

Waste Water Labs

ENGI 24056

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LAB 2: ION EXCHANGE WASTEWATER TREATMENT

Introduction

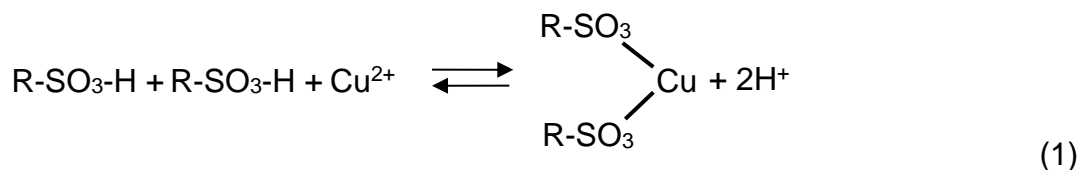
Ion exchange is commonly used to "soften" water as pretreatment for industrial uses by removing hard ions such as iron, calcium and magnesium. However ion exchange methods are now being applied to the treatment of industrial wastewaters to remove heavy metals as a recovery technology.

Ion exchange involves a reversible reaction whereby an ion in solution is exchanged for an ion attached to an immobile solid resin. A wastewater effluent containing contaminant ions is passed through the resin. The contaminant ions go onto the resin and non-hazardous ions (usually Na^+ , H^+ , Cl^- or OH^-) come off the resin and into solution. Heavy metal waste streams contaminated with copper, nickel, zinc or chromium are typically amenable to ion exchange treatment and recovery.

The solid ion exchange resins are typically made of naturally occurring inorganic zeolites or synthetically produced organic macromolecules. The latter type is commonly used in wastewater treatment. Organic resins can be synthesized to vary the number of ion exchange sites per unit volume of resin. A good ion exchange resin should be resistant to the solvent actions of water, acids and alkalis and should have good mechanical stability. Ion exchange resins may be either anionic (capable of exchanging negatively charged anions) or cationic (capable of exchanging positively charged cations).

Organic resins are typically made from copolymers of styrene and di-vinyl-benzene or from a phenol-formaldehyde condensation. Resin beads normally are 0.25 to 1.0 mm in diameter and exchange takes place not only on the resin surface but also in the resin pores.

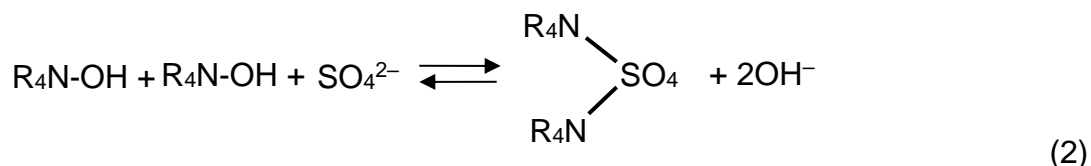
The active exchange sites on a typical strongly acidic cation resin occur at sulphonic groups ($-\text{SO}_3\text{H}$) as shown below, where copper ions are exchanged for two hydrogen ions.



"R" is the resin backbone or chain.

These exchange reactions are reversible and so are affected by solution concentrations, flow rate of solutions through the resin, solution temperature, contact time in the resin bed and affinity of the particular resin for a particular ionic species.

The active exchange sites on a strongly basic anion exchange resin are often quaternary ammonium ions. Below, OH⁻ ions from the resin are exchanged for SO₄²⁻ ions from solution.



Ion exchange resin beds can be placed in series with a cation resin bed followed by an anion resin bed. Sometimes resins are mixed together in a single bed. In either case, the effluent is pumped through the resin bed(s) where contaminant cations and anions are removed from solution and exchanged for relatively non-hazardous hydrogen and hydroxyl ions respectively. This exchange process is allowed to continue until all or most of the resin beds are exhausted. At this point, almost all of the hydrogen (H⁺) and hydroxyl (OH⁻) ions have been exchanged and the resin is saturated with contaminant ions. This condition is BREAKTHROUGH and the resin is no longer useful. At breakthrough, contaminant ions pass through the resin and are not exchanged. Generally, most systems are only allowed to reach 70% of the way to breakthrough. At this point the ion exchange resin is removed from service and "regenerated" by pumping concentrated acid through the cation resin and base through the anion resin. This has the effect of washing the contaminant ions off the resin allowing them to be collected in concentrated form for recycling or appropriate disposal. The resin is regenerated as H⁺ and OH⁻ ions reattach to the resin active sites. In effect, regeneration is achieved by forcing the reverse reaction in the two chemical equations above. It is not economical or in many cases even possible to regenerate a resin completely. Most regeneration only achieves 60 to 90% of original resin capacity. After regeneration the resin is normally washed by pumping water through the resin bed. Over time and after many regenerations, most resins will need to be replaced with fresh resin.

Flow rates in typical resin columns during normal ion exchange operations are 0.15 to 0.50 m³/min/m² of bed area (4 to 12 GPM/ft²). Typical space velocities are 0.03 to 0.7 m³/min/m³ of bed volume (0.25 to 5 GPM/ft³).

In this experiment, a cation resin and an anion resin in series will be used to remove copper(II) sulphate (CuSO₄) from a waste solution. Depletion of copper ions from solution during the ion exchange process will be monitored by measuring solution conductivity in micro Siemens/cm (μS/cm) with a HACH conductivity meter.

The overall ion exchange process is given by equations 1 and 2 above for this experiment.

Copper waste effluents are a common by-product of metal plating industries.

Purpose

The purpose of this experiment is to:

- 1) Study the process characteristics of an ion exchange system,
- 2) Evaluate the relationship between conductivity of the solution and copper sulphate concentration in the solution,
- 3) Determine the influence of flow rate on the performance of the ion exchange system.

Equipment

- 1) One cation/anion series ion exchange system (See Figure 1).
- 2) One HACK conductivity meter.
- 3) 30 L of copper effluent solution at a concentration of about 25 mg/L (25 ppm).

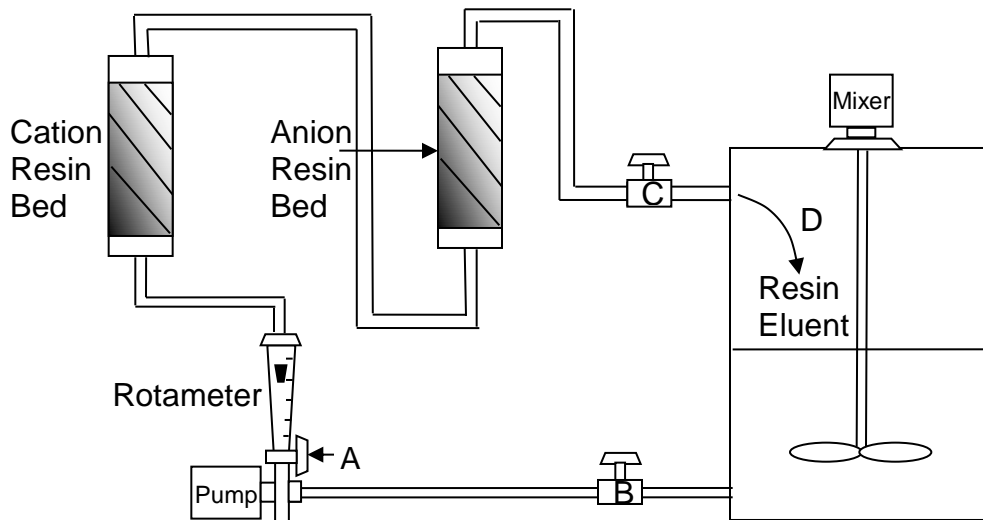


Figure 1: Ion Exchange System

PROCEDURE

1. Fill the "copper effluent tank" to the 30 L level with tap water.
2. Before adding copper to the tank, take an 18mL sample of the tap water from the tank. Save this for further analysis. Weigh 2.9 g of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ using the plastic weighing boats and the top loader electronic balance provided. Be sure to be precise to 2 decimal places. Completely dissolve the 2.9 g of copper sulfate pentahydrate in about 300 mL of water in a beaker and then add it to the 30 L of water in the copper effluent tank and turn on the mixer at slow speed. You have now produced a solution that is 25 mg Cu^{+2} /L. In your report, verify this by calculation. This is your copper effluent solution, which is well in excess of the sewer use bylaw limit of 3 mg Cu^{+2} /L.
3. Take a sample of solution from the copper effluent tank using the 8 inch sampling test tube provided.
4. Measure the conductivity of the solution in the copper effluent tank using the HACK -Model 44600 Conductivity/TDS meter as described below (also read pages 1,2,3,9,10 from the HACH manual provided).
 - a. Press the POWER I key and then the CND key (if a LO BAT message appears inform the instructor).
 - b. Select the range 2 MS/CM (microsiemens/cm) which is the basic unit of conductivity.
 - c. Insert the probe into the reference solution of 1000 microsiemens/cm and check that it is well calibrated ($\pm 2\%$). Insert the probe about 2 inches. Move the probe up and down a little to ensure there are no air bubbles trapped on the probe tip. Allow about 25 seconds for the conductivity reading to stabilize. If the measured value does not agree with the calibration value, notify the instructor.
 - d. To measure each sample, insert the probe into the sampling bottle. Move the probe around a little to ensure there are no air bubbles trapped on the probe tip. Allow about 25 seconds for the conductivity reading to stabilize.
 - e. Gently rinse the probe with distilled water from a wash bottle after the measurement is complete.
 - f. Once finished, return the probe to the water storage bottle.
5. Turn on the mechanical mixer (follow procedure from the orientation). The stirrer can be left on at very low speed throughout this experiment.

6. Refer to FIGURE 1 and open valves B and C completely. Open valve A only slightly since you are going to begin your first run at a very low flow rate of solution through the resin columns. With these valves adjusted in this way, the copper solution will now be able to flow from the tank through the pump and rotameter, through the two resin columns and then back to the tank.
7. Plug in the 1/15 hp. magnetic drive pump and quickly adjust valve A to produce a flow rate of 1.2 LPM as indicated on the rotameter. Read the rotameter at the widest part of the float. (Do not open valve A completely at any time since this will force the resin bed to the top of the column in a "plug"). Solution should now be moving slowly up the resin bed, wetting the bed slowly.
8. Once some solution begins to recycle to the tank at point D in FIGURE 1, start timing. **At 5 minute intervals over a 30 minute period take the following samples.**
 - a. Copper effluent sample from the tank: Take your tank sample exactly as you did in step 3 above. Transfer to a 30 ml previously labeled Nalgene bottle. Save your samples for later analysis.
 - b. Eluent returning to tank: Take a sample of the eluent at point D and save it.
 - c. Measure and record the conductivity of the collected samples using the HACH meter as described in step 4 above.
9. Measure the height and circumference of the resin bed and note any other bed behavior during the run. Unplug the pump and stop the stirrer. Close valves B and C. Use the cart pump provided to empty the contents of the copper effluent tank to the floor drain at the far end of the lab. This is acceptable since the copper content of this solution should now be below 3 mg/L.
10. Repeat steps 1 to 9 but this time, set the solution flow rate to 2.4 LPM using valve A (Note: to achieve this flow rate, open valve A very, very slowly since sudden surges of solution through the bed will cause the bed to rise in the column in a plug).
11. Clean up any spills at the end of this run and return all equipment to its original location.

