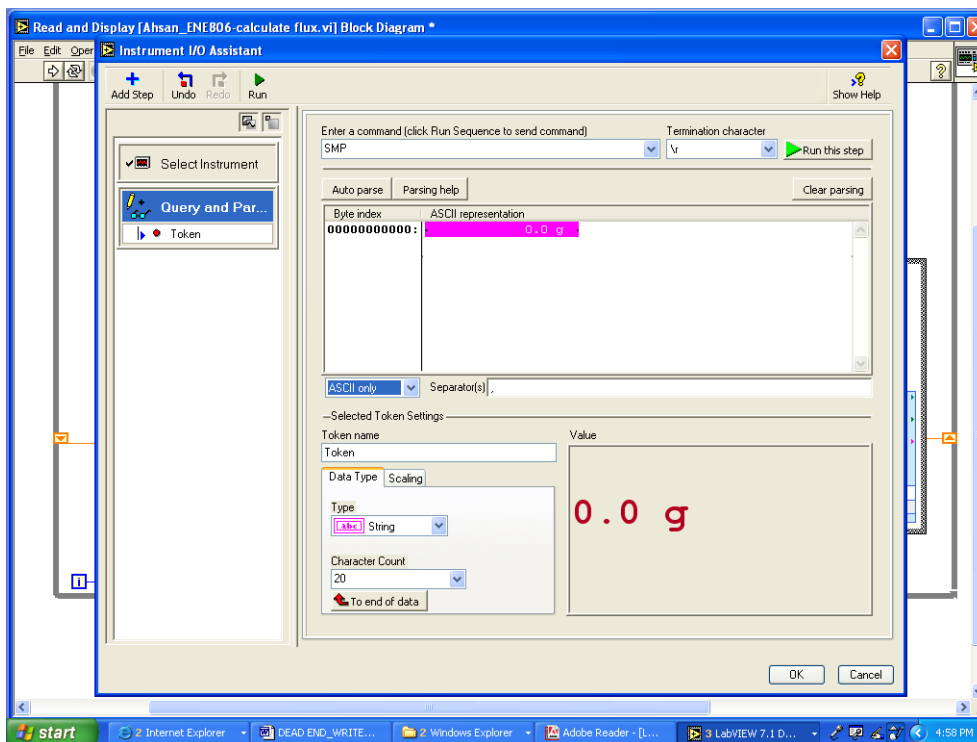

The above two methods will ensure that the digital balance is properly communicating with the computer, before proceeding make sure to save the setting of the second method that is Instrument I/O assistant which will be later used in LabVIEW program to acquire data.

3) Lab View program to acquire and display data.

a) Open LabView and Click to Open the user friendly VI program specially designed for measuring flux through membrane.(For creating a program please refer to LabVIEW manual provided by national Instruments)
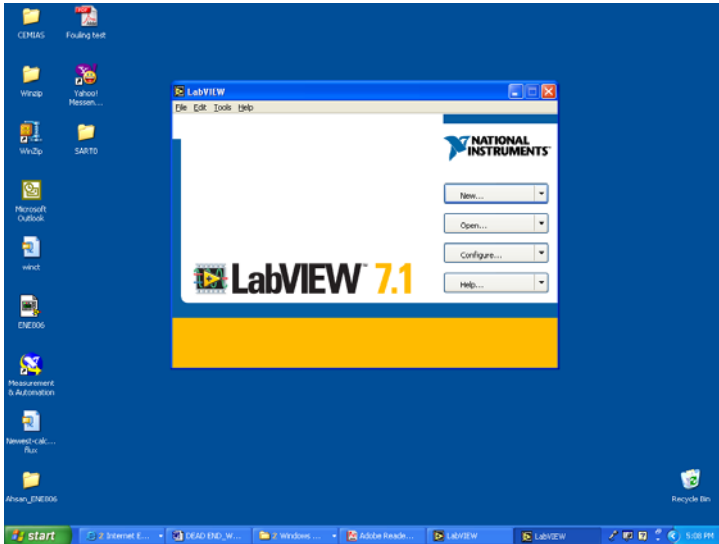

