AALTO UNIVERSITY

Department of Built Environment

Water and Environmental Engineering

WAT-E2120

Physical & Chemical Treatment Processes of Water and Waste

Laboratory Work Instructions

Section 1 tasks A and B are carried out in large groups (half of the course's students), and task C can be done individually or within a group. Each student should submit a short report from Section 1.

Section 2 is carried out in groups of 4-8 persons. Each group will be responsible for the laboratory water treatment pilot during one week, and will have one additional treatment method to study during their pilot operations. The group's data and notes should be submitted to MyCourses at the end of the week of pilot work.

Section 3 is carried out in the same group as before. Each group member must contribute in writing a report that is returned via MyCourses within the Period III. Groups will also present their results and their process to the rest of the students in a session on 15th of February.

Contents

Section 1: Preparation Work: Water Quality and Coagulation

Task A: Initial Water Quality Analysis

General description

In this laboratory assignment, students will analyse the water quality of the raw water for the water treatment pilot plant. The objective is to understand what kind of water will be treated and gather necessary design parameters for treatment methods. Water quality will be determined by pH, turbidity, and organic matter (measured using UV absorbance UV_{254}).

Water analyses

- Determine UV_{254} absorbance of water according to Greenberg et al. (1995) at a fixed wavelength of 254 nm
- Determine turbidity of water according to standard SFS-EN 7027:2000
- Determine the pH of water according to standard SFS-EN ISO 10523:2012

Reporting

Collect all the results of your group, enter them into the excel file in the laboratory, and present the results in a short report. Select raw water quality values to be used for the process design. What other parameters could have been analysed from the raw water?

Task B: Coagulation and Settling

General description

In this laboratory assignment, students test removal of colloidal matter in water by using water treatment chemicals. The objective is to understand the quality of the water to be treated and the need of coagulation chemicals. The results should be recorded and the chemical dosages obtained will be used later in running the water treatment pilot plant.

Determination of the chemical dosage

Using empirical experimentation, determine the optimal chemical dosage needed to precipitate the colloidal matter in the raw water. Ferrous sulphate salt (PIX) is used as a coagulant and sodium hydroxide (NaOH) or sulphuric acid (H_2SO_4) for pH adjustment.

Procedure:

Choose preliminary dosages of PIX so that they are 0.4, 0.6, 0.7, 0.8, 1.0 and 1.2 times the permanganate concentration (i.e. $KMnO_4$ value that you are given) of the raw water (in mg/L).

Add the chosen amounts of PIX to 100 mL of raw water and measure the pH. Slowly add increments of acid or base until the pH is within the range of 4.5-5.2. Acid and base addition is performed with e.g. a burette or pipette. Document the initial pH of water after adding the PIX dosage and the pH after adding the acid or base. Also record the amount of acid/base added (and the molarity of the acid/base).

The coagulation test is carried out as follows:

1. Pour 1000 mL of raw water to the six decanters of the serial mixer,
2. Add the previously measured pre-alkali dosages for each PIX dose (remember to check pre-alkali concentration to calculate the amount required, notice that you are now adding pre-alkali to a 10x larger volume!) to the decanters.
3. Each mixer can be switched on independently. Add coagulant dosages one by one and switch mixers on one by one after each coagulant addition.
4. Stir for 20 minutes. Mixers stir fast in the beginning (for approx. 30 sec.) and then decrease (automatically) to approx. 30 rpm.

Observe and record the time needed for the formation of visible flocs. Near the end of stirring, compare the shapes, sizes and amounts of flocs in different decanters. Let the deposits settle for about 20 minutes. Observe and record the differences in settling speed, the amount of settled flocs and the nature of the unsettled flocs.

Filter water from the top of the decanters using a paper filter. Determine the new $KMnO_4$ values from the filtered water according to SFS 3036:1981. Also determine the UV_{254} absorbance (use 40 mm quartz cell), the iron content (standard SFS 3028:1976), and turbidity.

Reporting

Collect and present the results from the coagulation test, including visual observations on flocculation and values from the $KMnO_4$, UV_{254}, iron, and turbidity tests on post-flocculation samples. Data should also be entered to the laboratory excel file.

Choose the most applicable chemical dosage and present the criteria you used for your selection. Explain briefly how the chemicals used affect the quality of the treated water. Also explain briefly how the use of calcium hydroxide instead of sodium hydroxide would have influenced the results.

Task C: Operational parameters for the pilot plant

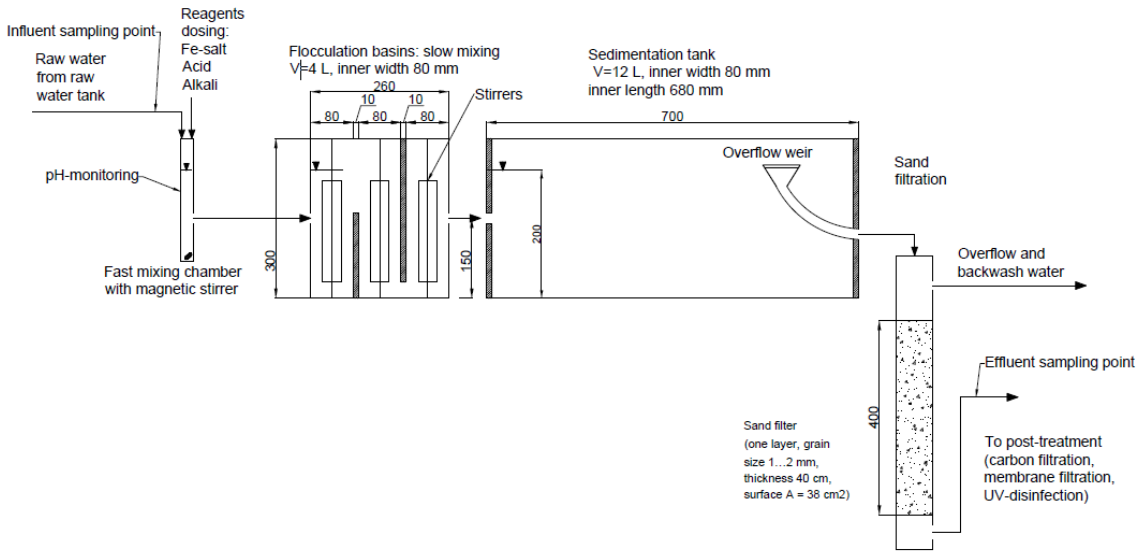


Figure 1. *Process scheme of the reactor.*

General description

The objective is to define the operational parameters of the pilot plant from the design flow rate (approximately 0.1 l/min) and raw water quality. The pilot plant will be started using parameters determined in this task.

Based on the dimensions of the pilot plant in Figure 1 and the given flow rate, calculate the following design parameters:

- detention times of the tanks
- hydraulic surface loading of the settling tank
- weir loading rate

Reporting

Report the calculated design parameters. Assess the applicability of the pilot plant to the flow rate in question by comparing the calculated detention times and loading rates to those recommended in literature (at least one source). Prepare a flow scheme (simple process flow diagram without dimensions) of the reactor and describe in short the water treatment procedure at hand.

Determine the chemical dosage according to the findings from the coagulation and settling experiments (Task B). Calculate a daily use of chemicals using the given design flow rate.

Section 1 Report

Combine the reports from Tasks A, B, and C into a word or PDF file and submit by 16.1.2019 (Group A) or 18.1.2019 (Group B). Each student must submit a report, but feel free to work together (especially with your laboratory partners). Laboratory excel files will be posted to MyCourses.