Note: There will be some solution left in the rotameter and resin columns. This is acceptable since the resin should be left wet.

REPORT

1. For each flow rate, a graph of the conductivity of each solution (tank and eluent) versus time.
2. Calculate for each flow rate the:
 - a. "linear velocity" through the resin bed in (m^3 of solution/minute/ m^2 of bed area).
 - b. "space velocity" through the resin bed in (m^3 of solution/minute/ m^3 of bed volume).
3. Compare and discuss your experimental results between the two solution sets taken in each run, and between the same solutions taken each in run at different flow rates. Try to explain your results. Why does conductivity change, even though ions are replaced by other ions? Comment on bed "behaviour" at the two different flow rates and compare resin performance for the two flow rates.
4. Look up in the chemistry literature about how to convert conductivity values into concentration values. What equation can you use and what parameters do you need to know about the ions? Is the relationship between conductivity and concentration linear? Does it matter which ions are in solution or do all ions result in the same concentration versus conductivity relationship? Does pure water have a conductivity value, or is it zero?

LAB 3: WASTEWATER NEUTRALIZATION

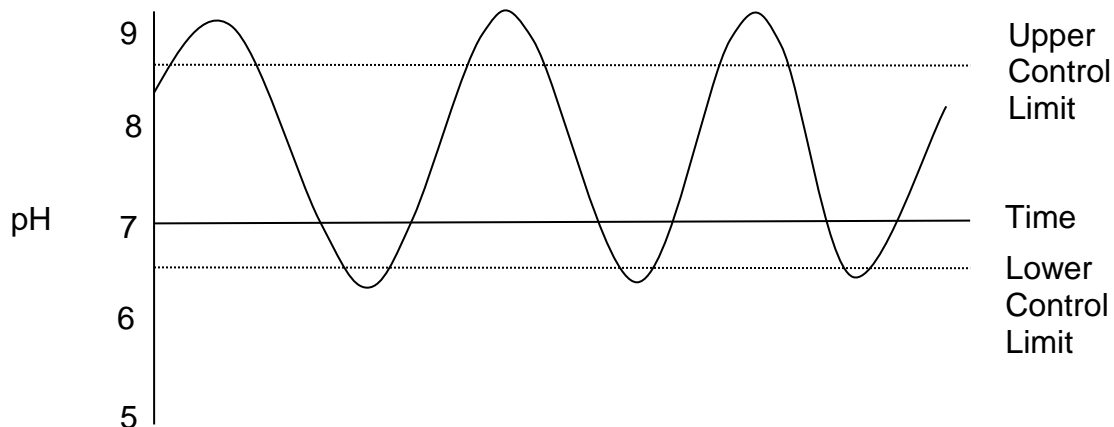
Introduction

One of the most common requirements for chemical treatments of wastewaters is the neutralization of excess acidity or alkalinity. pH is the indicator of acidity or alkalinity of a solution. Wastewaters with pH values above or below the range 6 to 10 may be corrosive to equipment and can cause serious effects on fish and plants if discharged to waterways. Discharges to the environment must typically fall between pH 6 and 9.5 according to most municipal sewer use by-laws. The effluent from most industrial processes tends to consist of a mixture of contaminants such as solvents, oils and grease, dissolved solids, heavy metals and the pH can range typically from 0 to 14. As well, the pH of a wastewater effluent can jump up or down rapidly as upstream events occur such as a process change or the cleaning of a tank, (etc). Because many wastewater streams are a mixture of contaminants they very often require a number of treatment methods such as oil skimming, emulsion breaking, heavy metals removal, BOD control (etc). In many of these treatment processes, pH control is also required and in any case, final pH adjustment to near neutral conditions is required prior to sewer discharge.

Neutralization of a wastewater stream involves the addition of acid or base reagents using a chemical feed pump connected to a pH Indicator-Controller (pH I/C). Common neutralizing chemicals are caustic soda, lime, calcium or sodium carbonate for acid wastes. For alkaline wastes, sulphuric, hydrochloric and nitric acids can be used. Selection of reagent depends on quantity needed, cost and by-product formation such as an undesired sludge. The neutralization usually occurs in a stirred tank as either a batch or continuous flow process. pH in the tank is monitored by a pH probe which sends signals to the pH I/C. The pH I/C can be adjusted to have an upper pH limit and a lower pH limit. If the measured pH falls outside of these limits, then the pH I/C will activate one of the chemical feed pumps to add acid or alkaline reagent to the tank as appropriate in an attempt to bring the pH back within the pre-set limits, (see Figure 1). This neutralization process is complicated by several factors as you will see in this lab:

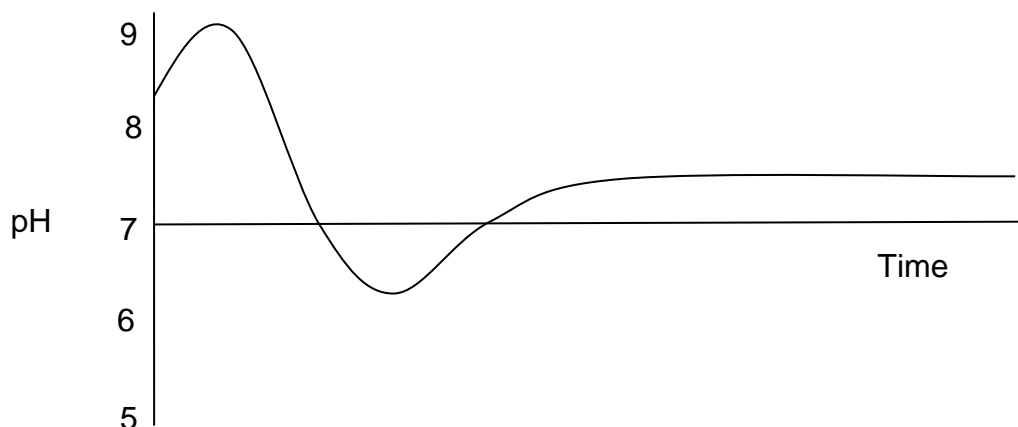
- a. In most cases there will be a "lag time" between the addition of a reagent and the response of the pH I/C to this addition. Consequently, the reagent feed pump will continue to operate for a period of time longer than necessary and so the volume of reagent added will exceed the quantity required. If concentrated reagents are used or if they are added at high flow rates it is possible that the pH will jump beyond the control point. In response, the system will initiate the injection of the other reagent and this might in turn cause the pH to move back outside the other control point. In extreme situations, the system may loop back and forth indefinitely resulting in excessive reagent consumption, wear and tear on equipment and overall lack of good pH control as shown below.

Figure 1: Excessive Cycling



The best situation is one where the pH rapidly stabilizes within the upper and lower control limits as shown below.

Figure 2: System in Control



- b. While it might seem logically that the best control can be achieved using dilute acid or base reagents added at low flow rates, this approach can also give unacceptable results in certain cases. In a continuous flow system, the "RESIDENCE TIME" (t) in a tank is expressed as $t = V/Q$ where:

V = tank volume, m^3

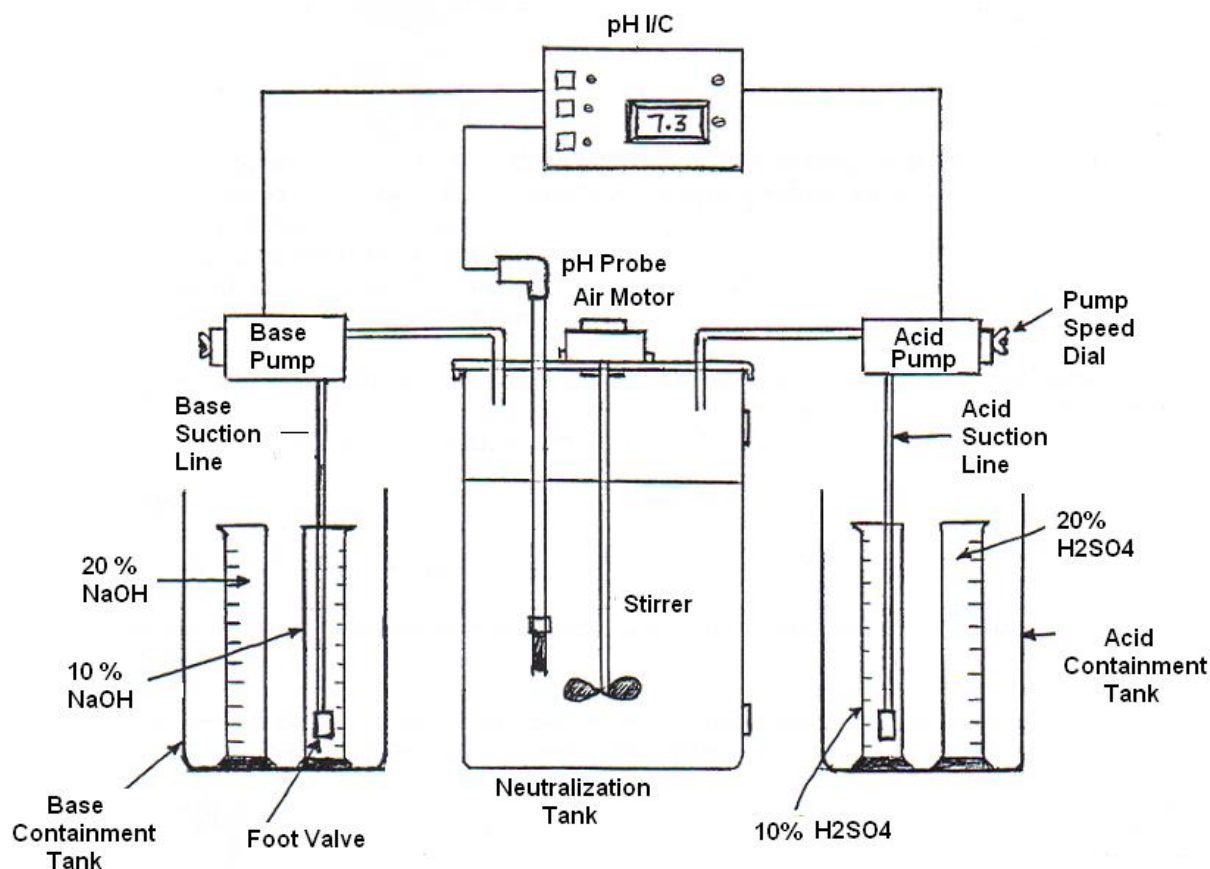
Q = flow rate of effluent into the tank, m^3/min

The pH adjustment must be fully completed while the "slug" of effluent is in the tank and so the reagent must have sufficient strength to affect the pH change within this residence time. Thus, reagent strength and the rate at which it is added is often a compromise and determined by experimenting with the particular system in use.

