كلية الهندسة
College of Engineering
QATAR UNIVERSITY جامعة قطر

Lab. Manual of Environmental Engineering

The Department of Civil and Architectural Engineering

ENVIRONMENTAL ENGINEERING LABORATORY SAFETY GUIDELINES AND AGREEMENT

GENERAL SAFETY

- You may only enter the laboratory when you are authorized to do so. Conduct yourself in a responsible manner at all times in the laboratory.
- No students are allowed to work in the lab without signing the lab safety agreement.
- Follow all written and verbal instructions carefully. If you do not understand a direction or part of a procedure, ask your supervisor before proceeding with the activity.
- You have the primary responsibility for your safety. Don't do anything if you feel unsafe.
- Don't do work alone in the lab. Only approved personnel are allowed to do work alone in the lab with the consent of the Lab Supervisor.
- Don't play or eat anything in the lab. Always keep the lab clean for the working area. Place trash in appropriate receptacle.
- Never fool around in the laboratory. Horseplay, practical jokes, and pranks are dangerous and prohibited.
- Don't leave food in the lab, dispose of in trash receptacles or dumpster.
- Level the reagents name and date after preparing it.
- Always work in a well-ventilated area.

PERSONAL PRECAUTIONS

- Always wear shoes that completely cover your feet – no sandals or opened-toed shoes.
- Avoid bulky, loose or trailing clothes.
- Tie back long hair.
- Place all belongings out of the work area.
- Know the locations and operating procedures of all safety equipment including: first aid kit(s), and fire extinguisher. Know where the fire alarm and the exits are located.
- While working in the environmental/chemistry lab, you should wear a protective apron and gloves to protect your clothes and skin. You should also wear safety goggles or a face shield when performing any test where there is potential danger to the eyes.

- Do not operate a hot plate by yourself. Take care that hair, clothing, and hands are a safe distance from the hot plate at all times. Use of hot plate is only allowed in the presence of the teacher.
- Heated glassware remain very hot for a long time. They should be set aside in a designated place to cool, and picked up with caution. Use tongs or heat protective gloves if necessary.
- Do not place hot apparatus directly on the laboratory desk. Always use an insulated pad. Allow plenty of time for hot apparatus to cool before touching it.
- Dry wet hands and clothing before working with electricity. Mop up all water spilled on the floor.
- Be as careful for the safety of others as for yourself. Think before you act.

CHEMICAL HAZARDS

- Know the hazards of chemicals you are using.
- Never eat, drink, or smoke in a lab. Never taste or smell chemicals.
- Never use an open flame when flammable chemicals are present.
- Wash your hands with soap before leaving the lab.

EQUIPMENT SAFETY

- Inspect all equipment prior to use.
- Don't operate equipment if you are not familiar with. Always receive proper training for the equipment prior to use.
- Before equipment is operated, the guards for all accessible moving mechanical components must be in place.
- Report immediately to the approved supervisor or lab personnel for any faulty equipment. Don't use it until it is inspected or repaired. Don't use equipment that has been tagged out or marked faulty.
- De-energize machinery or equipment before cleaning or adjusting.

WHAT SHOULD I DO IN AN EMERGENCY?

- Call the lab personnel/supervisor to take necessary action.

- For a minor cut, thoroughly rinse the cut. Use first aid kits to bandage small injuries such as scratches and minor burns.
- If someone is being shocked, DO NOT TOUCH THEM. Use an insulating object to pull the person free, or turn the power off.
- If a spill occurs, restrict access to the spill area.
- If chemicals come in contact with the skin or eyes, the affected body parts should be rinsed thoroughly with large amounts of clean water. Eye wash stations and emergency showers are used to deal with chemical spills.
- If a fire occurs and is small enough to fight, use the fire extinguisher in the lab.
- For any emergency call by the relevant number:

Emergency Number

for

Qatar University

Fire/Burning Emergency:	3999
Health Emergency:	5050 / 3294
Control Room (Electricity):	3600
Business Operations Department (BOD):	3500
Police Station:	999

AGREEMENT

Date: _____

I acknowledge that I have received the Environmental Engineering Laboratory Safety Guidelines. By signing below, I agree to adhere by the guidelines expressed above. In the event that I do not follow the stated guidelines, I understand that I may be subject to disciplinary action at the discretion of the immediate supervisor.