Section 2: Operating the Water Treatment Pilot Plant

General description

This laboratory work gives an example of how continuous processes are controlled and monitored. Each group will have responsibility over the lab pilot for all the weekdays during one week (see course schedule for weeks). Groups should divide responsibility so that each member will take water samples, analyse the water quality, and observe the performance of the process. Group members are also responsible to check that process equipment are working properly. The objective is for all students to gain hands-on knowledge of water treatment by adjusting and controlling the process for drinking water production during the four weeks of pilot operation. Each group will make at least one adjustment to the process during their week (recommended to make changes on or before day 3) and follow its effect on the process. Additionally, each group is responsible for studying the effects of a different treatment (see Appendix 1). The information gathered during pilot operations will be presented on and compiled to a laboratory report that should follow a typical scientific article format (see Section 3).

Pilot Plant Monitoring

Within the group, decide who is monitoring the process and when. Fill in a student and an approximate time to the monitoring plan in MyCourses. Monitoring should take place during working hours of the lab (8 am – 3 pm), and will likely take at least 1 hour for one person to run simple analyses and check pilot operations (longer for additional tests).

Daily and weekly samples and analyses

The following samples and analysis need to be performed on a regular basis:

- Take daily samples from the raw influent, post-sedimentation, and the sand filter effluent. Analyse UV_{254}, pH and turbidity.
- Twice a week, take additional samples from the post-sedimentation sampling point and the sand filter effluent to measure the alkalinity (standard SFS-EN ISO 9963-1:1996).
- Once a week, take additional samples from the post-sedimentation sampling point and the sand filter effluent to measure the iron concentration.

It is recommended to take samples before making changes to the pilot system, so that samples represent the steady conditions from the previous day.

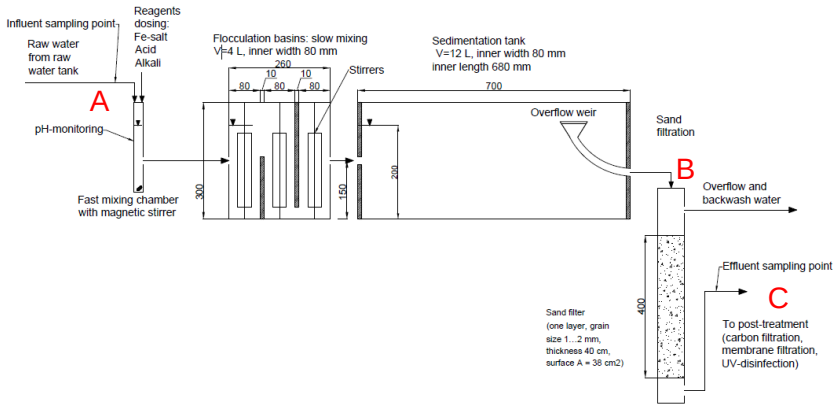


Figure 2. *Process scheme (Figure 1 from page 4) with sampling points called out.*

Process monitoring and pilot upkeep

Familiarise yourself with the operation of a pilot plant and its equipment by testing and adjusting the process. Perform at least the following steps every day:

- Verify the influent flow rate by measuring the yield of the pump per impulse with e.g. a graduated cylinder.
- Verify the functioning of the chemical pumps.
- Check that the chemical reserves are sufficient (can calculate based on flowrate and volume remaining). If needed, replace or refill the chemical container.
    - NOTE: If you prepare a new chemical solution, make sure to clearly label with the concentration and date prepared. It is common laboratory procedure to also write your initials on a solution you have prepared.
- Verify based on a visual judgement that the speed of the mixer is “suitable”
- Record the reported pH value and watch the pH meter to observe the sensibility of pH variance. (What might cause the pH to change too rapidly? Too slowly?)
- Verify the functioning of the sand filter. Check and write down the water level in the filter column. If needed, perform a filter backwash.
    - NOTE: Make sure you have taken your daily samples before performing a backwash or you will need to wait one full column HRT to ensure samples do not contain backwash water.
- At the end of your week, prepare new raw water for the next group. Determine the $KMnO_4$ value of the new raw water.

Process parameter adjustment

Follow the process operation at least for one day before making changes. Based on your assessment of the process performance, try to improve the process by making at least one of the process adjustments:

- Adjust the process pH
- Adjust the feed of ferrous salt
- Adjust the mixing speed

Note that in order to be able to determine the cause and effect correctly it is advisable to change only one parameter at the time and wait for results before making other adjustments.

Monitoring Reporting

Each day, record important information from the pilot operations in the monitoring notebook near the reactor. Be sure to include the date and sign your name. If you performed operation activities or adjustments, add these to the notebook.

Enter your water quality results and your pilot observations as soon as possible in the “Result sheet” provided in MyCourses.

Filtration

Introduction

Filtration is the process of removing solids from fluids (in this case water). Filter beds can be used in many configurations (upflow, gravity flow, vacuum filtration), but conventional water filtration consists of a gravity flow filter bed. This filter bed can be made of a uniform

filter media, often sand or quartz sand, or of two or more layers of different filter media, referred to as mixed-media or multi-media filters. These multiple materials are separated by density and particle size; a common multi-media filter contains low density large anthracite on top, sand in the middle, and denser and finer garnet at the bottom. Solid particulate matter from the water being treated is retained in the filter. When filter pores are clogged by particulate, the head loss in the filter increases. When head loss is above an acceptable level, filters must be cleaned. Filter cleaning is often done by reversing the flow direction and “backwashing” the filter with clean water.

Groups 1 and 2 will run the pilot using a sand filter, while groups 3 and 4 will use a mixed media filter containing calcium carbonate and sand. After all group’s data are available, it should be possible to see the differences caused by the change in filter media.

Filtration theory and filter operation

The removal of suspended particles occurs when suspended particulates are transported to the solid-liquid interface of the filter and then become attached to this surface.

It is generally assumed that suspended particles larger than about 1 µm are transported to the filter media by settling and become attached through interception. Smaller particles are typically transported by Brownian diffusion and Adhere to the surface due to the effect of the van *der* Waals forces. A coagulant might be added to promote additional adhesion. This enhancement is based on charge neutralization and bridging. Figure 3 illustrates different transport mechanisms in filtration.

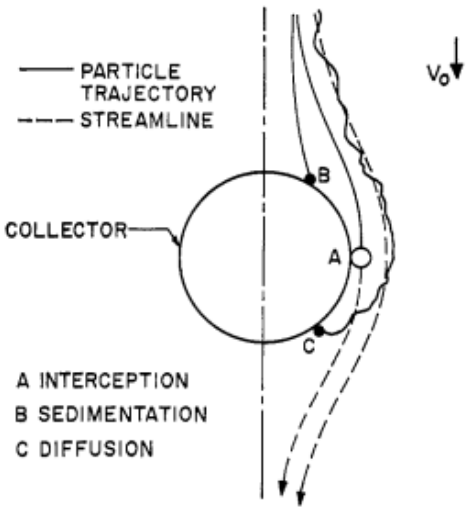


Figure 3. *Basic transport mechanisms in filtration.*

Laboratory exercise

Groups 1 & 2: Run the sand filter continuously during your week of laboratory work.

The sand filter and mixed media filter will be provided, and during the switch from Group 2 to Group 3 filters should be switched.

Groups 3 & 4: Run the mixed-media filter continuously during your week of laboratory work.

All groups:

- Calculate the surface loading of the filter.

- During filter operation, observe changes in the head loss and water level of the filter. After two days' operation, backwash the filter manually and restart the filtration. Observe the effect of washing on the head loss and observe the filtration performance by analysing turbidity of the filtered water.
- Sampling for filtration is included in the daily and weekly sampling plan. Specifically, pay attention to filter effects on UV_{254}, turbidity, alkalinity, and iron concentrations.