b)The following window will be displayed which shows the overall block diagram for acquiring data and than using it to calculate the flux.
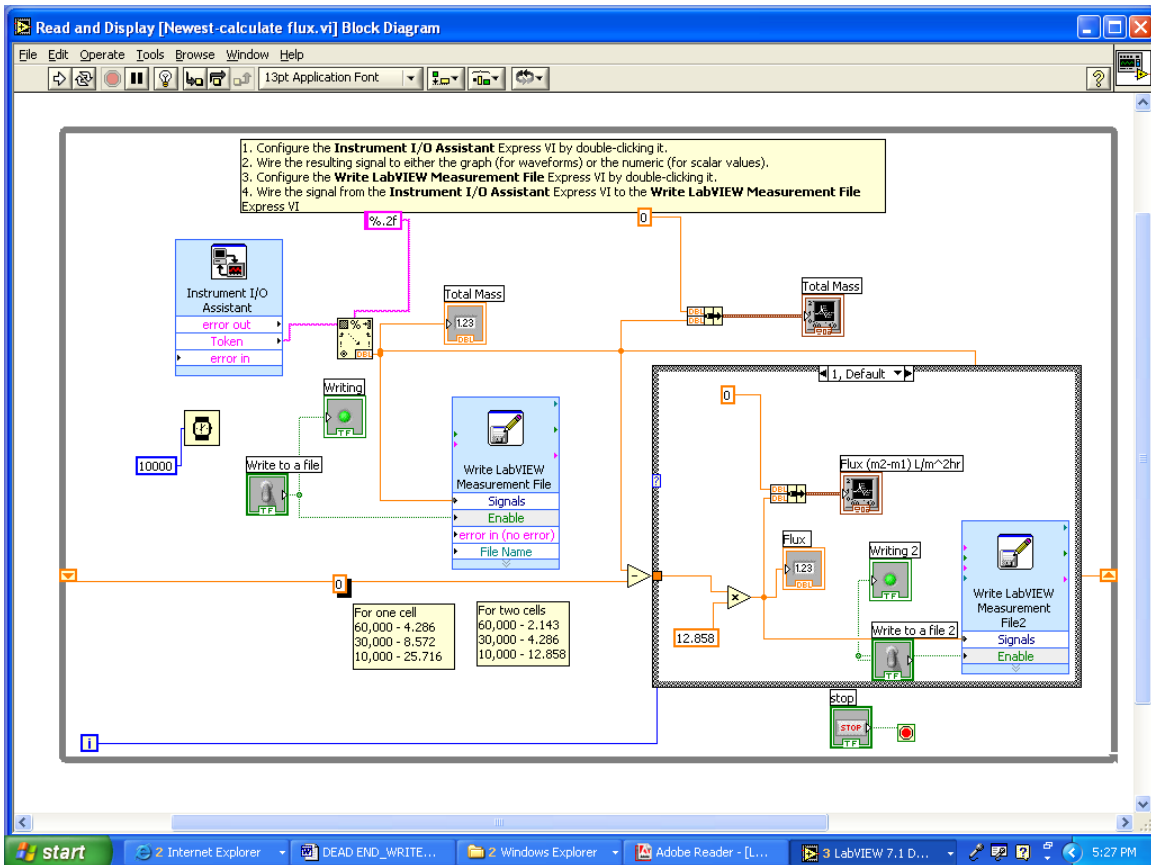
c) Configure the Instrument I/O Assistant Express VI by double clicking it. Follow instructions from section 2(b) "**Communicating using LabVIEW (Instrument I/O Assistant Express VI)".**Make sure to place correct parameters eg. baud rate, parity etc.

d) Wire the resulting signal to either the graph (for waveforms) or the numeric (for scalar values)