- c. In many industrial situations, the pH in a neutralization tank can be suddenly upset due to the addition of a strong acid or alkaline solution. This could result from suddenly discharging a cleaning line into the neutralization tank causing the pH to jump from 1.5 to 10.7. Then an acid etch solution might be discharged to the neutralization tank causing the pH to suddenly drop to 3. Thus, the pH control system must be capable of handling these sudden changes and returning the system back quickly to a stable pH within the control limits.
- d. Most sewer use by-laws allow effluent discharge in the range of 6 to 9.5. Thus, it would be safe to set the lower and upper pH control limits at 6.5 and 9.0 respectively. However, in other cases, much tighter pH control may be required to achieve certain chemical reactions such as the precipitation of nickel out of solution which occurs most effectively over the pH range of 10 to 11.

Thus, the variables affecting the process are: reagent feed rate, reagent concentration, mixing rate in the neutralization tank, tank volume and effluent residence time, upper and lower pH control limits, location of pH probe and lag time in the system.

Figure 3: Neutralization Apparatus



As can be seen from Figure 3, for this experiment you will be using feedback control on a batch neutralization tank. However, other neutralization control systems are also used in industry. These include:

Figure 4: Continuous "feedforward" control

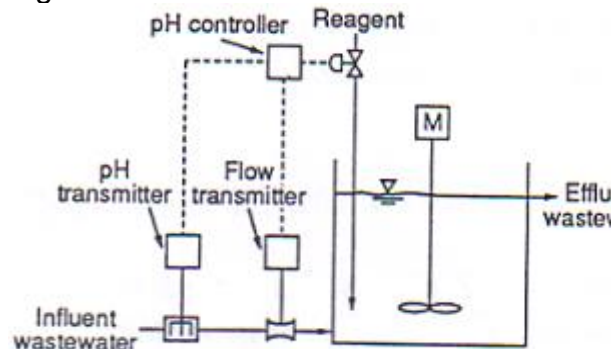


Figure 5: Continuous "feedback - feedforward" control

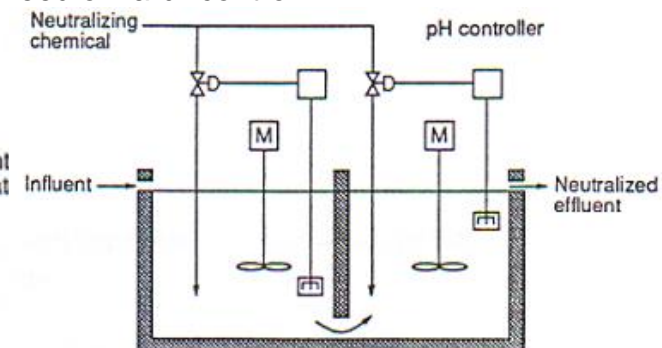
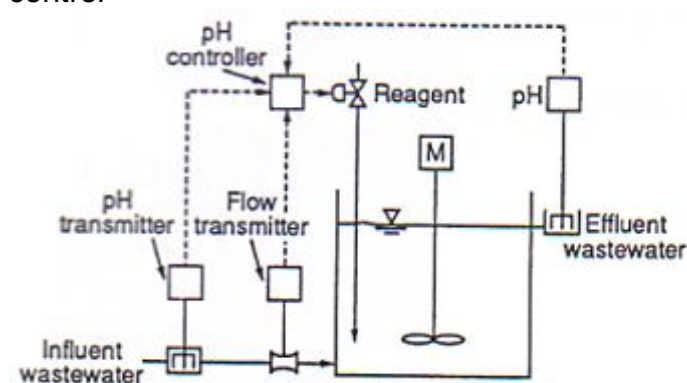


Figure 6: Continuous two stage feedback control



CAUTION: This experiment involves the use of 10 w/v% H_2SO_4 and NaOH . Both reagents are hazardous and can burn the skin. Safety glasses must be worn for this lab. Rubber gloves must be worn when handling any acid or base reagent.

Purpose

The purposes of this experiment are to:

- Identify the lag time between reagent addition and pH I/C response in a stirred, batch neutralization tank.
- Determine the influence of reagent concentration, reagent flow rate, span of control and agitator speed on the performance of the neutralization system.

Equipment

- One stirred neutralization tank (or reactor).
- One pH I/C with pH probe and buffers for calibration.
- Two CHEM-FEED peristaltic diaphragm reagent pumps (one for acid and one for

base reagent).

- 4) 10W/W% H_2SO_4 and NaOH reagent solutions.
- 5) 20 W/W% H_2SO_4 and NaOH reagent solutions.
- 6) Data acquisition computer (Madge Tech 4)

Procedure (Refer to Figure 1)

- 1) Fill the neutralization tank to the 80 litre level with tap water. This tank may already contain water and may only need to be topped up.
- 2) Ensure the acid and base suction line foot valves are well below the 10% concentration reagent liquid levels, as shown in Figure 3 (this may already have been done).
- 3) Carefully raise the pH probe out of the neutralization tank and turn on the pH I/C unit by plugging in the electrical cord.
- 4) Calibrate the pH probe by immersing the pH probe tip into the buffer pH 7 solution provided and pressing CAL until the measured pH value is shown. Wait until the measured pH value stabilizes, then press CAL again to set the measured value to 7.00.

Remove the pH probe from the buffer 7 solution and rinse the pH probe tip with RO water from the wash bottle provided and wipe with Kimwipe.

- 5) Immerse the pH probe tip into the buffer pH 4 solution provided. Press CAL to see the pH reading, wait for the reading to stabilize, then press CAL again to set the measured value to 4.00. Press CAL again to exit the calibration mode. Be careful not to press the CAL button during the experiment to avoid the risk of losing the calibration and having to repeat it. If you wish to check that the calibration was done correctly, immerse the wiped pH probe in the buffer pH 7 solution and note if the reading obtained is correct.

Return the pH probe to its original position in the neutralization tank. The pH I/C unit should now be reading the pH of the water in the neutralization tank. Now see Table #1 for the operating parameters for each of the runs in the lab.

- 6) Turn on the stirrer to the desired speed (see Table 1); ask instructor for guidance.
- 7) Make a table to manually record the following data for each run (a data acquisition computer connected to the pH/IC unit will record pH versus time for you):
 - a. Time elapsed between the addition of the NaOH and the activation of one of the pumps. This is the "lag time" of this particular system.
 - b. Total time in seconds that each pump is on during the run (no need to reset stopwatches, just stop and re-start to get total pumping time).

- c. Number of times the system cycles between the acid and base feed pumps (this can be interpreted from the pH data graph).
 - d. Length of time it takes for the system to stabilize (i.e. to achieve a pH that remains continuously between the upper and lower pH control limits that you set earlier). If the system does not stabilize after 10 minutes then stop the run. In this case, manually dose small quantities of acid or base to return the solution pH to near neutral before starting the next run.
 - e. Volume of acid and base reagent added to the neutralization tank in order to achieve a stable pH as described in d) above. To determine this, simply multiply the time a pump is on by the flow rate of the pump at that particular setting. Note the pump flow rates for each pump setting from the calibration curves posted on the wall.
- 8) To set the lower pH set point (according to Table 1), press SP1, then press up or down arrows to adjust the pH value. Press ENTER when done.
 - 9) To set the upper pH set point (according to Table 1), press SP2, then press up or down arrows to adjust the pH value. Press ENTER when done.
 - 10) Adjust the acid and base reagent feed pumps (CHEM-FEED) to the desired setting (Table 1) using the dial located on the side of each pump. By referring to the pump calibration curve provided, record the expected flow rate for each pump in ml/min. You will have to loosen the wingnut on the side of the pump to adjust the pump setting. Ensure the gear sprocket is well positioned before re-tightening the wingnut.
 - 11) Check that there are no air bubbles in the acid and base suction lines. Bubbles will prevent the pump from working correctly. If you notice bubbles, they must be pumped out from the line using the highest pump speed before continuing with the run; ask your instructor for guidance.
 - 12) Check the pH of the tank solution. If it is not approximately neutral (7.0 to 8.0), manually adjust the pH using small additions of 10% acid or base before continuing with the run. After each "small" addition, wait for the solution to mix well before deciding whether to keep adjusting.
 - 13) Prepare the computer to record data. Press Real Time Start, then press Start, then type run name. When ready to start recording (i.e. at the moment of solution spiking), press OK.
 - 14) Spike the solution adding 100 mL of 10% strength acid or base (see Table 1) to the neutralization tank. This addition simulates a sudden surge of alkaline or acidic wastewater effluent entering the neutralization tank. The pH in the tank should rise/fall dramatically, to outside the set point range to selected earlier. This sudden change should therefore activate the pH neutralization control system.
 - 15) Once the pH has stabilized between the pH control limits, or the time limit has been reached, stop data acquisition. Export the run data to Excel and save the Excel file.

16) Repeat steps 8 through 15 for each of the remaining run conditions on Table 1.

Make sure you acquire a good RUN 1, as you will be comparing all other runs to this first run.

Table #1

RUN	Upper pH Limit	Lower pH Limit	Pump Setting	Stirrer Speed	H ₂ SO ₄ and NaOH Strength	Spike solution (100ml, 10%)
1	9.0	6.0	2	Slow	10%	NaOH
2	9.0	6.0	2	Slow	10%	H ₂ SO ₄
3	9.0	6.0	4	Slow	10%	NaOH
4	9.0	6.0	2	Higher	10%	NaOH
5	9.0	6.0	2	Higher	20%	NaOH

At the end of all the runs, turn off the pH I/C unit and stop the stirrer. Empty the contents of the neutralization tank (if not neutralized during the run, manually neutralize) to the floor drain using the cart pump provided. Since the neutralization tank contents are within the sewer use by-law limits, discharging to the drain is acceptable. Refill the neutralization tank to the SOL mark with tap water. Carefully raise the foot valve on the acid and base suction lines so that they are slightly above the reagent levels in the acid and base containers.

