# BYM3202 ENGINEERING LABORATORY PRACTICAL HANDBOOK 

Course Coordinator
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Laboratory Coordinator
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## Laboratory Rules

## Class ID: 42835502 (Gr.1) Class Enrollment Key: BYM3202GR1

- Upload your reports to the Turnitin system by 23:59 on Sunday of the next week at the latest!!
- It is sufficient for one person from each group to register and upload your final report in the system. Write ING/Group Number on the 'Your First Name' tab to show that you are an English group. On the 'Your Last Name' tab, write your Experiment Name.
- Please do not write your own name and surname!!
- In the Turnitin plagiarism rate section of the report evaluation form;
- Students with a plagiarism rate of over $\mathbf{4 0 \%}$ will receive 0 points.
- Students whose plagiarism rate is between $30-40 \%$ will receive 50 points.
- Students whose plagiarism rate is between $20-30 \%$ will receive 75 points.
- Students whose plagiarism rate is below $20 \%$ will receive 100 points.


## There is no right to revise the Turnitin report.

- Upload your reports to the Turnitin system without removing the cover pages from your reports.
- Please deliver the original versions of the reports to the laboratory coordinator via e-mail, and the hard copies of the reports must be hand delivered to the relevant teaching assistants on Monday of the next week at the latest.
- If the reports are submitted late, each day 10 points will be deducted from reports.
- Laboratory, starts at 13:00, and 15 minutes late arrivals after this time are not accepted to the laboratory and cannot attend the experiment on that day. Students who will not attend the experiment due to acceptable reasons have to inform laboratory coordinators via e-mail.
- Responsible teaching staff will evaluate the students about experimental study and theory of the experiment. At least, one of the pre-experiment questions will be involved in the quiz. Students who are determined as not ready for the experiment will not be allowed to participate in the experiment.
- Final report prepared by each group must be delivered to the responsible research assistant following laboratory day. Every student will be examined about the content of his/her report by the responsible research assistant.
- All reports, worksheets and projects must be prepared in English.
- Each student will have 1 make-up right, and students who fail from the $2^{\text {nd }}$ experiment they are not allowed to get into another make-up experiment, and you will fail from this course.


## Safety Rules

- White lab coat must be worn in the laboratory.
- Students must also bring laboratory glasses and laboratory gloves.
- Long hair (chin-length or longer) must be tied back to avoid catching fire.
- Know what you are doing by reading the assigned experiment before you start to work. Check the Material Safety Data Sheets (MSDS) and pay close attention to any cautions described in the MSDS.
- Do not taste or smell chemicals.
- Do not lean, hang over or sit on the laboratory tables.
- There is a possibility of an accident in the laboratory during experiments, students should study carefully and not move away from the experiment area during the experimental period, and should report all accidents, injuries, and breakage of glass or equipment to the responsible research assistant immediately.
- All students must keep the experiment area, devices and other glassware materials clean and complete.
- Eating or drinking is prohibited in the laboratory and during presentations.
- Each student must read "Laboratory Safety Booklet" and "Laboratory Safety Information Form".
- Each student must deliver the signed "Laboratory Safety Information Form" to the responsible research assistant.


## Success and Evaluation Status

In-Year Assessment: 40\%

Pre-quizzes: 10\%
Reports: 30\%
Post-quizzes: 20\%
Quiz: 5\%
Presentation: 10\%
Team Member-Team Leader Evaluation Surveys: 15\%
Midterm Exam: 20\%
Final Exam: 40\%

- Before the experiments, responsible teaching assistants will take attendance. $80 \%$ attendance is mandatory.
- Those who arrive more than 15 minutes late for the experiment will not be allowed to get into pre-quiz.
- The student who does not get a passing grade (<50 points) in the pre-quiz (written quiz before the Experiment) will not be allowed to get into the experiment and will have to participate in the Make-up Experiment.
- Each student will have 1 make-up right, and students who fail from the $2^{\text {nd }}$ experiment are not allowed to get into another make-up experiment.
- Students who did not take the post-quiz (oral exam) will be graded zero regardless of their contribution to the report.
- Students who get <50 points in the post-quiz have only 1 make-up chance for each experiment.