Group-Specific Laboratory Work

In addition to daily monitoring, each group will test an additional common treatment process. The process tested will be one of the following: dissolved air flotation (DAF), activated carbon, UV and chlorine disinfection, or nanofiltration. All students from each group should be involved in their group's treatment studies. Please find your group's specific treatment in the Appendices.

Section 2 Report

Daily monitoring results and group treatment results should be entered to the pilot lab notebook and also to the "Result sheet" on MyCourses. Include the date and sign your name or initials when writing to the lab notebook. Your group's data (measurements and notes) should be posted to MyCourses within 2 days of the end of your laboratory work. Results will be used in the presentation and lab report, so make sure to keep clear and detailed notes.

Section 3: Presentation and Lab Report

The results will be reported first in a presentation for the whole course. The presentation should be approximately 20 min and it should contain:

- An overview of the pilot process performance during the week
- The process adjustments made and their effects
- General information about the process studied by the group
- Description of the lab pilot used and analyses carried out, test procedure scheme
- Explanation of the results obtained by the process and design information

The description of the group's process should contain a lot pictures from the lab work.

The results will be also reported in the written report containing:

1. Contribution of the members of the group
2. Introduction
3. Relevant literature background of your group's work (e.g. disinfection for UV group)
4. Methods
    a. Lab reactor description
    b. Group project description
    c. Analytical methods (only a table with reference to used standards)
5. Results of the lab reactor and process optimization
6. Results of the group project (DAF, PAC, UV+chlorination, nanofiltration)
7. Discussion of the treatment performance (comparison of your results to literature values)
8. Conclusions and suggestions for the treatment to be used

Table 1. Assessment weights for the submissions of the course.

Introduction and literature review	3 p
Method description	5 p
Lab reactor results	3 p
Post-treatment results	3 p
Discussion and conclusions	4 p
Total	18 p

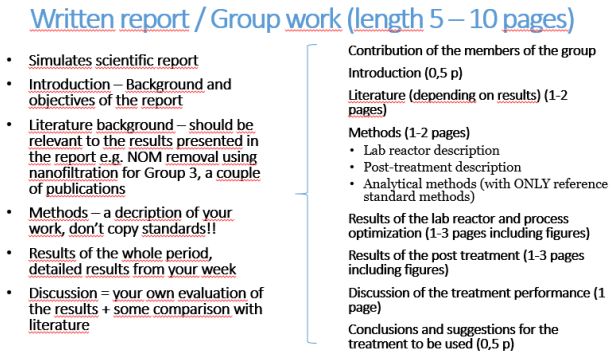


Figure 4. *Guidelines from 2018 course.*

Appendix 1. Group Specific Laboratory Work

Dissolved Air Flotation – DAF (Group 1)

Introduction

Dissolved air flotation (DAF) is an alternative method of solids removal from settling. In flotation, solid separation is obtained by introducing fine gas bubbles into the liquid. Air is dissolved in water under pressure. When this water enters the process, pressure drops and air forms microbubbles in the reactor. The bubbles attach to or form within solids, causing them to float. The buoyant force of the combined particle and bubble causes the particle to rise to the surface, where solids are removed with a skimmer. Flotation is typically used as dissolved-air flotation (DAF), but dispersed-air flotation also exists. Similar to settling, chemicals are often added to increase coagulation and flocculation of particulates together.

Coagulation is the process by which charges are neutralized so that dispersed colloids collect together. Proper mixing is necessary to ensure added coagulant chemicals are evenly dispersed.

Flocculation is a gentle mixing process in which particulate (flocs) come in contact with additional flocs until larger, visible particles are formed. Over-mixing will separate flocs into smaller particles and may hinder flotation or settling.

DAF is particularly effective for removal of oils or lower-density particulate. In general, turbidity and total organic carbon (TOC) content can be used to determine DAF applicability. If TOC and turbidity are low enough, filtration may be a better technology. Similarly, at higher turbidity or when containing denser mineral particulate then settling may be the preferred removal technology. Recommended technologies by turbidity and TOC content is shown in Figure 5 below.

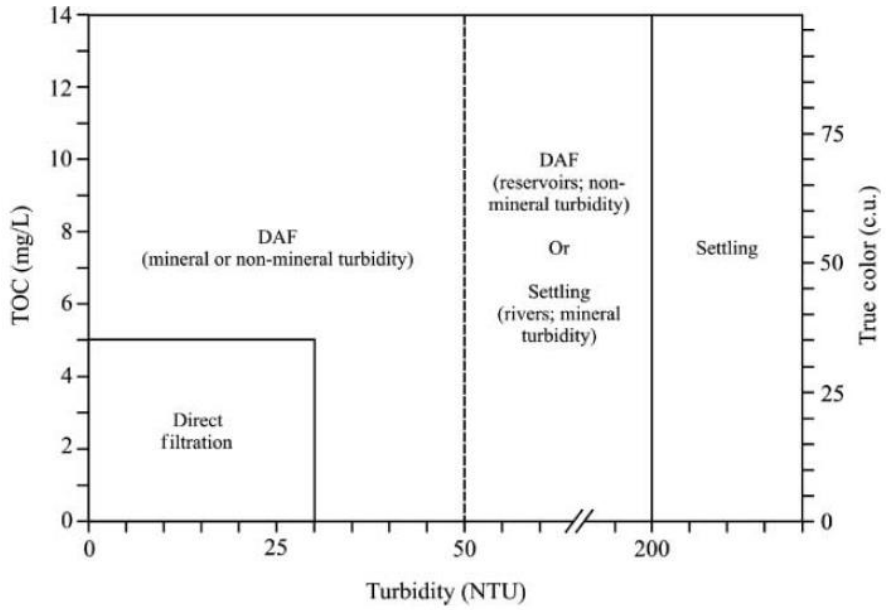


Figure 5. *Process selection diagram highlighting ideal conditions for DAF.*

DAF performance calculations

The performance of DAF can be estimated with the A/S ratio, which is the ratio of air introduced to the reactor (mg/L or ml/L) to the suspended solid concentration of the treated water (mg/L or ml/L).

The ratio can be calculated with the following equation:

$$\frac{A}{S} = \frac{C_S(\frac{f*P}{1atm}-1)}{X_0}$$

Where:

A/S = A/S ratio (mg_{air}/mg_{SS})
C_s is air solubility (mg/L)
f is fraction of air dissolved at pressure P
P is pressure (atm)
X_0 is influent suspended solids (mg/L)

The A/S ratio varies between the type of suspended solids and the type of DAF reactor and it should be determined case by case. A typical range for A/S is 0.01-0.1 mg_{air}/mg_{ss}.

Additional information

To determine the appropriate amount of air injected, use the formula above. A is the necessary amount of air needed for S amount of suspended solids. Typical values for terms are

C_s = 24.3
f = 0.5-0.8
P = ~ 6 bar, check the pressure meter
X_0 = 14.5 mg/l

After finding a value for A, calculate the needed volume of water supersaturated with air. Note that the theoretical value may be too low in reality.

Laboratory Exercise

Sedimentation and DAF columns will be run side-by-side to compare the effect of DAF and sedimentation on solids and turbidity.

Experimental condition:

Prepare DAF and sedimentation columns. Once columns are filled with 5 L of water, perform following steps in both columns for coagulation and flocculation:

1. Add 12,5 ml of diluted PIX while rapid mixing (once coagulate added, time is t_0=0)
2. Rapid mixing (300 rpm) for 60 seconds (t_1)
3. Slow mixing (45 rpm) 5 minutes (t_2)

Sedimentation:

4. Stop mixing and start sedimentation for 15 minutes (t_5)

5. Sample from the surface when the water is clear enough. Note the sampling time. Collect at least 2 liters.

Dissolved air flotation:

4. Inject air via opening 1 (Figure 6 below). Test beforehand how long you need to keep the valve open for the amount of water needed. The amount of injected air depends on the amount of solids in the processed water. See additional information.
5. Slow mixing for 15 seconds (t_4)
6. Stop mixing and allow flotation for 5 minutes (t_5)
7. Sample from the bottom with opening 2 (Figure 6 below). Note the sampling time. Collect at least 2 liters.