e) Configure the Write LabVIEW Measurement File Express VI by double-clicking it and make sure to provide a correct path for the file name.
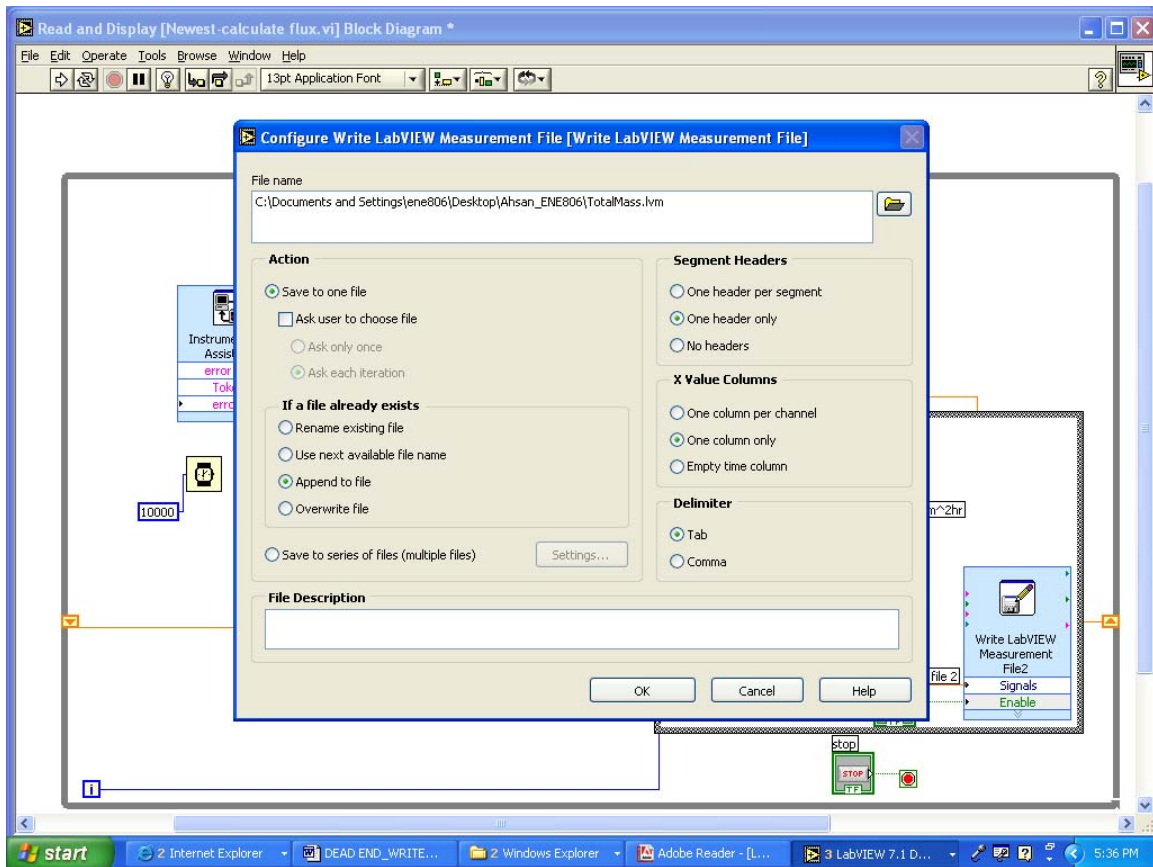
e) Wire the signal from the Instrument I/O Assistant Express VI to the Write LabVIEW Measurement File Express VI.Configure the Write LabVIEW Measurement File2 and provide a correct path to the file name.
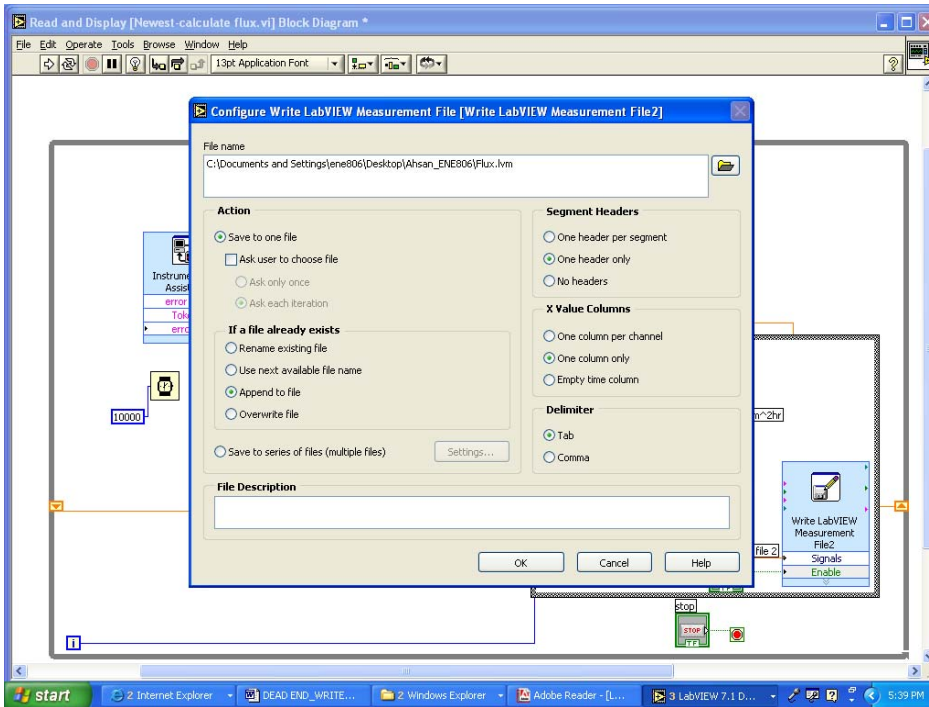
f) Save the file and before doing so make sure that the in Display window the panel for total mass and Flux are properly aligned.
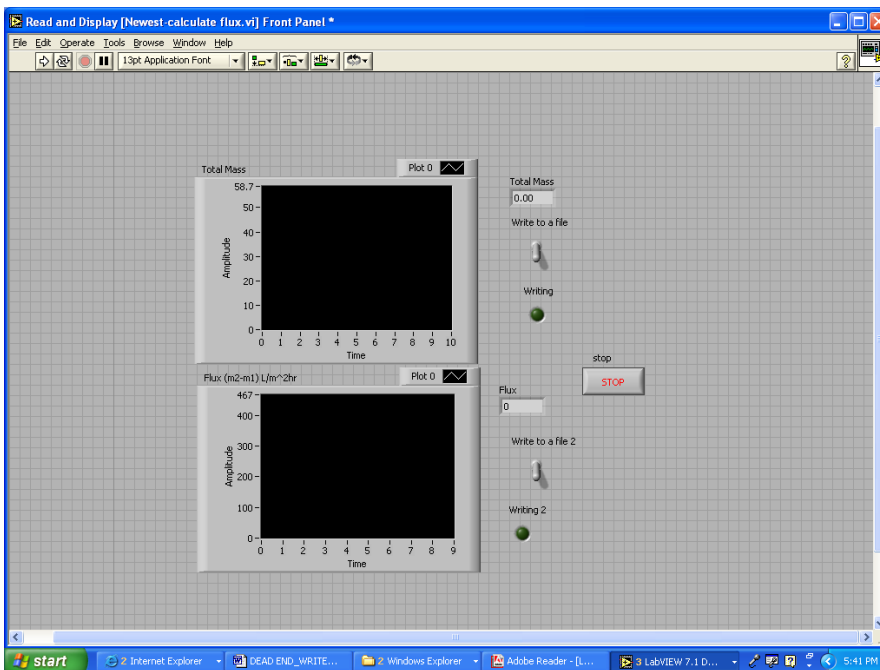
g) Turn on the digital balance and start reading the data in Lab VIEW environment. The data collected will be stored at the path provided before and can be imported to excel.