## Program Outputs

- 5.2: Ability to conduct experiments to study complex engineering problems or disciplinespecific research topics
- 5.3: Data collection skills for studying complex engineering problems or disciplinespecific research topics
- 5.4: Ability to analyze and interpret experimental results to study complex engineering problems or discipline-specific research topics
- 6.2: Ability to work effectively in disciplinary teams
- 7.1: Ability to make a presentation
- 7.2: At least one foreign language knowledge
- 7.3: Ability to write effective reports and understand written reports
- 7.5: Ability to give clear and understandable instructions
- 9.2: Information about the standards used in engineering applications
- ÖÖ.b: The ability to apply advanced mathematics, science, and engineering knowledge, including differential equations and statistics, to solving problems at the intersection of biology and engineering.

Even if you pass the course, you need to provide the following program outputs. In case you are unable to provide all the outputs in this lesson, you can achieve the outputs with another course that includes common outputs.

## Laboratory Schedule

| Week | Date | Topics |
| :---: | :---: | :---: |
| 1 | February 19 |  |
| 2 | February 26 | Meeting with Students, Introduction to course (Face to Face) (CONFERENCE SALOON) |
| 3 | March 4 | $1{ }^{\text {st }}$ Experiments (Face to Face) |
| 4 | March 11 | $2^{\text {nd }}$ Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of 1 ${ }^{\text {st }}$ Experiments |
| 5 | March 18 | $3{ }^{\text {rd }}$ Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of 2 ${ }^{\text {nd }}$ Experiments |
| 6 | March 25 | $4^{\text {th }}$ Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of $3^{\text {rd }}$ Experiments |
| 7 | April 1 | $5{ }^{\text {th }}$ Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of $4^{\text {th }}$ Experiments |
| 8 | April 8 | HOLIDAY WEEK |
| 9 | April 15 | MIDTERM WEEK |
|  |  | Presentation of Standards for Engineering Applications / Basic Statistics in Engineering Laboratory (Online) |
| 10 | April 22 | QUIZ (about standards and statistics) |
| 11 | April 29 | $6^{\text {th }}$ Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of ${ }^{\text {th }}$ Experiments |
| 12 | May 6 | $7{ }^{\text {th }}$ Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of ${ }^{\text {th }}$ Experiments |
| 13 | May 13 | Make up Experiments (Face to Face) |
|  |  | Final Reports and Oral Exams of ${ }^{\text {7h }}$ Experiments |
| 14 | May 20 | PRESENTATIONS |
|  |  | Final Reports and Oral Exams of Make-Up Experiments |
| 15 | - | FINAL EXAM |

## Experiments and Responsible Teaching Staff

| Experiment | Responsible Teaching Staff | Contact | Laboratory | Time |
| :---: | :---: | :---: | :---: | :---: |
| UNSTEADY STATE <br> HEAT CONDUCTION | Res. Assist. Cem ÖZEL | cemozel@yildiz.edu.tr | Faculty of Chemical- <br> Metallurgical <br> Engineering <br> Laboratory | 13:00 |
| $\begin{gathered} \text { OXYGEN TRANSFER } \\ \text { IN COLUMN } \\ \text { REACTOR } \end{gathered}$ | Res. Assist. Nurseda SÜRGİT | nursedasurgit@gmail.com | Faculty of Chemical- <br> Metallurgical <br> Engineering <br> Laboratory | 13:00 |
| ADSORPTION | Res. Assist. Ayça ASLAN | aslaanayca@gmail.com | Faculty of Chemical- <br> Metallurgical <br> Engineering <br> Laboratory | 13:00 |
| SHELL-TUBE HEAT EXCHANGER | Res. Assist. Yücel <br> Taha UŞA | taha.usa@yildiz.edu.tr | Faculty of Chemical- <br> Metallurgical <br> Engineering <br> Laboratory | 13:00 |
| ASTM DISTILLATION | Res. Assist. <br> Muhammet ÇELİK | mcelik@yildiz.edu.tr | KMD-104 | 13:00 |
| SOLID-LIQUID EXTRACTION | Res. Assist. Gülcan Ayşin KARACA | gakaraca@yildiz.edu.tr | KMD-104 | 13:00 |
| FLUID FLOW | Res. Assist. Elif Nur YILDIZ | nur.yildiz@yildiz.edu.tr | Faculty of Chemical- <br> Metallurgical <br> Engineering <br> Laboratory | 13:00 |