Name: _____

Phone Number: _____

Signature: _____

Emergency Contact Information:

Contact Name:_____

Relationship: _____

Contact Phone Number: _____

Approved by:_____

Signature:_____

List of experiments

Lab. 1: Introduction to the environmental chemistry lab

Lab. 2: Alkalinity test of wastewater sample

Lab. 3: Hardness test of wastewater sample

Lab. 4: Chemical Oxygen Demand (COD) of wastewater sample

Lab. 5: Biochemical Oxygen Demand (BOD) of wastewater sample

Lab. 6: Jar Test: Coagulation and flocculation processes of wastewater sample

Lab. 7: Solid Determination: Gravimetric methods

Lab. 8: Adsorption of methylene blue on activated carbon.

Experiment # 1: Introduction of Environmental Chemistry.

Please briefly discuss the following points:

1. a. Define concentration, titration, molarity, parts per million (ppm), and parts per billion (ppb).

   b. In the lab, how did you prepare 0.1M NaOHsolution?

   c. What was the pH of 0.1M NaOH? Why so?

   d. How did you titrate it with H_2SO_4 solution to know its concentration?

2. What will be the molarity if 15 g of NaOH is present in 225 ml solution?
3. How much Na_2SO_4 is present in 300 ml of 0.1 M Na_2SO_4 solution?
4. Describe the relationship between hydrogen ion concentration $[H^+]$ and pH.

Experiment # 2: Alkalinity

Introduction:

Alkalinity can be defined as the ability of a water to neutralize acid or to absorb hydrogen ions. It is the sum of all acid neutralizing bases in the water. The bacteria and other biological entities which play an active role in wastewater treatment are most effective at a neutral to slightly alkaline pH of 7 to 8. In order to maintain these optimal pH conditions for biological activity there must be sufficient alkalinity present in the wastewater to neutralize acids generated by the active biomass during waste treatment. This ability to maintain the proper pH in the wastewater as it undergoes treatment is the reason why alkalinity is so important to the wastewater industry. Another example is that of the softening reactions using lime. If there is no sufficient bicarbonate alkalinity, then carbonate ions must be added to the water so that calcium will precipitate out of the water in the form of calcium carbonate.

The main species that contribute to alkalinity are bicarbonate, carbonate and hydroxyl. However, since most natural waters have a pH value between 6 and 8, it is usually assumed that alkalinity is equal to the bicarbonate concentration.

Objective:

To measure the concentration of the various species that contribute to alkalinity in different types of water.

Materials:

Burette (25 ml), Porcelain dish, Magnetic stirrer and rod, Beaker (150 ml), Pipette, Measuring cylinder (100 ml), pH meter, 0.02N Sulphuric acid, Methyl Orange indicator, Phenolphthalein indicator.

Experiment # 2: Alkalinity

Experimental procedure:

For different water samples, the following procedures should be carried out to determine the total alkalinity and the contributing species.

Indicator Method:

1. Pipette exactly 50 ml of sample into a glass beaker or porcelain dish and drop in a magnetic rod.
2. Mount a 50 ml burette and fill it to the mark with 0.02N sulphuric acid solution.
3. Add 5 drops of Phenolphthalein indicator to the sample. If the solution turns pink, add acid slowly till pink color disappears. Record the volume of acid in milliliters as P.
4. Add 5 drops of Methyl Orange indicator to the same sample at the end of the first titration and add 0.02N sulphuric acid slowly till orange color turns to pinkish yellow. Record this volume as M. Then, T = P+M.

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Determination of alkalinity species:

Determine the various species of alkalinity present in the samples using the relationships shown below.

Condition	OH^-	$CO_3^=$	HCO_3^-
1. P = T	T	0	0
2. P = 1/2T	0	2P	0
3. P > 1/2T	(2P-T)	2(T-P)	0
4. P < 1/2T	0	2P	(T-2P)
5. P = 0	0	0	T

Experiment # 2: Alkalinity

Record the titration data in the following table:

Sample	P(ml)	T(ml)	P & T Condition
Sample A			
Sample B			
Sample C			

Using the above data, calculate the concentrations of the various species of alkalinity using the formula given below for each sample and list in the following table.

Alkalinity, mg/l as $CaCO_3$ = A x N x 50,000/ml sample

A = ml, sulphuric acid solution used

N = normality of acid solution.

Sample	OH^-		CO_3^{--}		HCO_3^-	
	ml	mg/l as $CaCO_3$	ml	mg/l as $CaCO_3$	ml	mg/l as $CaCO_3$
Sample A						
Sample B						
Sample C						

Report:

In addition to tables showing all experimental results, consider the following points while preparing your report:

a. Compare the concentration of the various species contributing to alkalinity for the different types of water.

b. How does alkalinity play vital role in wastewater treatment processes?