III. Experimental Procedure

The experiments were performed using Ultacel YM-10 membranes with a MWCO of 10000 Daltons. The membranes were asymmetrical with 0.1–1.5 mm thickness and a nominal diameter of 63.5 mm. The membranes were pretreated by soaking in distilled water for 1 hr with the skin side down as recommended by the manufacturer. The water was changed twice during the soaking process. The reasons for membrane preparation by soaking are to prevent the membranes from absorbing any of the filtration water and to remove any materials that may have been attached on the membranes during packaging and shipment. A new membrane was used for each test run. Filtrate from the UF process was collected in a graduated cylinder which was kept on digital balance, a data acquisition system called Lab view was employed to control the system as well as to continuously log data during filtration.

3.1 Clean Water Resistance Test (CWRT)

Initial membrane resistance experiment was conducted to find the membrane resistance of the new membrane. The pure water was withdrawn through the membrane and the pressure was measured by a pressure gauge. The experimental setup for the measurement of initial membrane resistance is shown in Figure 3.7.

Initial membrane resistance was conducted by following steps.

1. Filter the pure water through the membrane system.

2. Vary transmembrane pressure and record change in filtration flux.

3. Plot graph δP versus J and find out R_m, which is initial membrane resistance.

3.2 Fouling Test

In order to show the phenomenon of fouling, an experiment was performed using tap water from the laboratory. The transmembrane pressure was kept constant at 60psi throughout and the permeate flux for the given membrane(YM-10) was investigated for a period of about 1hr.The filtration experiments were performed at a room temperature that varied from 20^oC to 24^oC

IV. Results & Discussion

Table 4.1 shows the change in flux with increase in transmembrane pressure. This is done in order to calculate the initial resistance of the membrane using pure water. The experiments were run over period of 30min. We can see that increase in the pressure across the membrane also increases the flux.