## Experiment Cycle

|  | Mar 4 | Mar 11 | Mar 18 | Mar 25 | Apr 1 | $\underset{8}{\text { Apr }}$ | $\begin{gathered} \text { Apr } \\ 15 \end{gathered}$ | $\begin{gathered} \text { Apr } \\ 22 \end{gathered}$ | Apr 29 | May 6 | $\begin{gathered} \text { May } \\ 13 \end{gathered}$ | $\begin{gathered} \text { May } \\ 20 \end{gathered}$ |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| $\begin{gathered} \text { Group } \\ \text { A1, B1 } \end{gathered}$ | Experiment 1 | Experiment 2 | Experiment 3 | Experiment 4 | Experiment 5 |  |  | $\stackrel{0}{0}$ | Experiment 6 | Experiment 7 | $$ |  |
| $\begin{aligned} & \text { Group } \\ & \text { A2, B2 } \end{aligned}$ | Experiment 2 | Experiment 3 | Experiment 4 | Experiment 5 | Experiment 6 |  |  |  | Experiment 7 | Experiment 1 |  |  |
| Group $\mathrm{A} 3, \mathbf{B 3}$ | Experiment 3 | Experiment 4 | Experiment 5 | Experiment 6 | Experiment 7 |  |  |  | Experiment 1 | Experiment 2 |  |  |
| Group $\mathrm{A} 4, \mathrm{~B} 4$ | Experiment 4 | Experiment 5 | Experiment 6 | Experiment 7 | Experiment 1 |  |  |  | Experiment 2 | Experiment 3 |  |  |
| $\begin{aligned} & \text { Group } \\ & \text { A5, B5 } \end{aligned}$ | Experiment 5 | Experiment 6 | Experiment 7 | Experiment 1 | Experiment 2 |  |  |  | Experiment 3 | Experiment 4 |  |  |
| $\begin{aligned} & \text { Group } \\ & \text { A6, B6 } \end{aligned}$ | Experiment 6 | Experiment 7 | Experiment 1 | Experiment 2 | Experiment 3 |  |  |  | Experiment 4 | Experiment 5 |  |  |
| $\begin{aligned} & \text { Group } \\ & \text { A7, B7 } \end{aligned}$ | Experiment 7 | Experiment 1 | Experiment 2 | Experiment 3 | Experiment 4 |  |  |  | Experiment 5 | Experiment 6 |  |  |


| Experiment 1: | Unsteady State Heat Conduction | Engineering Laboratory |
| :--- | :--- | :--- |
| Experiment 2: | Oxygen Transfer in Column Reactor | Engineering Laboratory |
| Experiment 3: | Adsorption | Engineering Laboratory |
| Experiment 4: | Shell-Tube Heat Exchanger | Engineering Laboratory |
| Experiment 5: | ASTM Distillation | KMD-104 |
| Experiment 6: | Solid-Liquid Extraction | KMD-104 |
| Experiment 7: | Fluid Flow | Engineering Laboratory |