Experiment # 3: Hardness

Introduction:

Hardness in water is caused mainly by the ions of calcium and magnesium. Such ions exist as a result of the interaction between recharge water and certain geological formations (i.e. limestone) that contain these ions. Public acceptance of hardness varies from community to community, consumer sensitivity being related to the degree to which the person is accustomed.

Hardness of more than 300-500 mg/l as $CaCO_3$ is considered excessive and results in high soap consumption as well as objectionable scale in heating vessels and pipes.

Ethylenediaminetetraacetic acid and its sodium salts (abbreviated EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of dye such as Eriochrome Black T is added to an aqueous solution containing calcium and magnesium ions, the solution becomes wine red. If EDTA is added as a titrant, the calcium and magnesium will be complexed, and when all of the magnesium and calcium has been complexed the solution turns from wine red to blue, marking the end point of the titration. Analysis for hardness is performed in two stages by estimating total and calcium hardness separately calculating the magnesium hardness from the difference between the two.

Objective:

To determine the total hardness as well as calcium and magnesium of raw water and treated water samples using EDTA titrimetric method.

Materials:

Burette (50 ml), porcelain dish, magnetic stirrer and rod, pipette, measuring cylinder (100 ml), ammonia buffer solution, sodium hydroxide solution, Eriochrome black T indicator, Murexide (ammonium purpurite), EDTA, raw water sample, treated water sample

Experimental procedure:

For different water samples, the following procedure should be carried out to determine the total, calcium and magnesium hardness.

1. Pipette exactly 25 ml of raw water sample into a porcelain dish and drop in a magnetic rod.
2. Mount a 50 ml burette and fill it to the mark with 0.01M EDTA solution.
3. Add 1-2 ml of ammonia buffer and 1-2 ml of Eriochrome Black T indicator.
4. Start adding slowly 0.01M EDTA solution till the color of the solution changes from wine red to blue. Record the volume of EDTA solution and calculate total hardness using the following formula:

$$Hardness\ \left(as\ \frac{mg}{l}CaCO3\right) = \frac{A * B * 1000}{ml\ Sample}$$

Where:

A= ml EDTA used

B = mg $CaCO_3$ equivalent to 1 ml EDTA titrant

=100 (since molecular weight of $CaCO_3$ is 100) * Molarity of EDTA (0.01)=1.0

5. Add 1-2 ml sodium hydroxide buffer and 0.2 g murexide indicator into 25 ml of raw water sample.
6. Start adding 0.01M EDTA solution slowly till the color of the solution changes from purple to violet. Record the volume of EDTA used and calculate calcium hardness using the previous formula.
7. Calculate magnesium hardness (= total hardness - calcium hardness)
8. Repeat titration for the other water samples and calculate the hardness.

Experiment # 3: Hardness

Report:

In addition to tables showing all experimental results, consider the following points while preparing your report:

a. Compare the hardness obtained for the various types of water.

b. Would you expect groundwater to be softer or harder than surface water? Why?

Sample	Buffer	Indicator	Initial Color	Final Color	Vol. of EDTA	Hardness mg/l as $CaCO_3$
A						TH=
						CaH=
	MgH = TH - CaH					MgH=
B						TH=
						CaH=
	MgH = TH - Ca H					MgH=
C						TH=
						CaH=
	MgH = TH - Ca H					MgH=

Experiment # 4: Chemical Oxygen Demand (COD)

Introduction:

Chemical oxygen demand (COD) is used to estimate the organic strength of wastes. However in this test, the organics are oxidized chemically not using microorganisms. As a result of this the COD test needs much less time (say 2 or 3 hours) to be conducted unlike the five days for the standard BOD test. Also since all organics are oxidized chemically, COD values will be higher than BOD values especially if biologically resistant organic matter is present in the waste. It is also possible, for much waste, to generate a correlation between COD, the quick and easy test, and BOD, the time consuming test.