Note: the pH probe must be left sitting in the tank in water of pH between 6.5 and 7.5 or the probe will be damaged. Also, set the upper control limit to 12 and the lower control limit to 2 on the pH I/C unit. (This will ensure that the pumps will not be activated when the next group of students tries to calibrate the pH probe at pH of 4 and 7).

Clean up any spills and return all equipment to its original locations. Ask the instructor to inspect the apparatus before you leave.

REPORT

As part of your full report be sure to address the following:

- 1) For each run graph pH versus time (this is really just your Excel data). Be sure to indicate the upper and lower control limits on your graph as well.
- 2) By comparing the results of RUN 1 and RUN 2, comment on how different the process behaves when hit with a sudden basic versus acidic spike.
- 3) By comparing the results of RUN 1 and RUN 3, discuss how acid and base reagent flow rate affects the performance of the process.
- 4) By comparing the results of RUN 1 and RUN 4, discuss how mixer speed affects performance of the process.

- 5) Compare RUN 1 and 5 and discuss how reagent strength affects system performance.
- 6) Compare all five runs in terms of a) how often the system cycled between the acid and base feed pumps, b) the volume of acid and base consumed for each run, c) the time it took for the process to stabilize, and d) the influence of lag time on each run. Try to explain your results.
- 7) For each run, calculate the "Theoretical Flow Rate" for this system. The Theoretical Flow Rate is defined as:

$$\text{TFR} = (\text{volume of liquid in tank})/(\text{time needed to complete the neutralization})$$
- 8) By comparing all five runs, suggest what you think would be the most cost effective set of operating conditions for this particular system.
- 9) Describe the problems that can result if a wastewater that is very acidic or basic is discharged through a sanitary sewer to a wastewater treatment plant.
- 10) What chemicals (other than sulphuric acid and sodium hydroxide) are sometimes used for neutralization of wastewaters? What factors influence the choice of chemical?
- 11) Identify any design changes you might make to the neutralization apparatus used in your experiment to improve the effectiveness of the process.
- 12) What ongoing equipment maintenance would you recommend for a pH wastewater neutralization system similar to the one you used in this lab?

LAB 4: CARBON ADSORPTION TREATMENT OF WASTEWATERS

Introduction

Carbon adsorption technology has long been used to treat drinking water but has now also become popular in the wastewater treatment field. Carbon adsorption is used to treat non-ionic contaminants such as insoluble metal oxides and many organic compounds. Carbon is also excellent for elimination of colour, taste and odour problems in water and wastewater streams. It is often biological activity that causes odour problems but organics such as aldehydes, mercaptans, amines, ketones also contribute to this problem. Adsorption is the collection of a substance onto the surface of the activated carbon. There is no chemical reaction involved. Activated carbon has numerous capillaries within the carbon particles that provide an extremely large surface area for adsorption. Thus, the ratio of surface area to mass for carbon is enormous. Activated carbon can be manufactured from coal, wood, petroleum based residues (etc) where the material is carbonized and then "activated" by exposure to hot air or steam.

There are two common forms of activated carbon - powdered and granular. Powdered carbon is more commonly used for seasonal problems in water supply systems or in municipal wastewater treatment by addition to primary clarifiers to reduce BOD and solids loads. However, granular activated carbon is more common for wastewater treatment and particularly where there are relatively high and constant organic contaminant levels. Granular activated carbon (GAC) has a particle size of 8 to 40 mesh. Flow rates through beds of GAC are usually in the range of $0.3 \text{ m}^3 \text{ solution/minute/m}^3 \text{ bed volume}$. Bed sizes can be quite varied. Typical bed heights range from 3 to 40 ft. Beds can be arranged in series and flow can be up the bed or down through the bed.

Fixed bed adsorption systems with a single carbon column are sometimes used (see FIG 1 below) but columns in series are more common because it allows one column to be backwashed without shutting down the treatment process. The backwash removes any solid particles that are trapped in the carbon bed that restrict flow. Backwashing may be conducted several times a day in a continuous treatment process.

With continued use, the available surface area for adsorption on the carbon will drop very low and no further effective adsorption will occur. This is called "breakthrough" because the wastewater contaminants breakthrough the carbon bed without being reduced in any significant amount. The carbon can then be regenerated by passing the spent carbon through a furnace to drive off volatile organics that were previously adsorbed onto the carbon.

In a carbon adsorption system the amount of pollutant (or solute) adsorbed per mass of carbon depends on the equilibrium or final concentration. This relationship is described by the Freundlich Isotherm as follows:

$$\log\left(\frac{x}{m}\right) = \left(\frac{1}{n}\right)\log Ce + \text{Log}K \quad (1)$$

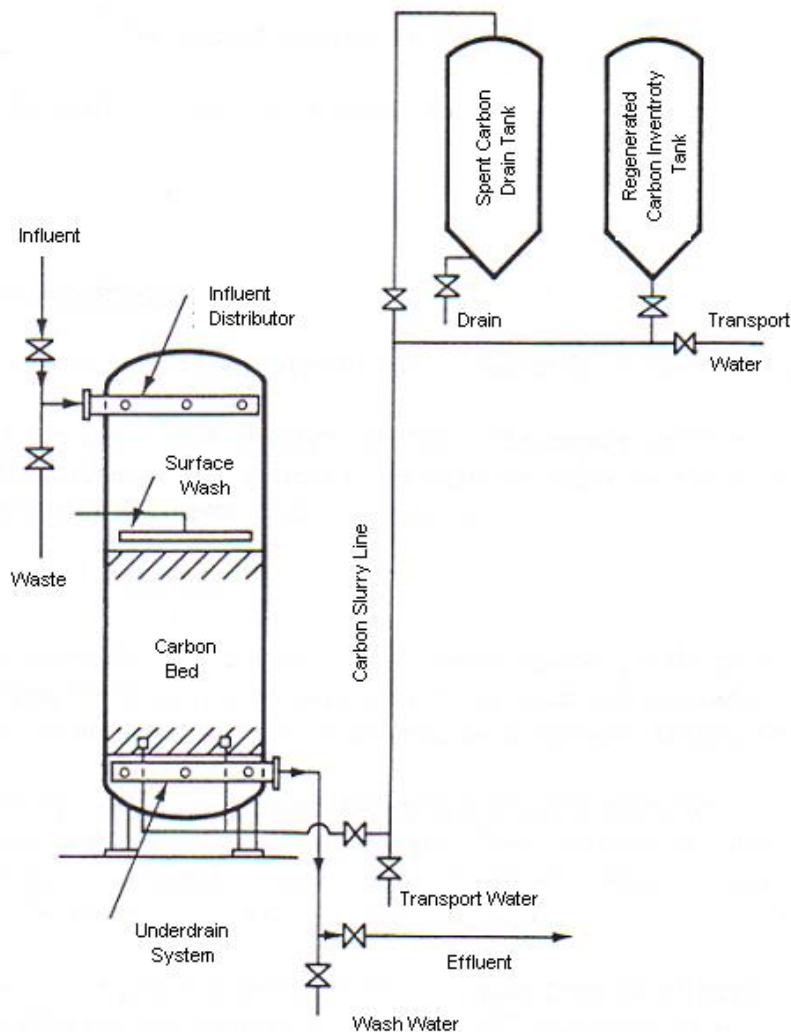
where: x/m = mass of solute adsorbed/mass of carbon

K and n are experimental constants

C_e = equilibrium concentration of solute (mg/L)

A plot of $\log(x/m)$ versus $\log C_e$ will produce a straight line of slope $(1/n)$ and intercept of $\log K$. This equation is useful for batch processes mainly or systems with long residence time where equilibrium between the adsorbed and un-adsorbed solute is achieved. However, this equation can be useful in making rough approximations about the amount of activated carbon needed in a continuous low flow system.

Figure 1: Fixed-Bed Adsorption System Using a Single Carbon Column



PURPOSE

The purpose of this experiment is to study the rate of removal of contaminant organic molecules from a waste stream using activated carbon at different treatment flow rates.

EQUIPMENT

- a) Activated carbon cartridge.
- b) Solution transfer pump, hoses and rotameter for flow control.
- c) One effluent tank with air driven agitator.
- d) 30 L batch of - methylene blue solution (an organic dye)
- e) Spectrophotometer for analysis of methylene blue samples.

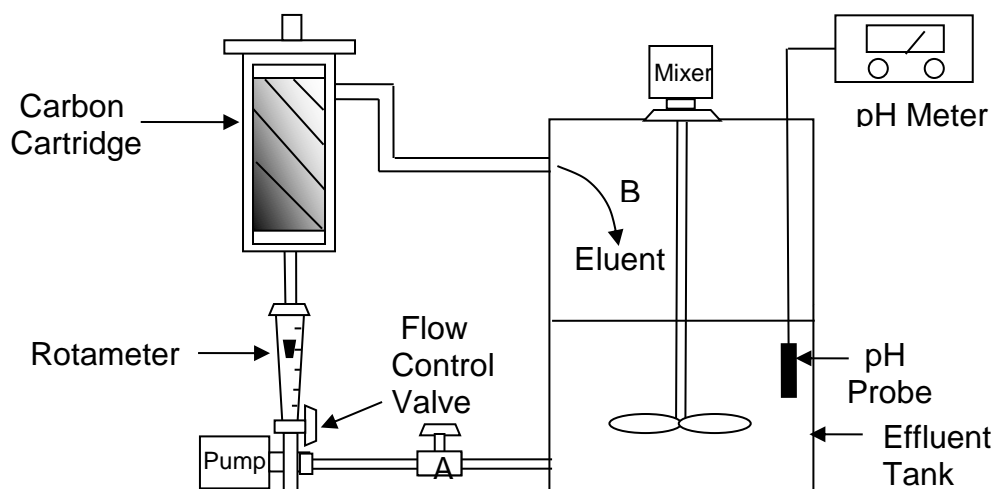
PROCEDURE

Removal of an organic dye - methylene blue

A carbon cartridge will be used to remove methylene blue contaminant from water.