## Experiment Groups Gr. 1

|  |  |  | $\underset{\mathbf{1 , 5}}{\operatorname{Exp} .}$ |  | $\underset{\mathbf{1 , 5}}{\text { Exp. }}$ |  | $\underset{\mathbf{1 , 5}}{\operatorname{Exp} .}$ | DAMLA | $\underset{\mathbf{1 , 5}}{\text { Exp. }}$ |  |  |  | Exp. |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| SELİN ELİTAŞ | $\begin{gathered} \text { Exp. } \\ \text { 1.6 } \end{gathered}$ |  |  |  |  |  |  |  |  |  |  |  |  |
| UĞUR <br> MERT KÖSE | $\underset{2,7}{\text { Exp. }}$ | KEVSER DUMAN | $\underset{\mathbf{2 , 6}}{\text { Exp. }}$ | MUHAMMET FATİH UÇAR | $\underset{\mathbf{2 , 6}}{\text { Exp. }}$ | HALE SELİN GÜNEŞ | $\underset{\mathbf{2 , 6}}{\text { Exp. }}$ | YAĞMUR <br> YILMAZ | $\underset{\text { 2,6 }}{\text { Exp. }}$ | ADNAN FURKAN YILMAZ | $\underset{\mathbf{2 , 6}}{\text { Exp. }}$ | NUR ECE DURAK | $\begin{gathered} \text { Exp. } \\ \mathbf{2 , 6} \end{gathered}$ |
| $\begin{gathered} \text { BETÜL } \\ \text { SURA } \\ \text { AKALIN } \end{gathered}$ | $\begin{gathered} \text { Exp. } \\ 3 \end{gathered}$ | $\begin{gathered} \text { MELİKE } \\ \text { NAZ ŞAYAN } \end{gathered}$ | $\begin{gathered} \hline \text { Exp. } \\ 3,7 \end{gathered}$ | SUDE AZIN | $\begin{gathered} \text { Exp. } \\ \mathbf{3 , 7} \end{gathered}$ | $\begin{gathered} \text { ZEYNEP } \\ \text { FATMA } \\ \text { KARAMAN } \end{gathered}$ | $\underset{\mathbf{3 , 7}}{\operatorname{Exp}}$ | HiLAL <br> YENICI | $\begin{gathered} \text { Exp. } \\ 3,7 \end{gathered}$ | DíLEK <br> AYGÜN | $\begin{gathered} \text { Exp. } \\ \mathbf{3 , 7} \end{gathered}$ | FEYZA AYDIN | $\begin{gathered} \text { Exp. } \\ 3,7 \end{gathered}$ |
| MERVE ORHAN | ${ }_{4}$ | ALARA BAĞCI | Exp. $4$ | MELTEM YILMAZ | Exp. | ESILA NUR KANLI | Exp. | HALE NUR AKARSU | Exp. | $\begin{gathered} \text { DİLEK } \\ \text { YAŞAR } \end{gathered}$ | Exp. | YAREN ELA TÜRTÜK | Exp. |
| $\begin{aligned} & \text { MERYEM } \\ & \text { ŞIFA } \\ & \text { ALTUNHAN } \end{aligned}$ | $\begin{gathered} \text { Exp. } \\ 5 \end{gathered}$ |  |  |  |  |  |  |  |  |  |  |  |  |


|  |  |  | $\underset{\mathbf{1 , 5}}{\text { Exp. }}$ |  | $\underset{\mathbf{1 , 5}}{\operatorname{Exp}}$ |  | $\underset{\mathbf{1 , 5}}{\text { Exp. }}$ |  | $\underset{\mathbf{1 , 5}}{\text { Exp. }}$ |  |  |  |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
| RÜMEYSA ALTAY | $\underset{\mathbf{1 , 5}}{\text { Exp. }}$ |  |  |  |  |  |  |  |  |  |  | ECE GÜLER | $\begin{gathered} \text { Exp. } \\ \mathbf{1 , 5} \end{gathered}$ |
| ZAHIDE <br> KARADAĞ | $\begin{gathered} \hline \text { Exp. } \\ \mathbf{2 , 6} \end{gathered}$ | SENA TÜRK | $\begin{gathered} \text { Exp. } \\ \mathbf{2 , 6} \end{gathered}$ | ESRA AVCI | $\begin{gathered} \hline \text { Exp. } \\ 2,6 \end{gathered}$ | İREM <br> BALIKCI | $\underset{\text { Exp. }}{\text { Exp }}$ | $\begin{gathered} \text { EMİRHAN } \\ \text { KÖSE } \end{gathered}$ | $\underset{\mathbf{2 , 6}}{\underset{\text { Exp. }}{ }}$ | MELIKE <br> SÜRMELİ | $\begin{gathered} \text { Exp. } \\ \hline \mathbf{2 p} \end{gathered}$ | MELİKE DEMIRCIOĞLU | $\begin{gathered} \hline \text { Exp. } \\ \mathbf{2 , 6} \end{gathered}$ |
| MERVE <br> NUR KAYA | $\begin{gathered} \text { Exp. } \\ \mathbf{3 , 7} \end{gathered}$ | RECAİ SONGÜL | $\begin{gathered} \hline \text { Exp. } \\ \mathbf{3 , 7} \end{gathered}$ | AYŞEGÜL RUMEYSA YIKILMAZ | $\begin{gathered} \text { Exp. } \\ \mathbf{3 , 7} \end{gathered}$ | SILA IŞIK | $\begin{gathered} \overline{\text { Exp. }} \\ \mathbf{3 , 7} \end{gathered}$ | BESTE <br> YAĞMUR <br> MANDAL | Exp. | SALİHA ORUÇ | Exp. | EDA AYÇA ARİ | $\begin{gathered} \text { Exp. } \\ 3,7 \end{gathered}$ |
| NiL <br> KORKMAZ | $\underset{4}{\text { Exp. }}$ | ULUÇ BORA <br> ARPACI | Exp. | SEVİM GÜL <br> LENGERLİ | $\begin{gathered} \text { Exp. } \\ 4 \end{gathered}$ | HATICE <br> SIMGE <br> ÖZTÜRK | Exp. | TUNA KOÇ | Exp. | $\begin{gathered} \text { BUSE } \\ \text { YAŞAR } \end{gathered}$ | Exp. | ELİF KÜBRA KILIC KILIÇ | Exp. |