Compare the separation times as the flotation and sedimentation tests are running in parallel.

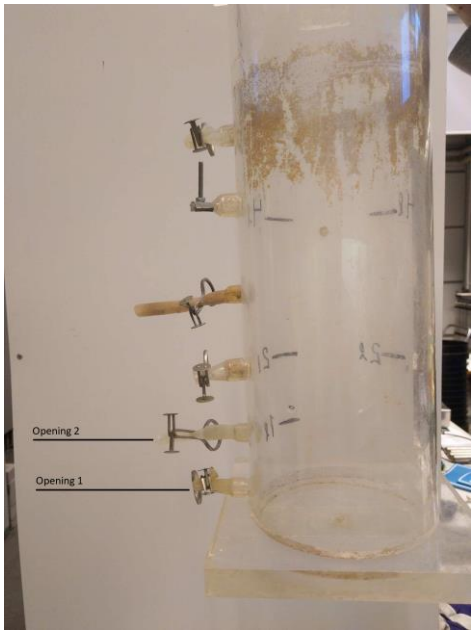


Figure 6. *Column for DAF tests. Air is added from opening 1, and samples can be taken from opening 2 to assess water quality.*

Samples will be analysed for turbidity, suspended solids (SS), and total solids (TS) following test standards SSFS-EN 872:2005, and SFS 3008:1990, respectively.

Activated carbon (Group 2)

Introduction

Adsorption of impurities to activated carbon is used often in water treatment to remove organic compounds and chlorine residual that may cause taste and odour problems. Activated carbon is manufactured by burning carbon-containing materials like wood, peat or coal in anoxic conditions. The materials are “activated” in high temperature as the organic matter is desorbed out of the material, creating a large capacity to adsorb organic molecules back into the carbon. Adsorption capacity increases as the surface area of the activated carbon pores increases. The theoretically measured surface area (BET surface area) often ranges from 500-1500 m^2/g. Used activated carbon can be regenerated by heating it to around 800 °C. Depending on the quality of the substances adsorbed, chemical cleaning might be needed.

Activated carbon can be used either in a batch adsorption process by adding it as powdered activated carbon (PAC) to the water to be treated, or in a continuous flow granular activated carbon (GAC) filter. The use of PAC is best applicable to small treatment plants or plants with a periodical need to use activated carbon. PAC is mixed with the water and, after a desired contact time (e.g. 1 hour), removed by post-coagulation settlement or by filtering.

When using GAC, adsorption of impurities usually takes place continuously as water flows through a GAC filter. As the adsorption capacity fills, the efficiency of the filter starts to reduce when the filter has reached a so-called breakthrough point. This reduction continues until the saturation/exhaustion point (whole adsorption capacity has been utilised), after which impurities are not removed. Changes in the efficiency can be seen in the measurements as an S-shaped curve (Figure 7).

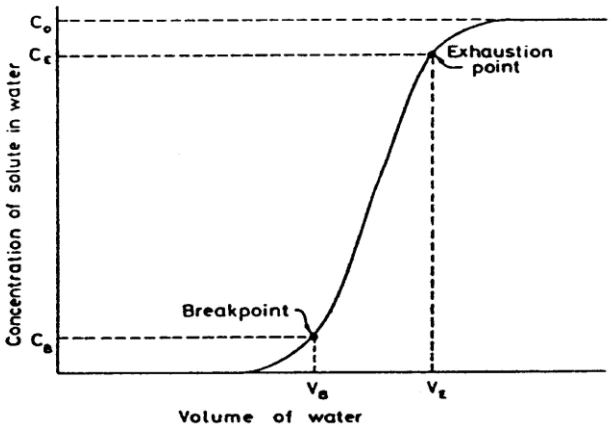


Figure 7. *An adsorption curve of an activated carbon filter (ideal).*

Batch adsorption calculations

The adsorption process can be described by a simple model (Freundlich)

$$q = K_F \cdot C^{\frac{1}{n}}, \quad (1)$$

Where:

q = ratio between the mass of the adsorbed matter and that of the adsorbent (mg/g)
C = concentration of the matter to be adsorbed in water (mg/l)
K_F = constant ((mg/g)(l/mg))
n = constant > 1 (unitless)

The constants K_F and n can be determined by examining how the degree of adsorption depends on different concentrations in solution. Thus, the adsorption model (1) can be modified to

$$\log q = \log K_F + \frac{1}{n} \cdot \log C. \quad (2)$$

By charting log q vs. log C a straight line is obtained, the slope of which is 1/n and the intersection K_F.

In the usage of a batch adsorption, the only thing taking place in water is the adsorption of the matter to the adsorbent. Thus, the mass balance can be formulated as follows:

$$V \cdot C_0 + M \cdot q_0 = V \cdot C_1 + M \cdot q_1 \quad (3)$$

$$V(C_0 - C_1) = M(q_1 - q_0) \quad (4)$$

Where:

V = the volume of the water treated (L)
C_0 = initial impurity concentration (mg/L)
C_1 = final impurity concentration (mg/L)
M = the mass of adsorbent (g)
q_0 = the initial adsorbed mass per adsorbent mass (mg/g)
q_1 = the final adsorbed mass per adsorbent mass (mg/g)

The event can be examined with an adsorption isotherm (Figure 8). C_0 is above the isotherm but C_1 and q_1 are on the isotherm, in which case a negative gradient is obtained for the ratio of the mass of activated carbon and the water treated (M/V).

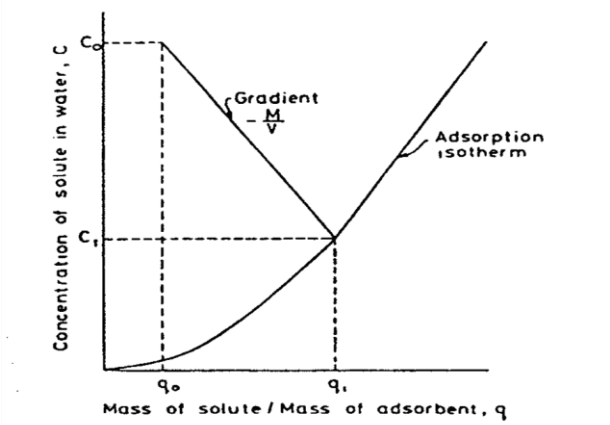


Figure 8. *A single-stage batch adsorption to activated carbon.*

Since the point C_1/q_1 lies on the isotherm, it also follows the equation (1), which, substituted in Eq. 4, gives

$$V(C_0 - C_1) = M(K_F \cdot C_1^{\frac{1}{n}} - q_0). \quad (5)$$

Laboratory exercise

PAC adsorption isotherm batch tests:
Experimental condition:

- Repetition: 2 replicates (conduct each dose in 2 flasks)
- Adsorbent dose: 0.2, 0.4, 0.6, 0.8, and 1.0 g/L

- Volume: 100 mL (per 250 Erlenmeyer flask, 10 flasks total)
- Speed of the shaker: 180 rpm
- Contact time: 1 hour
- pH: natural pH (measure and record the pH of raw water)
- Water: raw pilot water (target pollutant DOC (dissolved organic carbon))
- Room temperature

Experiment procedure:

Weigh required amount of activated carbon for each dose and place it in the flasks. You will need to calculate the mass of activated carbon for the given volume. Use 100 mL raw water for each flask. Put the samples on the shaker for 1 hour at 180 rpm. After the required contact time, filter the samples with a 0.45 µm filter and measure UV absorbance with the spectrophotometer set on 254 nm, the wavelength needed for DOC (dissolved organic carbon) measurement.