- 1) Unscrew the top of the carbon adsorption chamber and remove the carbon cartridge. Describe its appearance. Return the carbon cartridge to the chamber and screw the top back onto the chamber securely ensuring the rubber gasket is in place.
- 2) Fill the effluent tank with 30 L of tap water.
- 3) To test for water leaks or air bubbles in the lines, open all valves and plug in the pump. The water should now be circulating from the effluent tank through the pump and rotameter to the carbon cartridge and back to the effluent tank. Check for leaks or air bubbles. Unplug the pump.
- 4) Slowly and without splashing, add 150 ml of the 1000 mg/L methylene blue solution to the 30L of water in the effluent tank and stir slowly to mix. Calculate the new concentration of methylene blue in the effluent tank in mg/L. (Note * methylene blue is a very water soluble organic dye. Do not spill it or get it on clothes or skin).
- 5) Using the 8" test tube (with clamp) that is provided, take a sample from the effluent tank and transfer it to the plastic container that has been pre-labeled as "t=0", methylene blue. Cap and save for later analysis using a Spectrophotometer.

Figure 2: Carbon Absorption Apparatus



- 6) Plug in the pump and adjust the rotameter valve until you get a flow rate of 2.4 LPM (Litres per Minute). Using the 8" test tube, take an effluent tank sample every 5 minutes for 30 minutes or until the solution becomes clear. Save each sample in the appropriate pre-labeled plastic container provided, cap and save for later analysis. At the 5-minute and 30-minute marks of your run, take a sample of the "ELUENT" at point B as shown in Fig 2. Transfer these to the containers labeled as "ELUENT-methylene blue". Save all samples for analysis (analyze samples as you obtain them).
- 7) Unplug the pump. Empty the tank contents to drain using the pump provided. Refill the tank with fresh tap water.
- 8) Repeat steps 2) to 6) above but at a flow rate of 4.8 LPM.
- 9) At the end of your run, unplug the pump and empty the tank contents to drain using the pump provided. Refill the tank with fresh tap water and plug in the pump to flush the water through the carbon cartridge for 5 minutes. Then empty the contents of the tank to drain. Leave the carbon cartridge in the chamber for the next group of students to use. Now proceed to the Analysis of your methylene blue samples as described below

ANALYSIS for Methylene Blue in Water

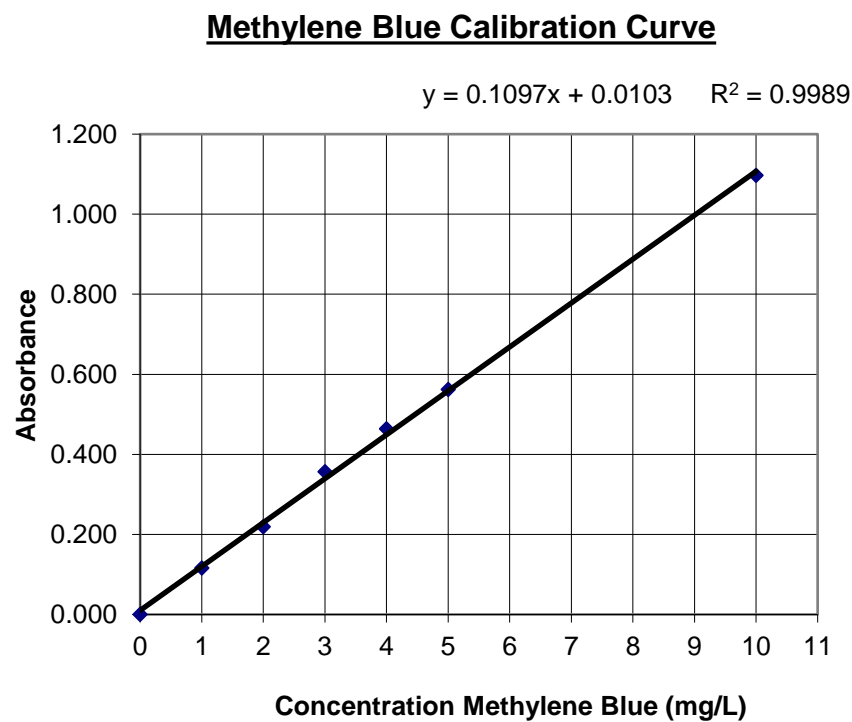
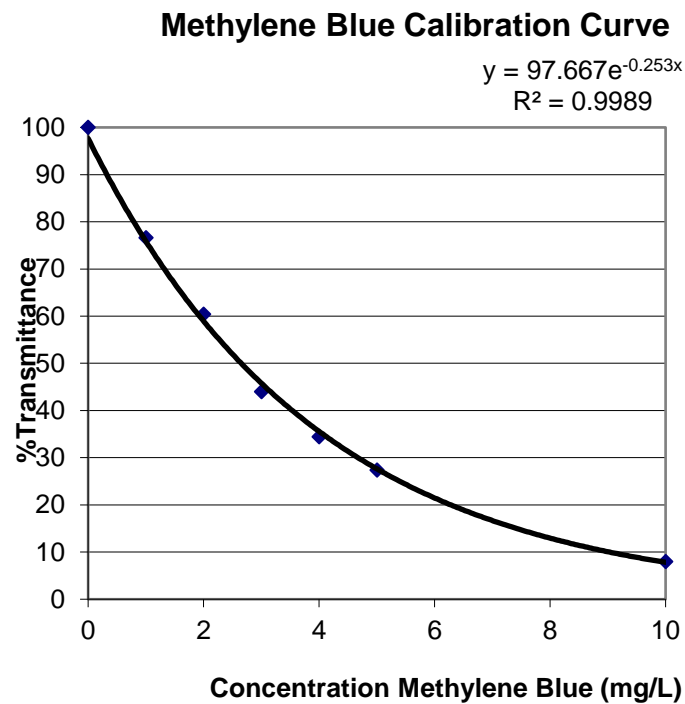
- 1) You will now analyze your methylene blue samples by spectrophotometry.
- 2) Turn on the SPECTRONIC-20 spectrophotometer and allow it to warm up for 15 minutes.
- 3) Set the wavelength to 660 nm on the instrument.
- 4) With nothing in the sample holder and the lid closed, use the left hand dial to set the meter to 0% Transmittance.
- 5) Place a "blank" solution of tap water in one of the cuvettes provided and insert into the sample holder. The white line on the cuvette should be lined up with the raised line on the sample holder. Close the lid of the sample holder and use the right hand dial to set the meter to 100% Transmittance. (Note - the cuvette must be wiped dry on the outside and be free of fingerprints. Grasp the cuvette only at the top).
- 6) Remove the "blank" and discard the water.
- 7) Rinse out the same cuvette with a small amount of your first methylene blue sample. Then fill the cuvette with the sample and insert into the sample holder and close the lid. Read and record the % Transmittance. Refer to the "standards calibration curve" provided to convert % Transmittance to mg/L of methylene blue.
- 8) Repeat step 7 for the rest of your methylene blue tank samples. Use the same cuvette for all your samples.
- 9) All your samples can be discarded in the "Waste Methylene Blue" container provided when you have finished your lab.

REPORT

- 1) Describe the appearance of the carbon cartridge and identify how the fluids move through the carbon in the cartridge (i.e. inside/out or outside/in)
- 2) Plot (mg/L methylene blue) versus (time) for your tank samples for each run. Discuss your results and compare the effectiveness of removal at the two different flow rates. Also compare the "Eluent" concentrations. What can you conclude?
- 3) If you were to continue to conduct more runs at increasingly higher flow rates, do you think the removal effectiveness of methylene blue would also continue to increase? Explain your thinking.
- 4) Explain why carbon cartridges are periodically backwashed during operation.
- 5) Research the relative advantages, disadvantages and uses of powdered activated carbon versus granular activated carbon in water treatment.

6) Research other environmental applications of activated carbon and list them.

Figure: Standard Calibration Curve for Methylene Blue 660 nm



LAB 5: JAR TESTING

Introduction

An important process in treating water and wastewater is the removal of suspended solids from the liquid. Course material is readily removed by sedimentation, but the smaller, finer particles called the colloids require additional treatment. These colloids are typically clay compounds, microscopic organisms and organic matter from decaying vegetation. These colloids range in size from 1 to 500 millimicrons and many are not visible even when using an ordinary microscope. A colloidal dispersion formed by these particles is stable in still water. They are so small that the effect of gravity is negated by the effect of colliding solvent molecules. Hydrophilic particles are stable because of their attraction for water molecules. Typical examples are soap, soluble starch and synthetic detergents. These types of colloids tend to be very difficult to remove from water. Hydrophobic particles (not attracted to water molecules) are suspended in solution because of their electrical charge. The majority of matter in a turbid natural water is of the hydrophobic type. Hydrophobic particles are held apart by the electrostatic repulsion resulting from the positive ions adsorbed onto their surfaces from solution (like two positive poles of a magnet that repel one another). Thus, they never clump together and fall out of solution.

To remove colloidal particles from solution, chemical coagulation is often used. In this process, metal salts (such as aluminum or iron sulphate) are added to reduce the repulsive forces between colloidal particles. Once the repulsive forces are reduced the particles can stick to one another and fall out of solution more readily due to gravity.

Another common method of removing hydrophobic colloids from water is by adding a coagulant aid or polyelectrolyte. These polyelectrolytes are anionic or cationic organic polymers that provide a "bridging effect" between colloidal particles by attaching themselves to the absorbant surfaces of colloids and thereby building larger flocculated masses. These larger masses then fall out of solution more readily due to gravity. Flocculation is the name of the process whereby the particles stick together and fall out of solution. Often metal salts and polyelectrolytes are used together to treat colloidal solutions. Refer to the figure on the next page.

These treatment processes depend on solution pH, temperature, contact time, concentration of polyelectrolyte used and mixing rate. Typically there is a short one minute "flash" mix of polyelectrolyte followed by 15 to 30 minutes of slow mix to allow flocculation to occur. Finally a longer period of 2 to 4 hours is allowed for complete sedimentation of all particles to the bottom of the tank as sludge. The sludge is then drawn off, dewatered in a filter press and disposed of. Even after all this treatment there may still be some stubborn colloidal particles left in the water that did not settled out. These particles would require removal by filtration as a next step. Given all the variables that can affect treatment of colloidal solutions, a series of experimental tests are conducted to determine the optimum treatment conditions. These are called "jar tests" and involve a variable speed mechanical stirring device, a set of 1-Liter containers, various polyelectrolytes and acids and bases to adjust the pH. The goal is to determine the optimum dosage of polyelectrolyte for the particular sample at a given pH and stirring rate. The optimum dosage is the minimum amount of polyelectrolyte needed to produce

good turbidity removal as measured by a turbidimeter. The results of these jar tests can then be used to estimate the best conditions and amounts of polyelectrolyte needed for a full scale water or wastewater treatment plant.