## Experiment 1: UNSTEADY STATE HEAT TRANSFER

## Aim and Theory

Aim of the experiment is the investigation of unsteady-state (transient) heat transfer.

The temperature ( $\mathbf{T}$ ), is measure of the average molecular kinetic energy in the system. Kinetic energy is the energy of motion of matter, according to this the temperature is a measure of the kinetic energy of the molecules. The SI unit of temperature is Kelvin ( K ) however centigrade degrees $\left({ }^{\circ} \mathrm{C}\right)$ unit is also widely used. The heat is the energy which transported between a system and the environment just because of the difference in temperature. The SI unit of heat is Joule ( J ). While the temperature is an internal property of the system, the heat is not. Thus, term of "body temperature" is true but the "body heat" is wrong.
Heat transfer and conversion of the heat into other forms of energy, are the two leading engineering problems. Nowadays, heat transfer has become more important.
$1^{\text {st }}$ and $2^{\text {nd }}$ laws of thermodynamics lay the framework for the science of heat transfer. The basic requirement for heat transfer is the presence of a temperature difference. The temperature difference is the driving force for heat transfer. The second law requires that heat be transferred in the direction of decreasing temperature. Unit area and said heat transmission rate is called the heat flux. The rate of heat transfer per unit area normal to the direction of heat transfer is called heat flux. Heat can be transferred in three basic modes, and each has a special law.

Table 1. Heat transfer modes, and their equations

|  | Heat transfer mode | Special law | Equation |
| :---: | :---: | :---: | :---: |
| 1 | Conduction | Fourier's law of heat conduction | $\dot{Q}=-k A \frac{d T}{d x}$ |
| 2 | Convection | Newton's law of cooling | $\dot{Q}=h A\left(T_{s}-T_{\infty}\right)$ |
| 3 | Radiation | Stefan-Boltzmann law of radiation | $\dot{Q}=\varepsilon \sigma A\left(T_{s u r}^{4}-T_{e n v}^{4}\right)$ |

## Conduction

Conduction is the transfer of energy from the more energetic particles of a substance to the adjacent less energetic ones as a result of interactions between the particles. Conduction is a transferred heat by neighboring electron or photons to each other as a result of vibration. A physical contact must exist between systems with temperature difference. Electrons cannot transfer heat, energy is transferred from neighbor to neighbor such as domino effect (There is no movement of substance). Convection can occur in the solid, liquid and gases. Metals show better conduction because of they have free electrons. Gases show the worst conduction because their distance between electrons is great.
Transfer of heat from the wall of furnace (made of brick) or wall of pipe (made of metal) is typical example for heat transfer with conduction.
Rate of heat conduction for one-dimensional:
$\dot{Q}=k A \frac{\Delta T}{\Delta x}$
$k=$ Thermal conductivity $(\mathrm{W} / \mathrm{m} \cdot \mathrm{K})$
$\Delta T=$ Temperature difference (K)
for $\Delta x \rightarrow 0$ :
$\dot{Q}=-k A \frac{d T}{d x}$
$\frac{d T}{d x}=\vec{\nabla} T$ = temperature gradient
The negativity (-) in the equation (2) indicates that the direction of heat conduction is to decreasing temperature.


Figure 1. The temperature distribution in furnace wall

The application of Fourier's law to two or three-dimensional of conduction, the heat transfer will form a curve instead of the correct line. In Fig. 7.1 is shown as one-dimensional. Figure shows a flat surface of a furnace. The wall is in equilibrium with air and its temperature is 27 ${ }^{\circ} \mathrm{C}$. The temperature distribution inside the wall is shown by line I. In equilibrium, T is not depending on time and situation. The temperature of one side of the wall rises to $650^{\circ} \mathrm{C}$ by a suddenly combustion of the gas then the heat transfer starts. After a certain time-temperature, curve will be as line II. The temperature at a certain distance is represented by c and increases. Now T depends on both time and location. This process is called the unsteady state conduction. Equation 1 can be applied for each temperature and each situation of the case. Lastly, if the wall remains contact with both hot gas and cold air for a long time, the distribution of temperature will be as line III. Heat distribution doesn't change with time. The distribution of constant temperature is called steady state conduction. In Steady state, the temperature is only function of location. The amount of heat transfer is constant in all locations. For steady state and one-dimensional conduction equation 1 becomes as; $q=-k A d T / d x$.