P(psi)	P(bar)	J(L/m²h)
15	1.034205	7.7148
30	2.06841	11.5722
45	3.102615	15.4296
60	4.13682	19.287

Table 4.1 Flux rate for increasing pressure using pure water

The flux can be related to the total hydraulic resistance by Darcy's law given by;

$$J = \frac{dV}{dt} = \frac{\Delta P}{\mu(R_m + R_c)} \tag{4.1}$$

Since we used pure water therefore there will not be any resistance due to cake formation (Rc=0).The equation than becomes;

$$J = \frac{\Delta P}{\mu(R_m)} \tag{4.2}$$

Where;

μ is the viscosity of water(cP)

R_m is the membrane resistance (1/m)

J is the flux (L/m²h)

ΔP is the transmembrane pressure (bar)

Figure 4.1 shows a graph of J vs ΔP where the slope of the curve can be used to calculate the initial resistance of the given membrane.
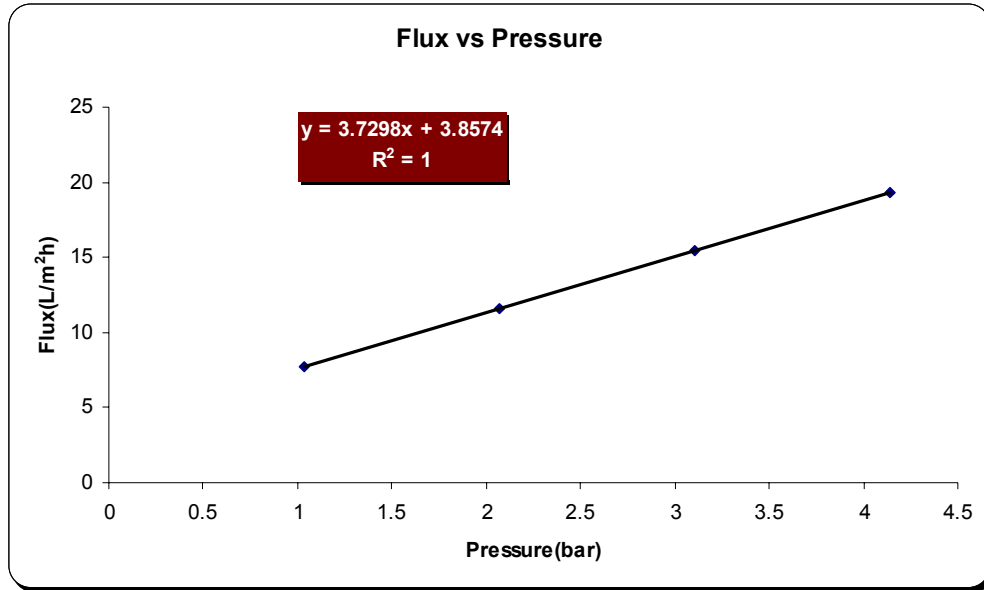
Figure 4.1 Variation of flux with change in pressure.

From the graph the initial membrane resistance was found to be $9.65 \times 10^{13} (1/m)$.

In order to observe the phenomenon of fouling. Test 2(chapter iii) was performed as described earlier. Membrane flux as a function of time is shown in figure 4.2 for tap water.
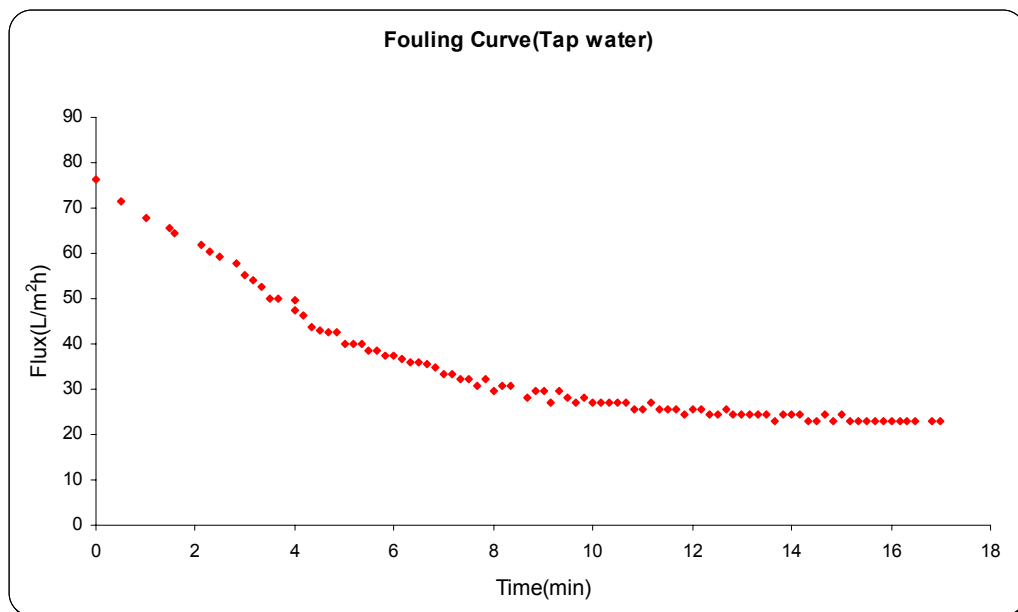

Figure 4.2 Fouling curve.