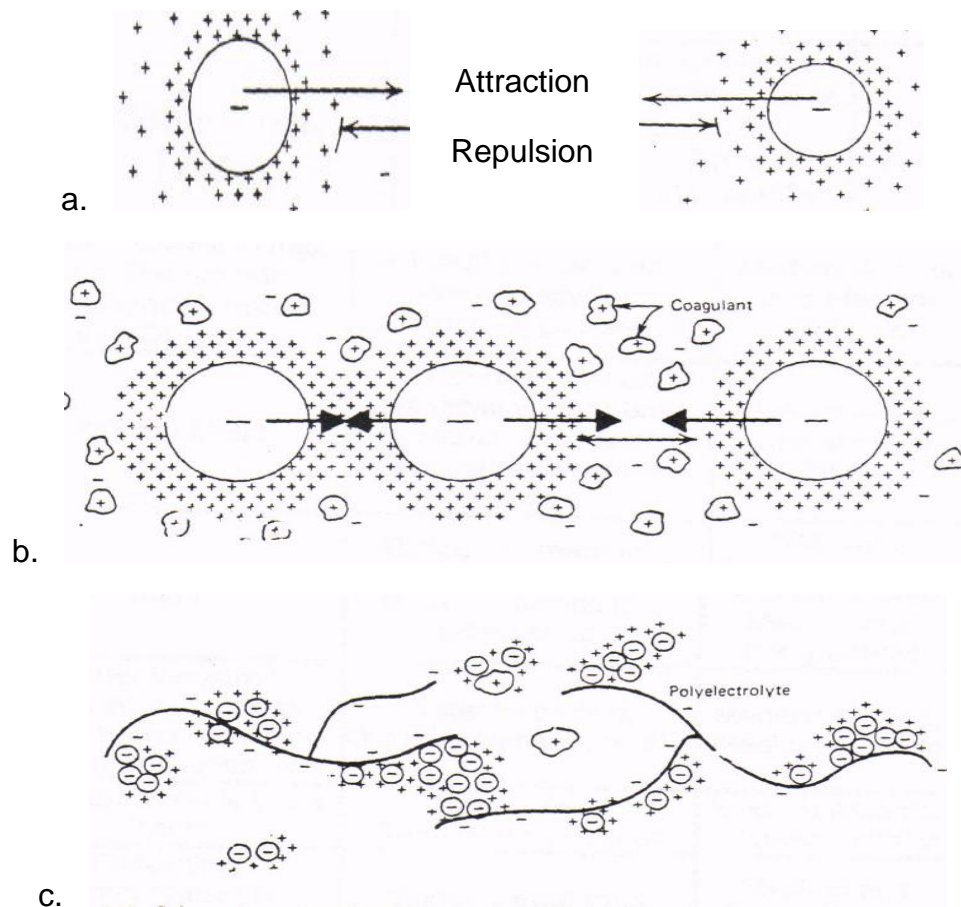


Figure 1 Schematic actions of colloids and coagulation, (a) Forces acting on hydrophobic colloids in stable suspension, (b) Compression of the double-layer charge on colloids (destabilization) by addition of chemical coagulants, (c) Agglomeration resulting from coagulation with metal salt and polymer aid.

Industry	Application	Charge
Foundries, Asphalt Plants, Cement Plants	Air/water scrubbers, settling ponds	Medium Anionic Medium Cationic
Food Processing, Vegetable Canning, Sugar Cane Processing, Fruit Preparation	Flotation and Settling Tanks, Clarification, Wash Water Systems, Biological Treatment Systems.	Non-ionic, Medium and Strong Anionic
Metal Plating or Finishing	Treatment for Heavy Metals Removal, Copper, Zinc, Iron, Nickel, Cadmium, Chromium Removal, etc., and Oil Recovery.	Non-ionic, Medium Anionic
Sand & Gravel Plants, Coal Preparation, Limestone Quarries, Iron Ore, etc.	Mineral Washing and Water Recycling Operations, Dewatering	Medium Anionic and Medium Cationic
Primary Metal	Oil Recovery, Influent Clarification, Scrubber Systems, Thickening, Biological Treatment Systems.	Medium Anionic and Medium Cationic
Paper	Biological treatment, Influent Clarification, Dewatering.	Non-ionic, Medium Anionic, Medium and Strong Cationic
Other Industrial Operations, such as Glass Plants, Chemical Plants, Refineries, etc.	Water Recycling, Oil/water Separators, etc.	Medium Anionic, Medium Cationic
Municipal Potable Water: Plants	Drinking Water Clarification, Filtration	Medium Anionic, Medium Cationic
Thickening, Centrifugation or Flotation	Sludge Dewatering	Medium and Strong Cationic

Table 1: Industry vs Application of Polymers

Purpose

The purpose of this experiment is to:

- 1) Observe how a polyelectrolyte is used to treat a clay suspension in water.
- 2) Determine the optimum dosage for this polyelectrolyte and suspension.

Equipment

- 1) Seven, pre-labeled 1- Liter beakers
- 2) One jar test apparatus (mechanical stirrer)
- 3) 20 or 25 ml graduated cylinder
- 4) a set of pipettes and suction bulb
- 5) One pH meter with buffers of pH 4 and 10 for calibration
- 6) One HACK 2100N Turbidity meter
- 9) One mortar and pestle set
- 10) cationic polyelectrolyte
- 11) mixing pail and stir stick.

Procedure

- 1) The following polyelectrolyte solution has been prepared for you: Cationic polymer - 200 mg/L (or 0.02%)
- 2) Weigh about 55 g of the clay and then grind it to a fine powder in the mortar and pestle.
- 3) Prepare a clay suspension by weighing 50 g of the clay powder and mixing it with 16 L of tap water in the pail provided. Note that this suspension must be stirred frequently during the lab.
- 4) Locate the seven, numbered, 1-L beakers. Add the clay suspension to the 800 mL mark in each beaker. Be certain to stir the clay suspension thoroughly before adding it to the beakers.
- 5) Measure the turbidity and pH of the clay suspension in beaker #7 and assume it is representative of the other six beakers. The turbidity meter does not need to be calibrated. (NOTE: Use the buffer solutions of 7 and 4 to calibrate the pH meter. Ask instructor if you are not familiar with calibration of pH meters).

To measure the turbidity, fill the sample cell provided (glass vial with screw cap) to the white line with the clay in water suspension. Do not touch the sample cell on the glass surface since your fingerprints will interfere with the turbidity readings. Gently wipe the glass surface of the cell with a Kimwipe to remove

any water or fingerprints. Place the sample cell into the sample compartment of the turbidimeter. Be sure the white arrow on the sample cell is facing you. Close the lid of the compartment and allow a few moments for the turbidity reading to stabilize. Record the turbidity reading in NTU (Nephelometric Turbidity Units).

- 6) Place the remaining six beakers on the mechanical stirrer and set the stirrer speed to 80 RPM. Turn on the stirrers. Turn on the light under the beakers.
- 7) Now you are going to add cationic polymer in varying amounts to the six beakers. However, the addition of the cationic polymer must be done at the same time to all the beakers! Using the various graduated cylinders provided, measure out the following volumes of cationic polymer: 0.0 mL for beaker 1, 4.0 mL for beaker 2, 8.0 mL for beaker 3, 12.0 mL for beaker 4, 16.0 mL for beaker 5, and 20.0 mL for beaker 6. With the help of your lab partner, add the cationic polymer volumes to the appropriate beaker. These additions must be done at the same time. Try to avoid hitting the stirrers since splashing may occur. Start timing at once.
- 8) After the polymer addition, stir the beakers for one minute at 80 RPM. Then reduce the stir speed to 30 RPM and continue stirring for 10 minutes. For each beaker, record the time when the first particles begin to settle out to the bottom of the beaker. This may happen quickly. These 'floe' particles may be very small but will appear as a discreetly visible particle.
- 9) After the 10 minute stir at 30 RPM, stop the stirrers and observe the nature and settling characteristics of the floe in each beaker. Rank the beakers in terms of observed turbidity where 1 is low turbidity and 6 is high turbidity. This is a very subjective assessment of course.
- 10) Allow the contents of each beaker to settle for 10 minutes. Pipette out sufficient supernatant from each beaker to test for pH and turbidity. About 100 mL of supernatant should be enough for these tests (use a graduated cylinder to measure this). Complete a table similar to Table #1.
- 11) Clean the beakers thoroughly using soap and test tube brushes. Otherwise the clay will stain the glass of the beakers. Also wipe off the stir paddles very carefully so as not to bend them.

Report

Write a complete report. Present all your results in tabular form.

- 1) State your observations upon addition of the polyelectrolyte (polymer) to the clay suspension.
- 2) What is the optimum polymer dosage?
- 3) How accurate is the result of this test and what factors would be different in an actual full scale treatment plant?

- 4) Plot turbidity versus polymer dosage. Comment on the shape of the graph.
- 5) In a water treatment plant, at what stage is a coagulant such as a polyelectrolyte added to clarify the water?
- 6) Describe an experiment that would allow you to determine the optimum time for clarification (i.e. the shortest time for the lowest turbidity) using a polyelectrolyte.
- 7) In an actual water treatment plant, what is typically done with the "sludge" that settles to the bottom of the tank after using a flocculating agent?
- 8) What changes would you make in the given procedure for this lab that you feel would yield more reliable results?
- 9) What types of substances would cause turbidity in water taken from a lake or river?
- 10) Identify the name of a company that sells polyelectrolytes for water treatment purposes.
- 11) What is the maximum recommended turbidity level for drinking water?
- 12) Do high turbidity levels in a drinking water supply have any impact on the effectiveness of disinfection of the water using chlorine? Explain.

Table #1 Jar Test Results

Jar#	Polymer Dosage (mL)	Polymer Concentration C_2 (mg/L) *	pH Initial	pH Final	NTU Initial	NTU Final	Time to first flow (sec)	Turbidity Ranking 1 = lowest 6 = highest
1								
2								
3								
4								
5								
6								

* Sample Calculation: Consider 4.0 mL of polymer of concentration 200mg/L being added to 800 ml of clay in water suspension. Use $V_1 \times C_1 = V_2 \times C_2$

$$(4.0 \text{ mL})(200 \text{ mg/L}) = (804 \text{ mL})(C_2) \quad \Rightarrow \quad C_2 = 0.994 \text{ mg/L}$$

LAB 6: FILTRATION

Purpose

To investigate the operation of a cartridge filtration system and to compare the performance of a variety of cartridge filters in the filtration of a calcium carbonate slurry.