## Convection

Convection is the mode of energy transfer between a solid surface and the adjacent liquid or gas that is in motion. The combined effect of the fluid motion and conduction is called as convection. Convection is heat which carried by the molecules themselves with movement of
the medium. The heated air climb, replaced, so that heat flow occurs. Convection $=$ conduction + hot particle motion (or fluid circulation).
The faster the fluid motion, the greater the convection heat transfer. In the absence of any bulk fluid motion, heat transfer between a solid surface and the adjacent fluid is by pure conduction. Convection is divided into two: if the fluid motion is caused by buoyancy forces that are induced by density differences due to the variation of temperature in the fluid (As the rises and circulation of heated air) "Natural (or free) convection"; if the fluid is forced to flow over the surface by external means such as a fan, pump, or the wind (As the heat transfer from a heated pipe to pumped fluid) "Forced convection". It is as flow of enthalpy. Two types of forces may act at the same time on the same fluid. In this time, natural and forced convection occurs at the same time together. The rate of heat transfer by convection is determined from Newton's law of cooling expressed as;
$\dot{Q}=h A\left(T_{s}-T_{\infty}\right)$ Newton's law of cooling
$h=$ convection heat transfer $\left(\mathrm{W} / \mathrm{m}^{2} .{ }^{\circ} \mathrm{C}\right)$
$A=$ surface area through which convection heat transfer takes place ( $\mathrm{m}^{2}$ )
$T_{s}=$ surface temperature ( ${ }^{\circ} \mathrm{C}$ )
$T_{\infty}=$ temperature of fluid sufficiently far from the surface $\left({ }^{\circ} \mathrm{C}\right)$

## Radiation

Radiation is the energy emitted by matter in the form of electromagnetic waves (or photons) as a result of the changes in the electronic configurations of the atoms or molecules. All solid, liquid and gases, emit, absorb or transmit of radiation of varying degree. Heat transfer takes place only in absorption. Unlike conduction and convection, the transfer of energy by radiation does not require the presence of an intervening medium. In fact, energy transfer by radiation is fastest (at the speed of light) and it suffers no attenuation in a vacuum. All bodies at a temperature above absolute zero ( 0 K ) emit thermal radiation. The maximum rate of the radiation that can be emitted from a surface at an absolute temperature $\mathrm{T}_{\text {sur }}$. is given by the Stefan-Boltzmann law as;
$\dot{Q}=\varepsilon \sigma A\left(T_{\text {sur }}^{4}-T_{\text {env }}^{4}\right)$
Stefan-Boltzman constant: $\sigma=5,67 \times 10^{-8}\left(\mathrm{~W} / \mathrm{m}^{2} . \mathrm{K}^{4}\right)$.
This three heat transfer mechanisms cannot exist together at the same time in the same environment.

## Steady-State Heat Transfer

Heat transfer in an environment, is often dependent on the three-dimensional and time. In another saying temperature may vary depending on time and location. $\mathrm{T}=\mathrm{T}(\mathrm{x}, \mathrm{y}, \mathrm{z} ; \mathrm{t})$. Under Steady state heat transfer the temperature within the system does not change with time. Conversely, under unsteady state heat transfer the temperature within the system does vary with time. In the one-dimensional heat transfer, heat transfer in one dimension is important and negligible in the other two dimensions. In the two-dimensional heat transfer, heat transfer in two dimensions is important and in the third dimension is negligible. In the three-dimensional heat transfer, heat transfer in all dimensions is significant.