Like the BOD test, oxygen is used to oxidize the organics to carbon dioxide and water. However, instead of free dissolved oxygen, chemically bound oxygen in potassium dichromate $K_2Cr_2O_7$ is used to oxidize the organics. As the potassium dichromate is used up the Cr^{+3} ion is produced. The amount of dichromate used is proportional to the amount of organics present. Likewise, the amount of Cr^{+3} ion present is proportional to the amount of organics digested.

$$\text{Organics} + \underset{\text{(Orange)}}{K_2Cr_2O_7} \rightarrow \underset{\text{(Green)}}{Cr^{+3}}$$

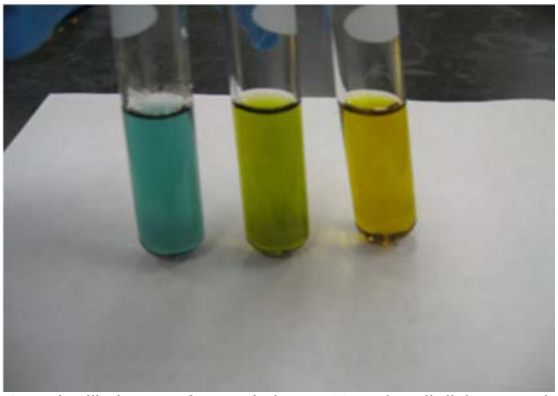


Figure: Sample dilutions. Left sample is >1500 mg/L. All dichromate is gone.

Objective:

To determine the chemical oxygen demand (COD) of a sample using the closed reflux

Materials:

Digestion vessels, block heater at 150 ± 2^0C, burette (25 ml), 250 ml flasks, measuring cylinders, standard potassium dichromate digestion solution, sulphuric acid reagent, photometer.

Experimental Procedure:

1) Place 2.5 ml sample in tubes and add 1.5 ml digestion solution.
2) Add 3.5 ml sulfuric acid reagent down inside of vessel so an acid layer is formed under the sample-digestion solution layer.
3) Chloride is the primary interference. Vials with mercuric sulfate can be used to eliminate chloride interference up to 2000 mg/L Cl-.
4) Tightly cap the tubes invert and shake well.
5) Place tubes in block digester preheated to 150 ^{0}C and reflux for 2 hours.
6) Cool to room temperature and place tubes in test tube rack.

The COD values can be determined either by **Titrimetric** or by **Photometric methods.** In this experiment, we will measure COD value by using photometric methods.

Theory of photometric method:

The dichromate ion does not absorb at the Cr^{3+} ion wavelength, and the Cr^{3+} ion absorbs only a small, correctable amount of the wavelength in the dichromate range. This slight interference is zeroed out in the calibration step. The dichromate ion is visible at 420nm, and the Cr^{3+} ion around 600 – 620nm.

Low range COD (<150 ppm) analysis measures the decrease in the oxidant, Cr_2O_7

High range COD (<1,500 ppm) analysis measures the increase in Cr^{3+}. A spectrophotometer sends the correct wavelength through the sample cell to a detector that measures transmittance.

COD has two common error sources. First, the oxidation step does not distinguish between organic and inorganic carbons. Where carbons are available, oxidation will create Cr^{3+} ions. It's the organic carbon fraction of the sample that's sought after, and it's the organics that found the correlation basis for Biochemical Oxygen Demand (BOD) and Total Organic Carbon (TOC). Unfortunately unknown oxidizable inorganics introduce positive error, and skew any attempt to directly substitute COD results for BOD or TOC. The more common interferent however, is chloride (Cl). The origin of chloride is chlorine (Cl_2). Chlorine is a great oxidizer of organics to include micro organisms. This is why chlorine is a powerful disinfectant in water treatment. Cl_2 works well because the two Cl atoms share an electron, and readily receive two electrons to become two stable chloride ions (Cl^{-2}). This electron grabbing oxidizes (or destroys) micro organisms, but adds many interferents to the COD sample. The reaction between chloride and silver sulfate creates silver chloride (AgCl). The reduction of silver sulfate correspondingly reduces the activity needed to oxidize straight chain hydrocarbons, a negative interferent one would think. But the cloudiness of silver chloride precipitate causes a false positive absorbance value. Furthermore, the rigorous COD digestion can actually result in the reaction of dichromate with chloride to form chromic acid (and the elemental form of chlorine). Adding chlorine to the sample causes a positive interference. Suffice it to say that all chloride interferences are overcome by complexing chloride with mercuric sulfate ($HgSO_4$). Chloride concentrations >2,000 mg/L cannot be corrected. High chloride samples must be run titrimetrically by EPA 410.3, COD for Saline Waters.

- **By Photometric Method:**

1. Place the blank vial into the holder (HI 83214 Model) and push it completely down.
2. Press ZERO and 'SIP' will blink on the display. Wait for a few seconds and the display will show "-0.0-". Now the meter is zeroed and ready for measurement.
3. Remove the blank vial. Place the sample vial into the holder and push it completely down.
4. Press READ DIRECT and 'SIP' will blink during measurement.
5. This will directly display concentration in mg/l of chemical oxygen demand.