Theory

In filtration, suspended particles in a fluid (defined as a liquid or gas) are physically or mechanically removed by using a porous medium that retains the particles as a separate phase (or cake) and passes the clear filtrate. In commercial filtration, the range of applications is very wide. Filtration plays a key role in environmental technology in the removal of contaminants from water, wastewater and air streams. The suspended particles in these streams can be very fine (in the micron range) or much larger, very rigid particles, spherical or very irregular in shape, aggregates of particles or individual particles. For most environmental applications, the solid residue or cake that is separated from the water or air stream is often the contaminant and is of no value. It is then discarded after further dewatering, drying or treatment. The filtrate generally tends to be the valuable product (such as purified water or air that can now be returned to the environment or manufacturing process). However, there are many environmental applications where both the filter cake and the filtrate are valuable and can be reused or sold. This is considered a pollution prevention strategy that can save money for a company. The selection of the filtering process and equipment depends on how much of the suspended particles must be removed from the fluid.

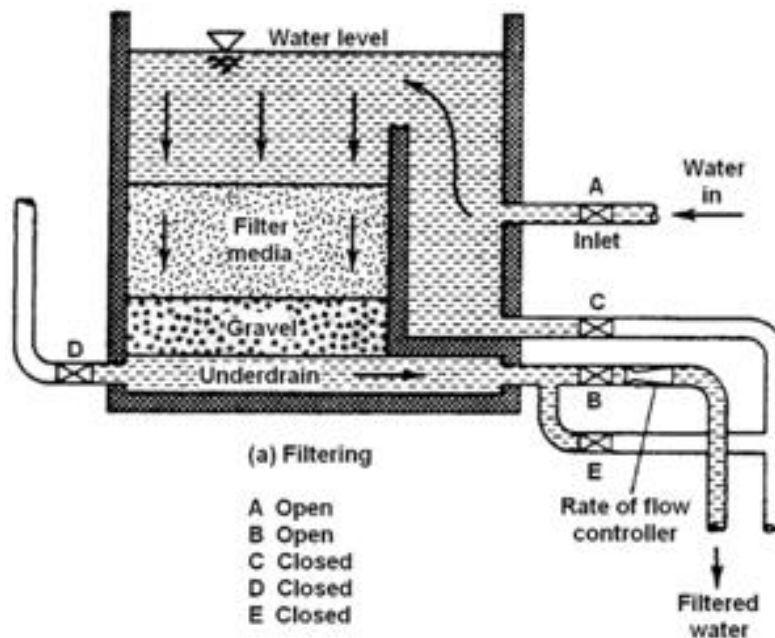
When the concentration of the feed “slurry” is very low, a filter can operate for very long periods of time before the filter needs cleaning. Industrial filtration equipment differs from laboratory filtration equipment only in the amount of material handled and in the necessity for low-cost operation.

Types of Filtration Equipment

A: Bed Filters

This is the simplest type of filter and is used where relatively small amounts of solids are to be removed from large amounts of water in clarifying the liquid (as in a municipal water treatment plant). Bed filters consist of a porous plate that supports a layer of coarse gravel. On top of the gravel is a layer of fine sand which acts as the filter medium. See Figure 1. The water stream is introduced over the top of the sand and the clarified water is removed at the bottom. The filtration continues until the precipitate of fine particles has clogged the sand so that the flow rate stops. The bed is then backwashed to remove the trapped particles.

Figure 6 - 1: Bed Filter



B: Plate and Frame Filter Presses

A plate and frame filter press consists of plates and frames assembled alternatively with a filter cloth over each side of the frames. The plates have channels cut in them so that clear filtrate liquid can drain down along each plate. The feed slurry is pumped into the press by a diaphragm pump or gear pump and flows through the duct into each of the open frames so that slurry fills the frames. The filtrate passes through the filter cloths and the solid builds up inside the frames until they are full. At this point, the process is stopped and the solid filter cake is removed for disposal. The apparatus is reassembled and the process is started again. This is a batch process. Plate and frame filters are used extensively in industries such as breweries, mining, plating, food, varnish processing, chemicals, cement, waste and sewage treatment, ceramics, etc. to dewater sludges from settling tanks.

C: Leaf (or Cartridge) Filters

These filters consist of a hollow framework covered by a sack of filter cloth or pleated paper or plastic or wound string. These are placed in a sealed filter chamber either individually or in parallel. The slurry to be filtered is forced under pressure by a pump through the filter medium. The filter cake is retained on the outside surface of the filter medium. Depending on the particle size to be filtered, the filter medium can be selected with an appropriate pore size opening. At the end of the filtering operation, the filter medium can be discarded along with the cake; however it is more common to dislodge the cake from the medium with an air or water jet. This system is also a batch process. Such systems are used extensively in the following industries to treat water and wastewaters: electroplating, electronics, food, pharmaceutical, photographic, chemicals, printing, petrochemical, beverage etc. Several suppliers of these systems are; Serfilco-Canada in Oakville, Filter Innovations in Toronto, U. S. Filter in the U.S.A. See Figure 3 for a schematic of the cartridge filter system that will be used in this lab experiment.

Figure 6 - 2: Plate and Frame Filter Press

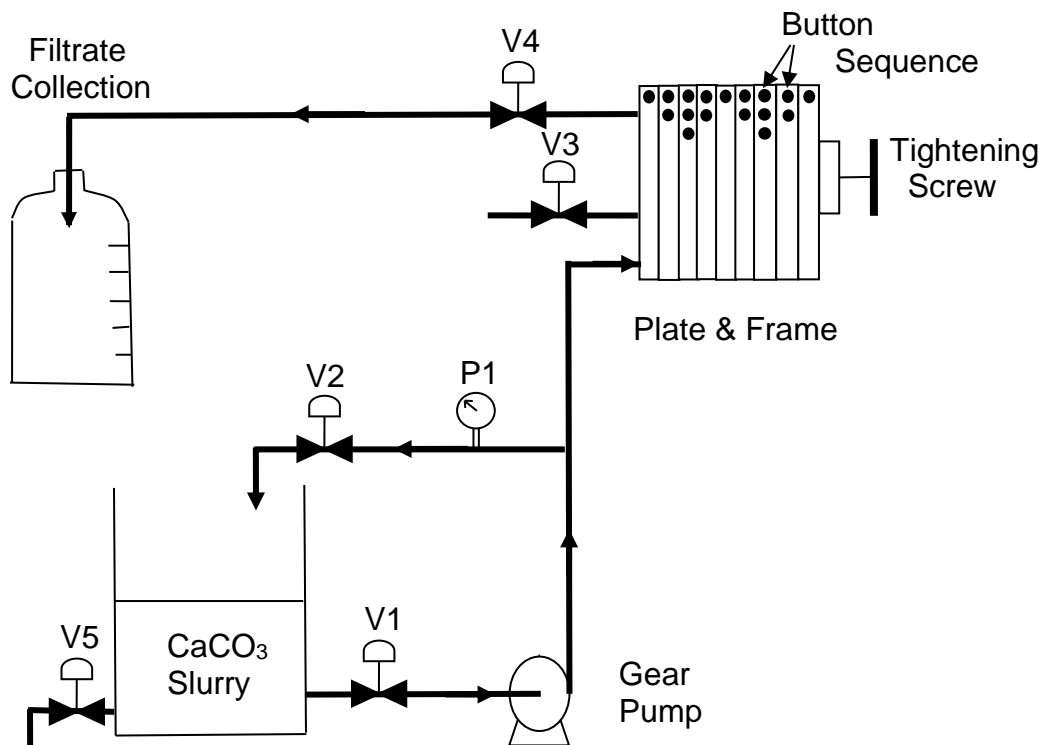
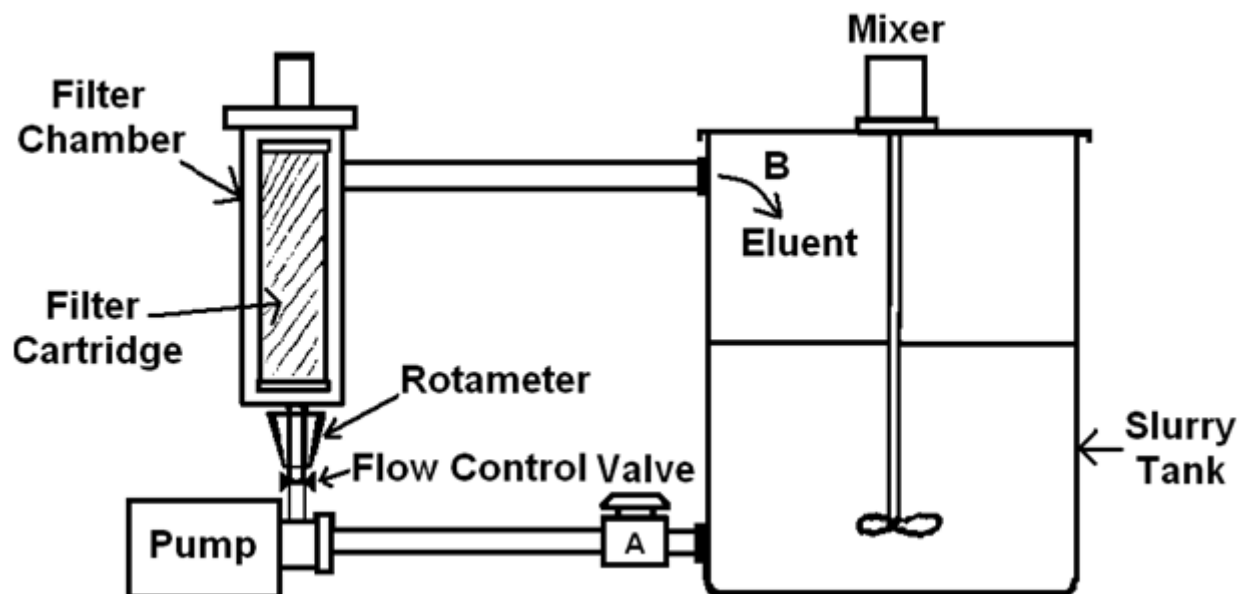


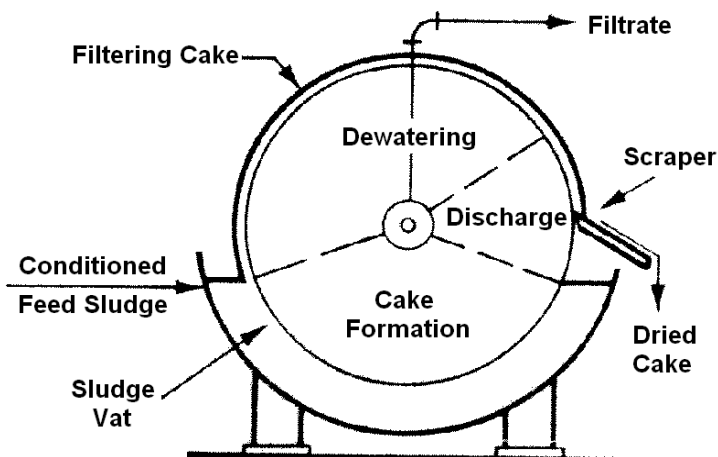
Figure 6 - 3: Cartridge Filter System



D: Continuous Rotary Drum Vacuum Filters

This process filters the slurry and washes and discharges the cake continuously. The drum is covered with a suitable filtering medium. The drum slowly rotates through the slurry tank and a vacuum from inside the drum draws the filtrate through the filter medium. The solids are retained on the surface of the drum where it is washed, dried and scrapped off with a knife. These systems are expensive but involve less manpower to operate.