The thermal conductivity $(\boldsymbol{k})$, of a material is a measure of the ability of the material to heat transfer. Equation 1 for heat transfer by conduction, is also an equation that defines the thermal conductivity. Thermal conductivity per unit area and unit temperature difference is defined as a unit thickness of the material for said heat transmission rate. High value for thermal conductivity indicates that material is a good heat conductor, low value for thermal conductivity indicates that material is a poor heat conductor or insulator.
Thermal conductivity is not dependent on the temperature gradient. Experiments indicate that the thermal conductivity is not depends on a large temperature gradient (excluding porous material). ( $k$ ) is a function of temperature, but this is not very important. In the slightest temperature change $(k)$ can be considered constant. In the wide temperature changes, thermal conductivity varies linearly with temperature.
$k=a+b T$
Thermal conductivity may vary widely. It takes the highest values for metals and also receives the lowest values for the finely powdered material (if in the vacuum). The average thermal conductivity of silver equals to $357 \mathrm{kcal} / \mathrm{m} . \mathrm{h}$. ${ }^{\circ} \mathrm{C}$. It is reaches to 0.0018 for silica-gel in vacuum.

Table 2. Thermal conductivity values of some materials in room temperature $\left(\sim 20^{\circ} \mathrm{C}\right)$

| Material | $\boldsymbol{k}\left(\mathbf{W} / \mathbf{m} \cdot{ }^{\circ} \mathbf{C}\right)$ | Material | $\boldsymbol{k}\left(\mathbf{W} / \mathbf{m} \cdot{ }^{\circ} \mathbf{C}\right)$ |
| :---: | :---: | :---: | :---: |
| Diamond | 2300 | Human skin | 0,37 |
| Silver | 429 | Wood | 0,17 |
| Cupper | 401 | He (gas) | 0,152 |
| Gold | 317 | Soft rubber | 0,13 |
| Iron | 80,2 | Cooling liquid-12 | 0,072 |
| Mercury | 8,54 | Glass paperwork | 0,043 |
| Glass | 0,78 | Air (gas) | 0,026 |
| Brick | 0,72 | Urethane (solid foam) | 0,026 |
| Water (liquid) | 0,613 |  |  |

Thermal diffusivity ( $\boldsymbol{\alpha}$ ). The product $\rho$. Cp, which is frequently encountered in heat transfer analysis, is called the heat capacity of a material. Both the specific heat Cp and the heat capacity $\rho \mathrm{Cp}$ represent the heat storage capability of a material. But Cp expresses it per unit mass whereas $\rho \mathrm{Cp}$ expresses it per unit volume, as can be noticed from their units $\mathrm{J} / \mathrm{kg} .{ }^{\circ} \mathrm{C}$ and $\mathrm{J} / \mathrm{m} 3 .{ }^{\circ} \mathrm{C}$, respectively. Another material property that appears in the unsteady state heat transfer analysis is the thermal diffusivity, which represents how fast heat diffuses through a material and is defined as;
$\alpha=\frac{\text { heat conducted }}{\text { heat stored }}=\frac{k}{\rho c_{p}}$
$\rho=$ density $\left(\mathrm{kg} / \mathrm{m}^{3}\right)$
$k=$ thermal conductivity $\left(\mathrm{J} / \mathrm{s} \cdot \mathrm{m} \cdot{ }^{\circ} \mathrm{C}\right)$
$C_{p}=$ specific heat $\left(\mathrm{J} / \mathrm{kg} \cdot{ }^{\circ} \mathrm{C}\right)$

Therefore, the thermal diffusivity of a material can be viewed as the ratio of the heat conducted through the material to the heat stored per unit volume.

Table 3. Thermal diffusivity values of some materials in room temperature

| Material | $\boldsymbol{\alpha}\left(\mathbf{x 1 0} \mathbf{0}^{-6} \mathbf{m}^{2} / \mathbf{s}\right)$ | Material | $\boldsymbol{\alpha}\left(\mathbf{x} \mathbf{1 0}^{-6} \mathbf{m}^{2} \mathbf{s}\right)$ |
| :---: | :---: | :---: | :---: |
| Silver | 149 | Ice | 1,2 |
| Gold | 127 | Concrete | 0,75 |
| Copper | 113 | Brick | 0,52 |
| Aluminum | 97 | Glass | 0,34 |
| Iron | 22,8 | Glass wool | 0,23 |
| Mercury (liquid) | 4,7 | Water (liquid) | 0,14 |
| Marble | 1,2 | Beef | 0,14 |
| Wood (oak) | 0,13 |  |  |

## Unsteady State Heat Transfer

The temperature of a body, in general, varies with time as well as position. Conversely, in many cases, for example heating or cooling a mixture, until it reaches to steady state, it is observed that the temperature varies with the time at various points of the system. The term steady state heat transfer implies no change with time at any point within the medium, while unsteady state heat transfer implies variation with time or time dependence. In particular, to determine the specific time of heating and cooling, unsteady state heat conduction equations are used which summarized below.
Consider a one dimensional system -a solid slab- as shown in Figure 2. Assume that the thermal conductivity is constant and that no heat is generated within the slab. of thickness dx at a distance x from the hot face of the slab. Assume that two sides of the element of thickness dx are isothermal surfaces.