The observed flux decline might be due to the presence of species of molecular weight cut-off (MWCO) of more than 10000 in laboratory tap water. The effect of the pressure on the flux was also investigated in this study. The starting pressure was kept at 60psi and than it is decreased to 30 psi and again increased to 60 psi.Finally the pressure was reduced to zero. Flux was recorded throughout the filtration run. Figure 4.3 shows the variation in flux due to change in pressure. Indicated on the figure are the points when the pressure was increased or decreased. This experiment was performed using tap water for a period of 40 minutes.
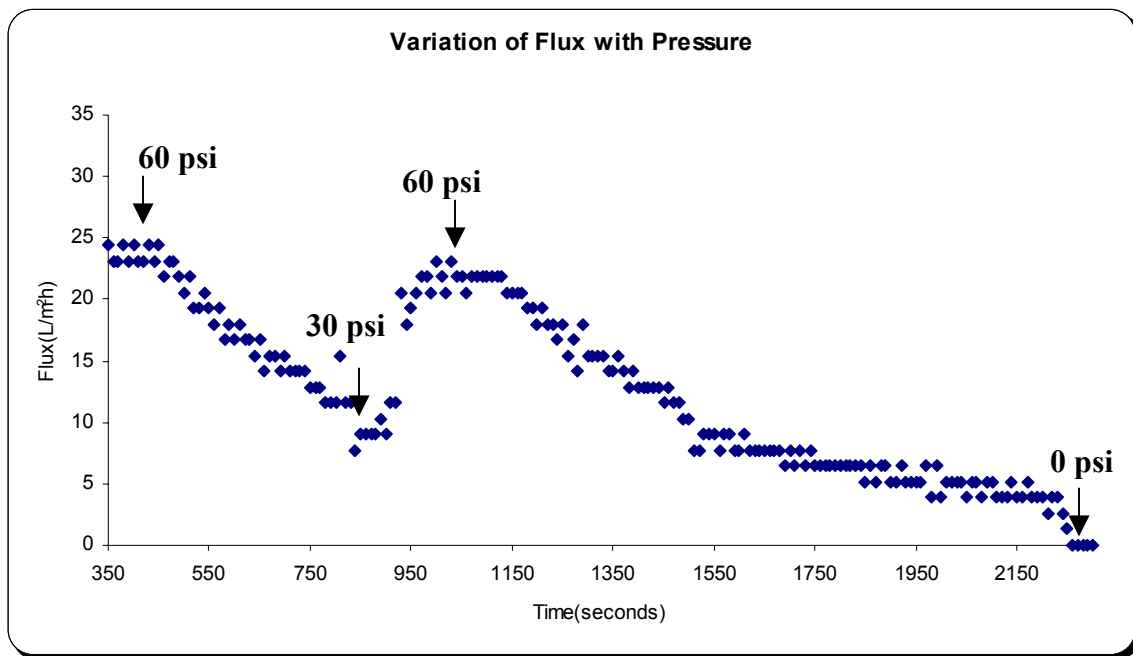

Figure 4.3 Variation of flux with change in pressure.

V. Conclusion & Recommendations

A novel lab-scale experimental setup,employing ultrafiltration membrane,was developed and employed to measure the flux across the membrane.It was configured with labview to get data in real time.Intital membrane resistance was calculated using pure water.The effect of change in pressure on flux and fouling phenomenon was also demonstrated using ordinary tap water.

In future this setup can be used for various applications. This unit can be ideal for removal or exchange of cells, nucleotides and protein, as well as other materials of high molecular weight. One of the most important areas where dead end filtration unit could serve as powerful tools is in molecular biology applications, such as DNA or RNA concentration and desalting procedures. Concentrating dilute DNA solutions is a key step for many subsequent preparative and analytical procedures. For example, standard plasmid preparations involving cesium chloride, equilibrium centrifugation, and gel filtration yield DNA in large volumes that require concentrating prior to precipitation. DNA concentration is also necessary in purifying restriction fragments from gels. Higher DNA/RNA recovery could be achieved using tighter membranes in this unit.

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