Note: you need to also measure the UV absorbance for the lake water to calculate the initial concentration of DOC.

Task:

Calculate the final concertation of DOC for each batch test. Use the average of two replicates to draw the isotherm curve based on the initial (concentration of stock solution) and final concentrations you obtain from the experimental batch tests. Please note that you need to change the measured UV absorbance to DOC concentration, for which you need to use the calibration curve of DOC versus absorbance (provided).

PAC time effect tests:
Experimental condition:

- Repetition: 2 replicates (duplicate samples for each time)
- Constant adsorbent dose: 0.6 g/L
- Volume: 100 mL (per 250 Erlenmeyer flask, 12 flasks total)
- Speed of the shaker: 180 rpm
- Contact times: 1, 5, 10, 30, 60, and 120 minutes
- pH: natural pH (measure and record the pH of raw water)
- Water: raw pilot water (target pollutant DOC (dissolved organic carbon))
- Room temperature

Experiment procedure:

Weigh required amount of activated carbon and place it in the flasks. Use 100 mL raw water for each batch. Put the samples on the shaker at 180 rpm. After each required contact time, remove two flasks and filter samples with a 0.45 µm filter then measure the UV absorbance with the spectrophotometer set on 254 nm.

Note: you can use same initial concentration of DOC from batch tests.

Task:

Calculate the final concertation of DOC for each time. Use the average of two replicates at each time to graph the reduction percentage over time. You will again need to convert the measured UV absorbance to DOC concentration in order to calculate percent reduction of DOC.

Oxidation - UV and chlorine disinfection (Group 3)

Introduction

To secure the hygienic quality of water, water treatment processes normally include disinfection. This refers to eliminating or reducing the number of pathogenic organisms in water (sterilization refers to the total annihilation of pathogens). The disinfection of water can be performed in a variety of ways. The purpose of this laboratory work is to familiarise the student with the use of chlorine and UV disinfection.

Analytical methods

Efficiency of disinfection is evaluated through enumeration (counting colony forming units, CFUs) by membrane filtration method (Tchobanoglous et al., 2003). In membrane filtration method (MF) known volume of water is passed through a membrane filter with pore size 0.45 µm (step 1 in Figure 9).

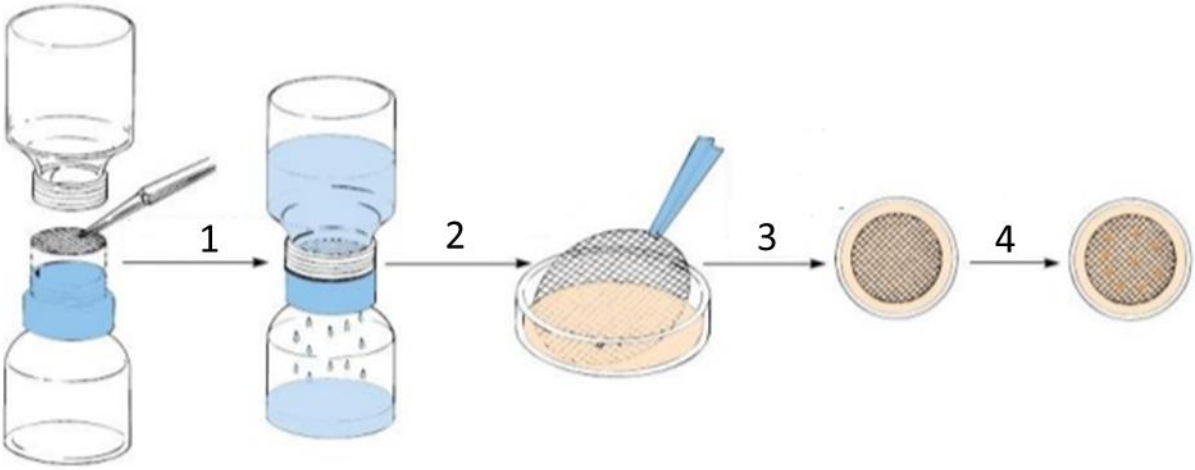


Figure 9. *Membrane filtration method for bacteria enumeration*

Bacteria are retained in the filter because they are larger than the pore size of the filter (see Figure 10).

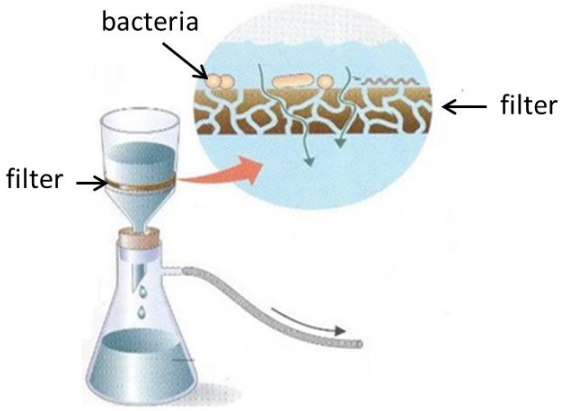


Figure 10. *Retention of bacteria on membrane filter*

The filter with bacteria on it is then placed in Petri dish (step 2 in Figure 9) containing selective medium (needed for growth of target bacteria). After incubation at 35 ± 2ºC during 24 - 48h (step 3 in Figure 9), the colonies formed on the surface of the filter can be counted in order to calculate concentration of target bacteria in water sample (step 4 in Figure 9). *Enterococci* and *Vibrio* spp. are target bacteria in this laboratory work.

The adenosine triphosphate (ATP) assay should be conducted after chlorination (optional) in order to check microbial activity in treated water. In comparison with membrane filtration method, ATP assay is faster and allows to obtain results within minutes. ATP (Figure 11) is primary energy carrier in all living cells. Energy is conserved from inorganic and organic molecules oxidation and stored in cells in the form of ATP (Brock & Madigan, 1991).

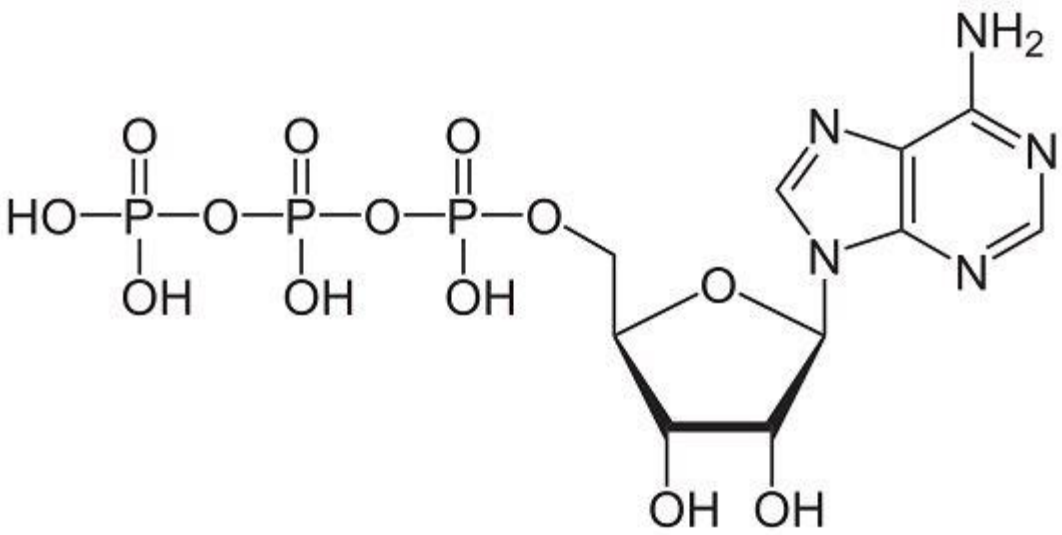


Figure 11. *Adenosine triphosphate (ATP) molecule*

The main principal of ATP bioluminescence method (ASTM, 2009) consists of *i)* extraction of ATP; *ii)* bioluminescence reaction and *iii)* light emission measurement (Figure 12).