Report:

In addition to the standard report, please consider the following points in your report:

a) Explain the advantages and disadvantages of the COD versus BOD tests.

b) Based on today's experiment, could you estimate the BOD for the sample used today?

c) Would you expect larger difference between COD and BOD for domestic or industrial wastewater? Explain.

Experiment # 5: Biochemical Oxygen Demand (BOD)

Introduction:

Estimating the organic content of a wastewater is an essential information needed for planning proper management and treatment of wastewater. The Biochemical oxygen demand (BOD) gives an estimate of the strength of industrial or domestic wastes in terms of the oxygen consumed by microorganisms to decompose the organic matter present in the waste. The higher the BOD, the more oxygen will be demanded from the waste to break down the organics. The BOD test is most commonly used to measure waste loading at treatment plants and in evaluating the efficiency of wastewater treatment. The BOD test is performed by incubating a sealed wastewater sample for the standard 5-day period, then determining the change in dissolved oxygen content. The bottle size, incubation temperature, and incubation period are all specified. All wastewaters contain more oxygen demanding materials than the amount of DO available in air-saturated water. Therefore, it is necessary to dilute the sample before incubation to bring the oxygen demand and supply into appropriate balance. Because bacterial growth requires nutrients such as nitrogen, phosphorous, and trace metals, these are added to the dilution water, which is buffered to ensure that the pH of the incubated sample remains in a range suitable for bacterial growth. Complete stabilization of a sample may require a period of incubation too long for practical purposes; therefore, 5-day period has been accepted as the standard incubation period.

Objective:

The objective of the experiment is to determine the biochemical oxygen demand of a wastewater sample. BOD will be measured in this experiment by the pressure difference within a closed system (respirometric BOD).

Materials:

Respirometric BOD cell (Oxidirect), potassium hydroxide (KOH), nitrification inhibitor B (= Allyl Thiourea, or ATH) and wastewater sample.

Biochemical Oxygen Demand (BOD)

The biochemical oxygen demand (BOD) of waste water, industrial effluents and surface water is an expression for the amount of oxygen consumed by the decomposition of organic matter in a biochemical process.

Principle of Measurement

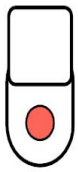
The BOD system, consisting of the sample bottle and the BOD sensor, represents a closed system. In the bottle, above the sample itself, is a defined volume of air. During the BOD measurement, the bacteria in the sample consume the dissolved oxygen in the sample. This is replaced by oxygen in the bottle above the sample. The carbon dioxide released at the same time react with the potassium hydroxide in the seal gasket. This generates a decrease in pressure within the system. This is measured by the BOD sensor and displayed as a BOD value in mg/l O2.

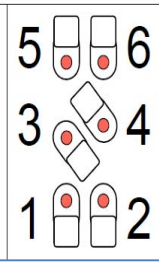
Preparing the Sample / Brief Summary

• Estimate the measurement range and select the volume for the sample
• Carry out necessary pre-treatment of the sample (e.g., setting the pH value; filtering; etc.)
• Measure the volume of the sample precisely, using the overflow measurement flask and transfer it to the BOD bottle, with the aid of a funnel. If necessary, add nitrification inhibitor
• Insert the magnetic stirring rod.
• Place 3-4 drops of KOH solution into the seal gasket. Then insert the gasket in the neck of the bottle.
• Screw the BOD sensors to the sample bottles.
• Place the bottle in the bottle rack.
• Start the measurement
• Incubate the sample in accordance with the instructions (e.g. BOD5 for 5 days at 20 °C)

Operation:

Key and its operation are described below:

Key	Functions
On Off	• Switches the unit on and off (automatic switch off approx. 45 sec after last operation) • Quits sub-menus • Breaks off operations
START	• Set Up of measurements
READ	• Reading of a current value for a selected position (does not store value!)
+	• Increases parameter / figure
-	• Reduces parameter / figure
ENTER	• Confirms input • Switches to other sections in menus
	• Head key : selects the position required • *Head LED : indicates the position which has been selected*



BOD Measurement

Sample Volume

The sample volume is related to the expected BOD value. The OxiDirect® is designed to operate with the following ranges and sample volumes, allowing BOD measurement up to 0 - 4000 mg/l, without any dilution.