Figure 6 - 4: Rotary Vacuum Filter (Drum Type)



Pressure Drop and Filter Aids

Filtration processes of the type described above will result in a buildup of pressure across the filter medium as the cake thickness increases. Thus, monitoring of pressure drop is often a normal part of the operation. Filtration processes can be run at either constant pressure or at constant flow rate. Filtering materials consist of heavy cloth, glass cloth, felted pads, heavy paper products, metal cloth, and nylon cloth. Filter aids such as diatomaceous earth are sometimes added to the feed slurry. These aids tend to increase the porosity of the filter cloth and reduce the resistance of the cake during filtration. Filter aids are limited to cases where the filter cake will be discarded at the end of the run.

Equipment

1. A cartridge filtration system consisting of
 - slurry tank
 - magnetic drive centrifugal pump
 - rotameter
 - filter chamber
 - valves
 - a variety of filter cartridges
2. A HACH 2100N Turbidimeter with accessories

Method

Part A: Performance of Cartridge Filters

1. Fill the slurry tank to the 30 L mark with water. See Figure 3.
2. Weigh about 5 grams of calcium carbonate on the balance provided, record the exact amount.
3. Add the calcium carbonate to the slurry tank.
4. Start the air driven mixer by opening the ball valve on the steel air line and by turning the valve (black plastic) on the air line pressure reducer.
5. Mix the slurry vigorously for several minutes to ensure the calcium carbonate is well suspended in the water in the tank. Leave the mixer running at slow speed throughout the experiment.
6. You will now test several filter cartridges to assess their performance in filtering the calcium carbonate slurry. Select the 10 micron paper pleated cartridge.
7. Unscrew the top of the cartridge chamber on the apparatus and place the cartridge into the cartridge chamber. Replace the top of the cartridge chamber (do not over tighten).
8. Using the tank sampling test tube provided, take a sample from the slurry tank. See Part B for turbidity analysis.
9. Open all the valves on the filtering apparatus.
10. Plug in the pump and adjust the valve immediately below the rotameter to obtain a flow setting of 12 LPM (Liters Per Minute) and begin timing. Read float at its largest diameter.
11. **Every five minutes** over a 20 minute period:
 - a. Take a slurry tank sample and measure the turbidity. See Part B for turbidity analysis.
 - b. Take a sample of the “eluent” and measure the turbidity. The “eluent” is the filtrate that is returned to the slurry tank at point B in Figure 3.
12. At the end of the 20 minute filtering run, unplug the pump, stop the mixer and remove the 100 micron string wound filter cartridge from the chamber.
13. Wash the cartridge thoroughly in the large sink using the water hose. Allow the cartridge to drain in the clear plastic bucket provided.
14. Use the cart pump to pump the contents of the slurry tank to the floor drain.
15. Repeat steps 2 to 15 above, but use a 20 micron paper pleated cartridge.
16. Repeat steps 2 to 15 above, but use a 100 micron string wound cartridge.
17. Clean the work area and return all equipment to its original location.

Part B: Turbidity Analysis

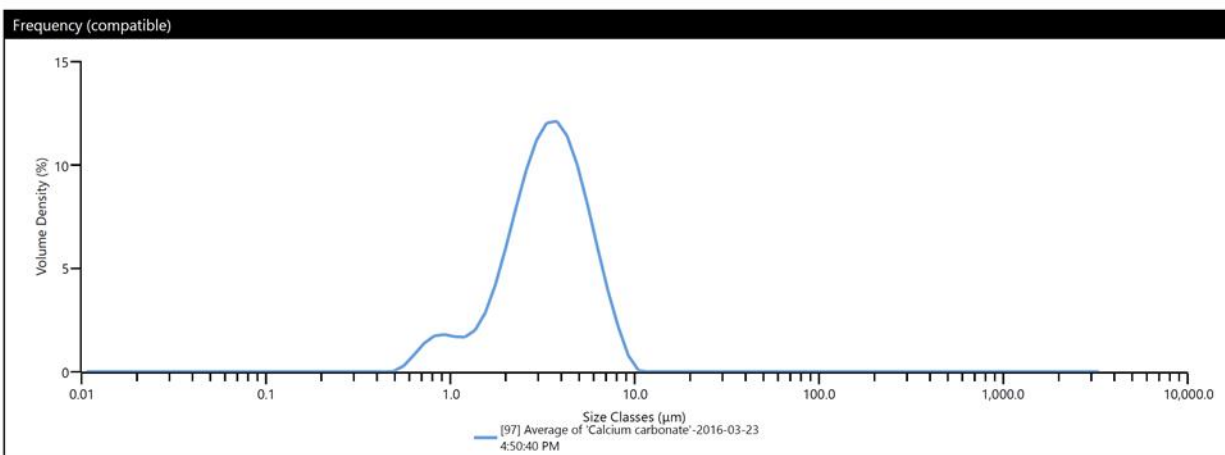
1. The turbidity meter does not need to be calibrated. You may check the performance of the meter using the standards provided (shake bottles prior to measurement; do not open the bottles).
2. Fill a Turbidimeter sample cells (small glass vial with screw cap) to the top white mark with the slurry sample.
3. Cap the sample cell and gently wipe it dry with a Kimwipe tissue. Do not handle the sample cell near the center or your fingerprints will interfere with the turbidity readings.
4. Place the sample cell into the Turbidimeter sample compartment (be sure the white arrow on the sample cell faces you).
5. Close the lid of the sample compartment.
6. Press: I/O to turn the meter on.
7. Make sure the measurement range is set to "auto" (if you get a measurement value of 9.99, this means you are out of range).
8. Press: READ. It will take about 12 seconds for the reading to show. Record the turbidity of the sample in units of NTU (Nephelometric Turbidity Units).
9. Remove the sample cell from the Turbidimeter and discard the sample (back to the slurry tank).

Note: Use the same sample cell for all your turbidity measurements since this will eliminate the effect of any scratches on the sample cell.

Questions

1. The decrease in turbidity of the slurry and the eluent during the course of the run is related to the removal of calcium carbonate solids from the water. On a single graph plot the turbidity vs. time for the slurry and eluent of all three cartridges (six data sets or lines).
2. Calculate the percent change in turbidity (which is related to but not necessarily proportional to the percent removal of the calcium carbonate from the water).
3. Compare the three slurry plots in terms of effectiveness of removal of calcium carbonate.
4. Compare the three eluent plots in a similar way.
5. What would be your recommendations in terms of filter selection for this slurry?
6. In water treatment operations, turbidity of the water supply is an important consideration. What causes turbidity in natural water sources (lakes, rivers, and groundwater)?
7. Other than filtration, what are some methods of removing turbidity from water and wastewater?
8. The following datasheet presents particle size characterization of the calcium carbonate used in this experiment, determined by laser diffraction analysis. Comment on how the data relates to the filtration performance you observed.

Measurement Details	Measurement Details
Operator Name nbatista Sample Name Average of 'Calcium carbonate' SOP File Name HydroMV.cfg	Analysis Date Time 2016-03-23 4:50:40 PM Measurement Date Time 2016-03-23 4:50:40 PM Result Source Averaged
Analysis	Result
Particle Name CaCO ₃ (calcite) Particle Refractive Index 1.572 Particle Absorption Index 0.100 Dispersant Name Water Dispersant Refractive Index 1.330 Scattering Model Mie Analysis Model General Purpose Weighted Residual 1.65 % Laser Obscuration 7.66 %	Concentration 0.0025 % Span 1.347 Uniformity 0.408 Specific Surface Area 2269 m ² /kg D [3,2] 2.64 µm D [4,3] 3.59 µm Dv (10) 1.50 µm Dv (50) 3.35 µm Dv (90) 6.01 µm



Result															
Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In	Size (µm)	% Volume In
0.0100	0.00	0.0463	0.00	0.214	0.00	0.991	1.41	4.58	8.38	21.2	0.00	98.1	0.00	454	0.00
0.0114	0.00	0.0526	0.00	0.243	0.00	1.13	1.38	5.21	6.77	24.1	0.00	111	0.00	516	0.00
0.0129	0.00	0.0597	0.00	0.276	0.00	1.28	1.64	5.92	4.97	27.4	0.00	127	0.00	586	0.00
0.0147	0.00	0.0679	0.00	0.314	0.00	1.45	2.34	6.72	3.25	31.1	0.00	144	0.00	666	0.00
0.0167	0.00	0.0771	0.00	0.357	0.00	1.65	3.49	7.64	1.81	35.3	0.00	163	0.00	756	0.00
0.0189	0.00	0.0876	0.00	0.405	0.00	1.88	4.98	8.68	0.61	40.1	0.00	186	0.00	859	0.00
0.0215	0.00	0.0995	0.00	0.460	0.00	2.13	6.61	9.86	0.00	45.6	0.00	211	0.00	976	0.00
0.0244	0.00	0.113	0.00	0.523	0.21	2.42	8.15	11.2	0.00	51.8	0.00	240	0.00	1110	0.00
0.0278	0.00	0.128	0.00	0.594	0.67	2.75	9.37	12.7	0.00	58.9	0.00	272	0.00	1260	0.00
0.0315	0.00	0.146	0.00	0.675	1.16	3.12	10.08	14.5	0.00	66.9	0.00	310	0.00	1430	0.00
0.0358	0.00	0.166	0.00	0.767	1.47	3.55	10.16	16.4	0.00	76.0	0.00	352	0.00	1630	0.00
0.0407	0.00	0.188	0.00	0.872	1.52	4.03	9.57	18.7	0.00	86.4	0.00	400	0.00	1850	0.00