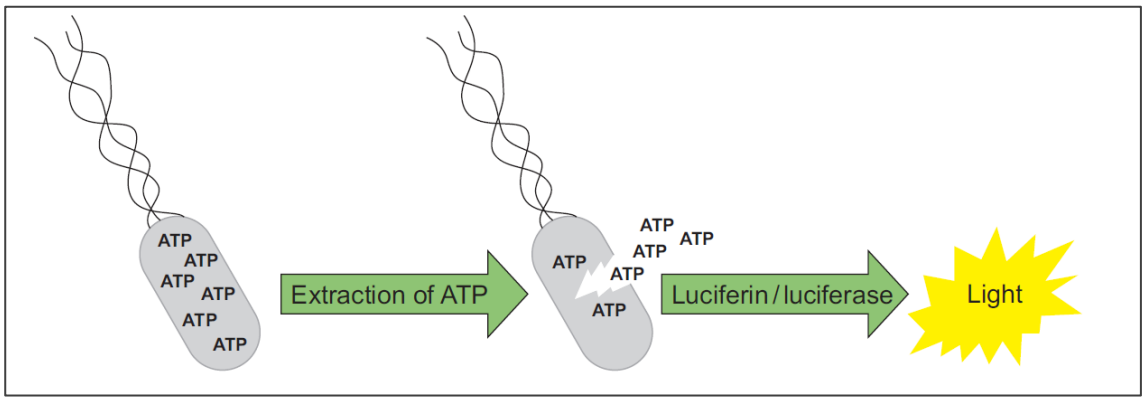
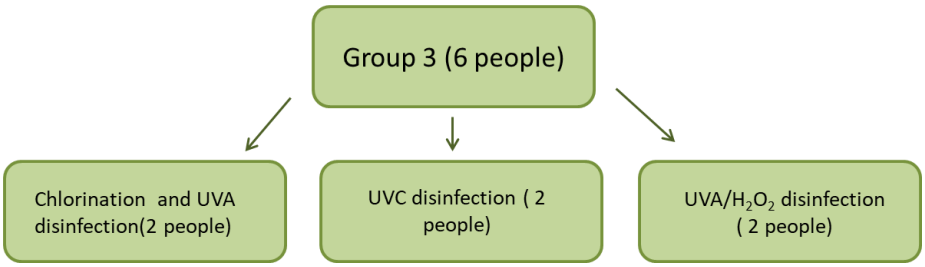


Figure 12. *Principle of ATP assay*

Plan of experiments on disinfection

Group 3 will be divided on three sub groups and each one will conduct different disinfection tests.



Chlorine disinfection

Chlorine is added to water to oxidize bacteria and viruses, and can be added as chlorine gas, sodium hypochlorite (NaOCl), or calcium hypochlorite ($Ca(OCl)_2$).

Higher concentrations of chlorine are able to inactivate viruses and bacteria faster than lower concentrations, so chlorine doses are calculated based on concentration and time (CT).

$$CT_{calc} = t_{contact} * C_{residual\ chlorine} \quad (6)$$

Where:

$t_{contact}$ = *contact time of chlorine. Equal to total detention time multiplied by baffling factor (when traveling through a baffled tank, resulting in mixing). In absence of baffles, contact time is equal to detention time.*

$C_{residual\ chlorine}$ = chlorine remaining after reactions with organisms and organic substances. If chlorine dose is insufficient, there may be no residual chlorine. Too high residual chlorine affects taste and reacts with organic molecules to form disinfection by-products.

Laboratory exercise

Surface water chlorination

Raw water should be poured into four 250 mL sterile bottles, with total volume of 200 mL in each bottle.

Chlorine solution should be added to each bottle in order to get final chlorine concentration 0.1 mg/L, 0.5 mg/L, 1.5 mg/L and 3.5 mg/L. A stock chlorine solution (100 mg/L of Cl) is available.

After adding chlorine, mix the water. After 30 min, take 50 ml sample from each bottle and add 0.25 mL of sodium thiosulfate ($Na_2S_2O_3 \cdot 5H_2O$, 3.5 g/L) to the taken sample in order to interrupt effect of chlorine. The rest of the water (not sample) should be placed in the dark place for 24h or 48h for bacteria regrowth test.

Please use membrane filtration method for enumeration of *Enterococci* and *Vibrio* spp. after chlorination. The volume to be filtered is 50 mL. Place petri dishes for incubation at 35 ± 2ºC for 24h (*Vibrio* spp.) and 48h (*Enterococci*). Obtained results should be

represented as CFU/100mL as a function of chlorine dose. Based on obtained results please discuss how the chlorine dose affects the disinfection.

UVC disinfection

There are many different reactor design for UV disinfection of water. The most commonly used reactors are tubular UVC reactor (Figure 13).

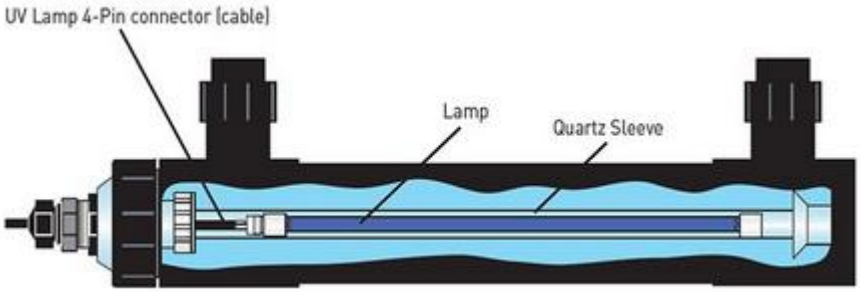


Figure 13. *Tubular UVC reactor. Water passes through the column within a limited range of distances from the UVC lamp and receives a nearly uniform UV dose during residence time within the UV column.*

UV dose is estimated in millijoules per cm^2 (mJ/cm^2) and is calculated using the following simplified equation:

$$UV\ dose = I \cdot t \quad (7)$$

Where:

I = UVC Intensity, measured in milliwatts per cm^2 (mW/cm^2)

t = contact time (seconds)

Laboratory exercise

UVC disinfection of surface water

The UVC disinfection of surface water will be conducted in tubular UVC reactor. The volume of water needed for this experiment is 1.5 L. Experimental set up is shown in Figure 14.

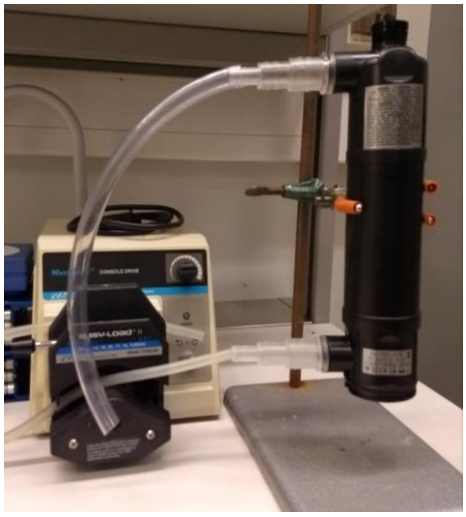


Figure 14. *Experimental set up for UVC disinfection. Water is pumped from a reservoir (not pictured) through the UVC reactor and back to the reservoir.*

Sampling time and volumes of sample are shown in Table below.

Table 2. Sampling time and volume of the sample.