Range BOD mg/l	Sample volume in ml	Dosage ATH
0 – 40	428	10 drops
0 – 80	360	10 drops
0 – 200	244	5 drops
0 – 400	157	5 drops
0 – 800	94	3 drops
0 – 2000	56	3 drops
0 – 4000	21.7	1 drops

Note

The expected results should be in the upper half of the range.

For domestic waste it is generally appropriate to consider a BOD_5 value which is approximately 80 % of the COD value.

Preparing the Water Sample

1. Check the pH value of the effluent sample. The optimum pH value for biochemical oxidation is between pH 6.5 and 7.5. If the pH value of the sample is higher or lower, it should be pre-adjusted. Any significant deviation will result in a lower BOD value. If the pH value is too high, it can be reduced by adding dilute hydrochloric acid (1 mol/l) or dilute sulphuric acid (1 mol/l). If the pH value is low, it can be adjusted with a sodium hydroxide solution (1 mol/l).

2. Mix the water sample well and allow to settle for a short while. It may also be advisable to filter or homogenise the sample.

3. Measure the sample volume precisely, using the appropriate overflow measurement flask and pour the sample into the sample bottle (it may be helpful to use a funnel for this). Ensure that the sample in the bottle contains a representative portion of any solids in suspension. It is recommended that each sample should be tested twice or three times.

4. To inhibit nitrification, we recommend the addition of nitrification inhibitor B (= Allyl Thiourea, or ATH). This is particularly important for the low range 0 - 40 mg/l (for example, when checking discharges from effluent treatment plants). The right amount of nitrification inhibitor B is related to the measurement range -

5. Add a clean magnetic stirring rod to each sample bottle and add 3-4 drops of 45% potassium hydroxide solution to the seal gasket (this will absorb the carbon dioxide). Then insert the seal gasket in the neck of the bottle.

The sample must never come into contact with the potassium hydroxide solution. Never use grease or any other lubricants as an additional sealing agent, for the BOD sensors or for the seal gasket. Products of this kind may contain solvents which will attack the sensor, resulting in severe damage to the plastic housing and even to a failure of the sensor. The use of sealing greases and lubricants is not covered by our guarantee !

6. Before measurement begins, the prepared sample must be brought to the desired temperature (e.g. BOD5, 20 °C). This can be achieved by placing the

sample in a thermostatically controlled cabinet, while stirring the sample continuously with the inductive stirring system.

The OxiDirect® has an optional “Auto-Start” function, which enables it to start with samples at temperatures from 15° to 20°C. When this “Auto-Start” function is switched on, the system checks in specific intervals whether there has been a decrease of the pressure in the BOD bottle and will not start the timer until a pressure decrease is detected (latest, the timer will start 3 hours after the BOD sensor has been started).

7. Place the BOD sensors on the sample bottles and tighten carefully. This is extremely important – the system must be completely air-tight. Then place the BOD bottle, with the sensor screwed in position, into the bottle rack. This can be done in the thermostatically controlled cabinet itself.

Alternatively, because of the user-friendly design of the OxiDirect®, you can remove the entire BOD unit, with its integral bottle rack, from the thermostatically controlled cabinet, while leaving the inductive stirring system in the cabinet. There is no need to disconnect the cabling. Once the BOD bottles have been placed in the rack, the system is positioned over the inductive stirring system so that the 4 adjustment screws fit in the associated recesses of the stirring system.

8. Start the measurement process

9. Incubate the sample in accordance with the instruction (e.g. BOD5 for 5 days at 20 °C).

References:

1. Manual of OxiDirect.

Experiment # 6: Jar Test-Turbidity removal by coagulation and flocculation

Introduction:

Water as well as wastewater may contain some solids that remain in suspension even if left for a long time to settle by gravity. Such particles are called colloids, which are characterized by their light weight and the surface charge that will prevent them from agglomeration. One of the objectives of water treatment is to promote the settling of suspended matter. The coagulation process utilizes what is known as a chemical coagulant (aluminum or iron salts) to neutralize the surface charge and therefore promote particle agglomeration. Chemical coagulants are added to the raw water and for a brief period, rapid mixing is carried to produce what is called a microfloc. The next process is to subject the microfloc solution to controlled turbulence in order to bring the microflocs together to form a floc of adequate size that will settle under gravity. This process is called flocculation. Removal of turbidity by coagulation depends on the type of colloids in suspension, the temperature, pH, and chemical composition of the water, the type and dosage of coagulants, and the degree and time of mixing provided for chemical dispersion and floc formation.

Objectives:

1) To understand the process of coagulation and flocculation using alum and ferric chloride to remove turbidity of water.
2) To determine the optimum coagulant dose for a particular water.

Materials:

Jar test, aluminum sulphate solution, ferric chloride solution, beakers, turbidimeter, measuring cylinders, kaolin powder, sodium carbonate solution, sampling bottles.

Experimental Procedure:

1. Arrange two sets of Jar test apparatus. Check all units of the jar test apparatus before starting.
2. Prepare a turbid water sample by dissolving kaolin powder in distilled water
3. Determine the turbidity of the sample and record its value.
4. Prepare stock solution of alum and ferric chloride by dissolving 10 g powder in 1 liter distilled water.
5. Prepare sodium carbonate solution by dissolving 10 g salt in 1 liter distilled water.
6. Determine total alkalinity of the sample.
7. In the jar test units, fill each numbered beaker with sample.
8. If the measured alkalinity was low, add 6-8 ml sodium carbonate solution to each beaker.
9. Start the stirrers at 100 rpm and add quickly the doses of alum (given in table 1) in each beaker of set one and keep rapid mixing for exactly 1 min.
10. Reduce the speed of stirrers to 40 rpm and continue for 40 minutes.
11. Stop and raise the paddles above water level and leave the beakers for flocs to settle for 30 minutes.
12. Siphon out clear sample from each beaker without disturbing settled sludge.
13. Find out the turbidity of each sample.
14. Perform the same procedure with the ferric chloride (doses given in table 2) setup in parallel with alum setup.

Report:

In your report prepare a plot of the resulting turbidity values versus the coagulant dose then use these figure to estimate the optimum dose for both alum and ferric chloride. You should also compare and contrast between the two types of coagulants used when you discuss the results.

Table 1: Alum Data

Beaker #	1	2	3	4	5	6
Coagulant (mg/l)	0	10.0	20.0	30.0	50.0	100.0
Turbidity, NTU						

Table 2: Ferric Chloride Data

Beaker #	1	2	3	4	5	6
Coagulant (mg/l)	0	10.0	20.0	30.0	50.0	100.0
Turbidity, NTU						

Adapted from:

Al-Suwaiyan Mohammad S.(2003) "Laboratory Manual: Water Supply and Wastewater Engineering" Civil Engineering Department, King Fahd University of Petroleum & Minerals.

Experiment # 7: Solid Determination by Gravimetric Analysis

Introduction:

The concentrations of the various solids that exist in water and wastewater are important indicators of their quality. Solids present in water and wastewater can be broken into two categories, suspended and dissolved solids (non-filterable and filterable, respectively). Each of the aforementioned categories is also divided into organic (volatile) and inorganic (non-volatile) constituents. The processes that are used to separate the different solid categories are filtration and combustion.

Total Solids is the term applied to the material residue left in the vessel after evaporation of a sample and its subsequent drying in an oven at a defined temperature (103-105^{0}C). Total suspended solids refer to the non-filterable residue retained by a standard filter disk and dried at 103-105^{0}C. Total dissolved solids refer to the filterable residue that pass through a standard filter disk and remain after evaporation and drying to constant weight at 103-105^{0}C.

Objective:

To use the principles of gravimetric analysis to characterize the quality, in terms of solids concentrations, of three types of water, namely: tap water, drinking water, and secondary effluent.

Materials:

Porcelain dish (100 ml), steam bath, drying oven, muffle furnace, desiccator, filter paper, analytical balance, glass fiber filter disk, filtration apparatus, pipettes, measuring cylinders.

Experimental procedure:

a) Total Solids

1. Ignite a clean evaporating dish at 550^{0}C in a muffle furnace for 1 hr.
2. Cool the dish, weigh and keep it in a desiccator.
3. Transfer carefully 50 ml of sample into the dish and evaporate to dryness on a steam bath.
4. Place the evaporated sample in an oven adjusted at 103^{0}C and dry it for 1 hr.
5. Repeat drying at 103^{0}C till constant weight is obtained.
6. Determine the total solids with the following formula:

$$\text{Total solids (mg/l)} = \frac{(A-B) * 1000{,}000}{sample,ml}$$

where

A = weight of residue + dish (g)

B = weight of dish (g)

b) Total suspended solids:

1. Place a filter paper on the filtration apparatus.
6. Pour 50 ml of sample. Wash pipette with distilled water and pour the washing also into the crucible.
7. After filtration, dry the crucible at 103^{0}C for 1 hr
8. Weigh till constant weight is obtained.
9. Determine the total suspended solids with the following formula:

$$\text{total suspended solids (mg/l)} = \frac{(A-B) * 1000{,}000}{sample,ml}$$

where:

A = weight of residue and crucible (g)

B = weight of crucible (g)

c) Total Dissolved Solids:

Mg/l total dissolved solids = total solids – total suspended solids

Report:

In addition to tables showing all experimental results, consider the following points while preparing your report:

a. Compare the TS, TSS and TDS for the three samples.

b. Describe the results with the standard of wastewater treatment facilities.