Contact time (sampling time), s	Total sample volume, mL	Sample volumes for MF (Enterococci), mL			Sample volumes for MF (Vibrio spp.), mL		
0	0	0.1	1		0.1	1	
30 (2 min 30 sec)	5	0.1	1		0.1	1	
60 (4 min 59 sec)	15	0.1	1		0.1	1	10
120 (9 min 52 sec)	130	0.1	1	10	1	10	100
240 (18 min)	130	0.1	1	10	10	100	
480 (32 min 32 sec)	130	0.1	1	10	10	100	
600 (36 min 20 sec)	230	1	10	100	10	100	
900 (43 min)	230	1	10	100	10	100	
48 h (bacteria regrowth)			10	100	10	100	

Obtained results should be represented as $Ln(N/N_0)$ as a function of UVC dose (N – number of CFU/100mL). Disinfection efficiency (rate constants) should be evaluated through fitting experimental points in traditional log-linear approach (Chick's law). Rate of disinfection using UVC lamp should be compared with results on UVA disinfection as well as UVA/H_2O_2.

Section specific references

ASTM D4012-81 (2009) Standard Test Method for Adenosine Triphosphate (ATP) Content of Microorganisms in Water, American Society for Testing and Materials (ASTM), standard designation: D4012 – 81 of 1997, reapproved in 2009.

Surface water disinfection via UVA and UVA/H_2O_2

The UV disinfection of surface water should be conducted under UVA intensity (I_0) 22 mW/cm^2, shown in Figure 14 The total volume of surface water is 1200 mL.

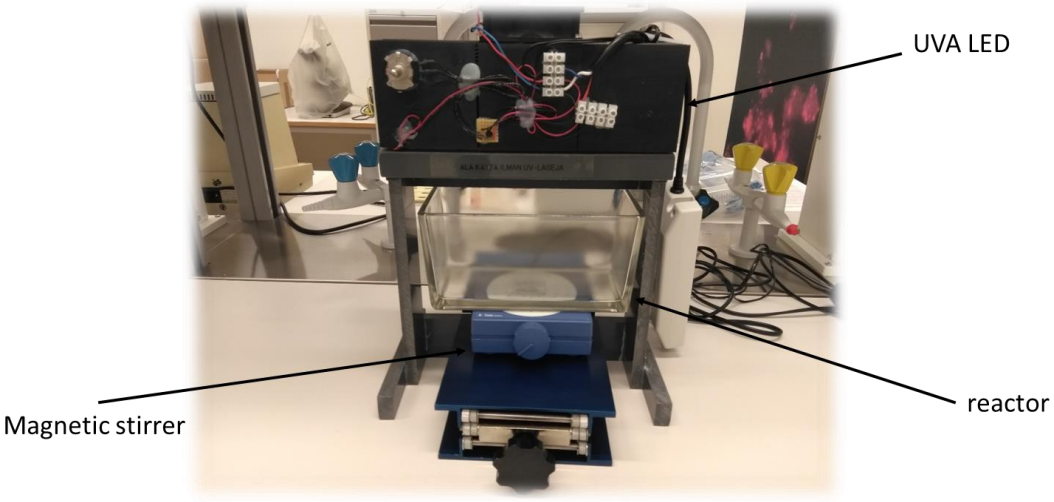


Figure 15. *Experimental set up for UVA water disinfection*

Sampling time and volumes of samples are shown in table below.

Table 3. Sampling time and volume of the sample.

Sampling time, min	Total sample volume, mL	Sample volumes for MF (*Enterococci*), mL			Sample volumes for MF (*Vibrio* spp.), mL		
0 min (**no UVA**)	0	0.1	1	10	0.1	1	10
5 min	15	0.1	1		1	10	
15 min	130	1	10		10	100	
40 min	230	10	100		10	100	
60min	230	10	100		10	100	
90 min	230	10	100		10	100	
48h in darkness (bacteria regrowth)	100		100			100	

Obtained results should be represented in a form of $Ln(N/N_0)$ as a function of UVA dose (N – number of CFU/100mL). Disinfection efficiency (rate constants) should be evaluated through fitting experimental points in traditional log-linear approach (Chick's law). Rate of disinfection should be compared with UVC and UVA/H_2O_2 disinfection tests. Disinfection efficiency should be evaluated through fitting experimental points in traditional log-linear approach for describing microbial inactivation curves:

$$(N=N_0 \cdot e^{-k \cdot UVDose}) \quad (8)$$

where N_0 - initial concentration of bacteria in water, N - CFU/100mL at certain time, k – rate of disinfection.

After that the same experiments (UVA disinfection) should be conducted in combination with hydrogen peroxide (H_2O_2). Concentration of hydrogen peroxide used in this experiment is 20 mg/L. Before switching on the UVA lamp, H_2O_2 should be added to the water. Sampling time and volume of samples are the same as for UVA experiment. Rate of disinfection should be calculated and compared with that of UVA disinfection.

Brock, T.D., Madigan, M.T. (1991) Biology of microorganisms. Prentice Hall International, 6th edition, USA

TCHOBANOGLOUS, George; BURTON, Franklin L.; STENSEL, H. David. *Wastewater engineering treatment and reuse*. Boston, US: McGraw-Hill Higher Education, 2003.

Nanofiltration (Group 4)

This section will be updated closer to the time of the experiment.

Introduction

In nanofiltration, pre-treated water is fed through a membrane with a pore size of 1-10 nm. Particles, polyvalent ions, bacteria, and viruses become concentrated on the feed side of the membrane as concentrate and the treated water (permeate) is recovered from the other side of the membrane. The typical operational pressure is 7-10 bar and clean water recovery is around 85% of the influent water. Nanofiltration has been proved to be very efficient in the removal of organic matter and disinfection by-products in water treatment. The product quality is uniform, and fulfils or exceeds drinking water quality standards. Membrane fouling is one of the most important technical problems in membrane filtration. Fouling is influenced by feed water quality, membrane pore size, and membrane washing frequency/procedure.

Membrane units

There are four kinds of membrane structures on the market: spiral wound modules, hollow fiber modules, tubular modules, and plate and frame modules. In drinking water nanofiltration applications, the spiral modules are most common.

The spiral wound module is made of two nanofiltration membranes with a porous support plate mounted between them. The porous support plate is connected to and wound in a spiral around the permeate collection tube, and feed water must pass through the membrane to exit as permeate (also called product water). Water and impurities that do not pass through the nanofiltration membrane exit as concentrate/brine (Figure 11).

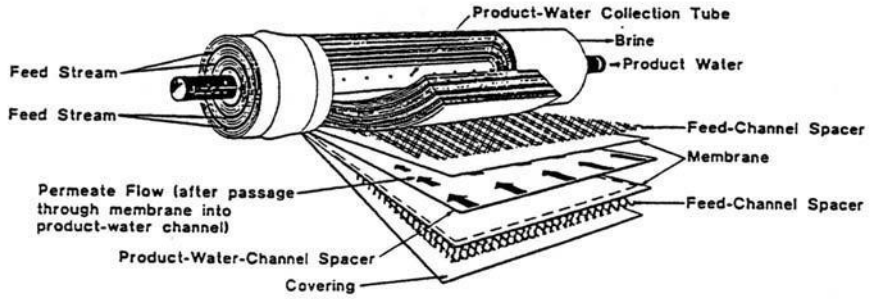


Figure 11. *Spiral module.*

Operation parameters and calculations of nanofiltration

Recovery is calculated by the formula:

$$R = \frac{Q_p}{Q_f}, \qquad (10)$$

Where:

Q_p = permeate flow (m^3/h)
Q_f = feed flow (m^3/h)

Concentration factor of membrane filtration process is calculated as:

$$C_c = \frac{C_f}{1-R} = XC_f \quad (11)$$

Where:

C_c = concentration (mg/l) in concentrate
C_f = concentration (mg/l) in feed water
R = recovery
X = concentration factor (Table 4).