Experiment # 8: Adsorption of Methylene Blue on Activated Carbon

Introduction:

Adsorption and adsorption processes are important fields of study in physical chemistry. Adsorption process using activated carbons are widely used to remove pollutants from wastewaters.

In this experiment, we will observe the adsorption of Methylene Blue (MB) on activated carbon

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Adsorption Isotherm:

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and solid phase, when the adsorption process reached an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models is an important steps to find the suitable model that can used for design purpose. Adsorption isotherm is basically important to describe how solutes interact adsorbents, and is critical in optimizing the use of adsorbents. Adsorption isotherm study is carried out on two well-known isotherms:

1. Langmuir Isotherm, and
2. Freundlich isotherm

Langmuir Isotherm: This can be defined as.

$$\frac{x}{m} = \frac{abC_e}{1 + bC_e}$$

Which can be rewritten as:

$$\frac{C_e}{(x/m)} = \frac{1}{ab} + \frac{1}{a}C_e$$

Where x/m= mass of adsorbate adsorbed per unit mass of adsorbent, mg adsorbate/g activated carbon.

a, b=empirical constants

Ce=equilibrium concentration of adsorbate in solution after adsorption, mg/l

Freundlich Isotherm: This can be defined as.

$\frac{x}{m} = K_f C_e^{1/n}$, can be written as:

$$log\frac{x}{m} = logK_f + \frac{1}{n}logC_e$$

Where x/m= mass of adsorbate adsorbed per unit mass of adsorbent, mg adsorbate/g activated carbon.

Kf=Freundlich capacity factor

Ce=equilibrium concentration of adsorbate in solution after adsorption, mg/l

1/n= Freundlich intensity parameter

Materials:

Methylene blue, activated carbon, spectrophotometer,

Experimental Procedure:

The adsorbent used in the experiment was a commercial activated carbon. A methylene blue (MB) solution with a concentration of 25 mg/l was prepared from analytical grade reagent and distilled water. The position of maximum absorbance (λmax) of this solution was determined to be 630 nm on spectrophotometer.

1. At first, calibration curve needs to be plotted by using Table 1. Measure the values of absorbance (A) with the spectrophotometer for corresponding concentration of Methylene Blue. Fit the straight line and get the slope (b) and intercept (a).
2. Prepare 25mg/l of MB solution of 100 ml in eight separate 250 mL flask
3. Place 0.00, 0.001, 0.005, 0.01, 0.0125, 0.025, 0.05, 0.10 and 0.25 gram of Activated Carbon (AC) in nine separate 250 mL flask.
4. Put rubber stopper in each flask, and shake vigorously on a mechanical shaker for 72 hrs, to reach equilibrium.
5. At the end, the supernatant need to be analyzed to get absorbance by spectrophotometer. Find the actual conc. from the calibration curve.
6. Fill the table-2 by the data found in the above steps.

Table 1: Calibration curve for fitting straight line (C=a + bA)

Nos.	Methylene Blue (C), mg/l	Absorbance (A)
1	1	
2	5	
3	10	
4	25	
5	50	

Table 2: Isotherm data

No.	C_0 (mg/l) of MB	m (g) of AC	Absorbance (A)	C_e (mg/l) of MB according to A	C_0-C_e (mg/l) of MB	$\frac{x}{m} = \frac{(C0-Ce)V}{m}$; mg/g	C_e/(x/m);
1	25	0.00					
2	25	0.001					
3	25	0.005					
4	25	0.01					
5	25	0.0125					
6	25	0.025					
7	25	0.05					
8	25	0.1					
9	25	0.25					

C_0= Initial concentration of MB, which is 25 mg/l in this experiment

C_e=equilibrium concentration of MB in solution after adsorption, mg/l

Calculation:

Fit the Langmuir and Freundlich isotherms from the above data. You can take help from page 1144 of Wastewater Engineering (Metcalf & Eddy) Fourth edition.

Conclusion:

Make comments from the above fitted isotherms