Table 4. *Relationship between recovery and concentration factor*

R	33 %	50 %	67 %	75 %	80 %	90 %	95 %	97,5%	98%	99 %
X	1,5	2	3	4	5	10	20	40	50	100

Flux equations (12-13):

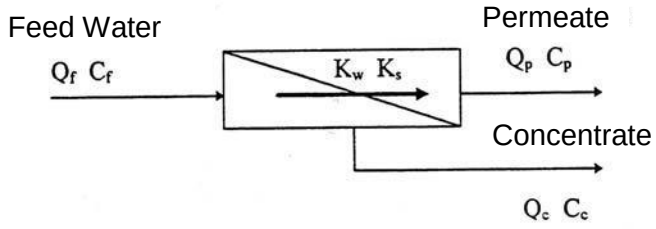


Figure 12. *Mass balance diagram for filtration unit.*

$$F_w = K_W(\Delta P - \Delta\pi) = \frac{Q_p}{A} \quad (12)$$

Where:

F_w = water flux through membrane ($m^3/m^2/h$)
K_w = mass transfer coefficient for water ($g/m^2/h/bar$)
ΔP = pressure gradient across the membrane (bar)
Δπ = osmotic pressure gradient (bar)
Q_p = permeate flow (m^3/h)
A = surface area of membrane (m^2)

$$F_s = K_s\Delta C = \frac{Q_p C_p}{A} \quad (13)$$

Where:

F_s = salt flux through membrane ($g/m^2/h$)
K_s = membrane permeability for salt (m/h)
ΔC = concentration gradient across the membrane (g/m^3)
C_p = concentration in permeate (g/m^3)

Temperature correction factor:

$$k = e^{-0.0352 \cdot (T-20)} \quad (14)$$

Where:

k = temperature correction factor (to 20 degrees ^{o}C)
T = temperature ^{o}C

Net driving pressure (NDP):

$$NDP = \frac{P_f - P_c}{2} - P_p - \pi \quad (15)$$

Where:

P_f = feed pressure (bar)
P_c = concentrate pressure (bar)
P_p = permeate pressure (bar)
π = osmotic (average) pressure of feed water (bar), calculated below (16)

Osmotic pressure:

$$\pi = \frac{nRT}{v} = cRT \quad (16)$$

Where:

n = total amount of dissolves (ions and molecules) (mol)
R= gas constant = 8,314 J/K/mol
T = solution temperature (K)
v = volume of the solution (m^3)
c = sum of the molar concentration of solution (mol/m^3)

Units: $kJ/m^3 = kN/m^2 = kPa = 10^{-2}$ bar

Laboratory exercise

A pilot process described in the Figure 13 is used in the laboratory exercise. The diameter of the element is 6.1 cm, the length is 101 cm, and the surface area 2.6 m^2. Before the membranes, treated water from the pilot is fed through a pre-filter, pore size 5 µm. Concentrate will be recirculated to the feed tank while permeate will be sent to the drain. Permeate and concentrate flows can be read from rotameters. Pressures before and after membranes can be seen from pressure meters. Since permeate filters to an open container, the pressure should be zero.

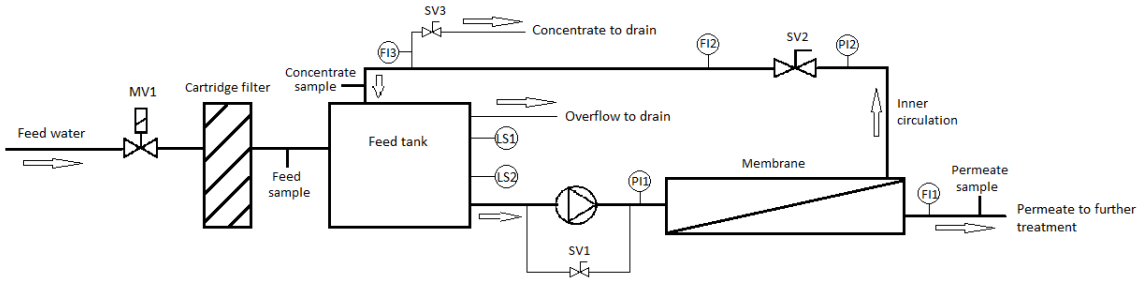


Figure 13. *Nanofiltration pilot.*

Start up the nanofiltration pilot plant. Check the pressures and flows and write them down in the beginning and end of the run. Take samples roughly every 30 minutes from pre-treated water, concentrate, and permeate and measure the UV-absorbance, TOC, $KMnO_4$ and conductivity for each sample. Compare TOC, $KMnO_4$ and UV_{254} analyses.

The effect of nanofiltration on bacteria is followed by analysing the heterotrophic plate count (HPC). Bacteria counts must be made from the original sample water as well as the permeate water. Note that raw water samples must be diluted to 1/10 and 1/100 in saline solutions, while permeate water should not need dilution.

The fouling of the membrane cannot be observed reliably on the grounds of a short test run. Fouling depends on the quality of the pre-treated water and the way the process has been run, requiring test periods of several weeks. In Table 5 are results of a manufacturer-performed five day test run. Calculate (and present as a chart) the reductions of the real and normalized flux during the run.

Table 5. Results of a nanofiltration test by Filmtec NF270 –membrane.

Date	Feed temp	Feed pressure	Concentrate pressure	Permeate pressure	Osmotic pressure	Permeate flow	Retentate flow
	°C	bar	bar	bar	bar	l/h	l/h
3/8	19.0	4.0	2.8	0	0	73	395
3/8	19.2	4.0	2.7	0	0	70	400
4/8	19.1	4.0	3.0	0	0	64	384
7/8	19.3	4.0	2.9	0	0	62	379
7/8	19.2	4.0	3.0	0	0	60	363
8/8	19.2	4.1	3.1	0	0	66	378
8/8	19.2	4.0	3.0	0	0	62	362
9/8	19.0	4.0	3.0	0	0	60	344

Calculate the recovery, concentration factor, flux, net driving pressure, and reduction of analysed parameters. Be prepared to introduce TOC analysis as a part of the group presentation. Normalize the recovery to the temperature of 25 ^oC and to the pressure of 4 bar. The osmotic pressure is not taken into account.

Appendix 2 – List of Standards for analytical methods

SFS-EN ISO 8199, dated 2008 Water quality. General guidance on the enumeration of micro-organisms by culture

SFS-EN ISO 6222, dated 1999 Water quality. Enumeration of culturable micro-organisms. Colony count by inocculation in a nutrient agar culture medium

SFS 3036, dated 1981 Veden kemiallinen hapenkulutus (in Finnish)

SFS-EN ISO 7027, dated 2000 Determiantion of turbidity

SFS 3028, dated in 1976 Veden raudan määritys

SFS-EN ISO 7887, dated 2012 Examination and determination of colour

SFS-EN 1484, dated 1997 Guidelines for the determination of total organic carbon (TOC) and dissolved organic carbon (DOC)

SFS-EN 27888, dated 1994 Determination of electrical conductivity

SFS-EN ISO 10523, dated 2012 Water quality. Determination of pH

SFS-EN ISO 9963-1, dated 1996 Water quality. Determination of alkalinity. Part 1: Determination of total and composite alkalinity

SFS-EN 872, dated 2005. Water quality. Determination of suspended solids. Method by filtration through glass fibre filters

SFS 3008, dated 1990 Veden, lietteen ja sedimentin kuiva-aineen ja hehkutusjäännöksen määritys

ATP Biomass kit HS instructions by BioTherma