The temperature gradient at $\mathbf{x}$ at a definite instant of time is $\partial \mathrm{T} / \partial \mathrm{x}$, heat input in the time interval dt at $\mathrm{x}=-\mathrm{kA}(\partial \mathrm{T} / \partial \mathrm{x}) \mathrm{dt}$

Where A is the area of the slab normal to the heat flow and $k$ is the thermal conductivity of the slab material. The temperature gradient at $(\boldsymbol{x}+\boldsymbol{d x})$ is slight greater than at x , and is given as:

$$
\begin{equation*}
\frac{\partial T}{\partial x}+\frac{\partial}{\partial x} \frac{\partial T}{\partial x} d x \tag{7}
\end{equation*}
$$



Figure 2. Unsteady state heat conduction in slab (one dimensional heat conduction)
Heat flow out of the slab at $(x+d x)=Q x+d x=-k A\left(\frac{\partial T}{\partial x}+\frac{\partial}{\partial x} \frac{\partial T}{\partial x} d x\right) d t$
The heat balance over the element dx is:
Accumulation of the heat in the element $\mathrm{dx}=$ heat input-heat output
$-k A \frac{\partial T}{\partial x} d t-\left[-k A\left(\frac{\partial T}{\partial x}+\frac{\partial^{2} T}{\partial x^{2}} d x\right) d t=k A \frac{\partial^{2} T}{\partial x^{2}} d x\right] d t=k A \frac{\partial^{2} T}{\partial x^{2}} d x d t$
The accumulation of heat in the elemental layer must increase the temperature of the layer. Let $C_{p}$ be the specific heat of the material and $\rho$ be the density of the metal slab,
Mass of the element dx=(density). $($ volume $)=(\rho A d x)$
Accumulation of heat in the elemental layer dx= (mass).(specific heat).(increase in temperature).(dt)
Accumulation of heat in the elemental layer $d x=(\rho A d x) C_{p}(\partial T / \partial t) d t$
This is the change in internal energy of the element due to temperature rise.
The heat balance yields $\rightarrow k A \frac{\partial^{2} T}{\partial x^{2}} d x d t=\rho C_{p} A d x \frac{\partial T}{\partial t} d t$
$\frac{\partial T}{\partial t}=\frac{k}{\rho C_{p}} \frac{\partial^{2} T}{\partial x^{2}} \quad$ and $\quad \alpha=\frac{k}{\rho C_{p}}$
$\frac{\partial T}{\partial t}=\alpha \frac{\partial^{2} T}{\partial x^{2}} \rightarrow \frac{\partial^{2} T}{\partial x^{2}}=\frac{1}{\alpha} \frac{\partial T}{\partial t}$
$T=$ temperature $\left({ }^{\circ} \mathrm{C}\right) \quad t=$ time (h) $\quad x=$ distance (m) $\quad \rho=$ density $\left(\mathrm{kg} / \mathrm{m}^{3}\right)$
$\alpha=$ thermal diffusivity $\left(\mathrm{m}^{2} / \mathrm{h}\right) \quad \mathrm{k}=$ heat transfer coefficient $\left(\mathrm{kcal} / \mathrm{m} \cdot \mathrm{h} \cdot{ }^{\circ} \mathrm{C}\right)$
$C_{p}=$ specific heat $\left(\mathrm{kcal} / \mathrm{kg} \cdot{ }^{\circ} \mathrm{C}\right)$

Where $\alpha=k / \rho$. $C_{p}$ and is called as the thermal diffusivity of the solid material and is a property of the material.

General solutions of unsteady state conduction equations are available for certain simple shapes such as the infinite slab, the infinitely long cylinder, and the sphere. For example, the integration
of equation 11 for heating or cooling of infinite slab of known thickness from both sides by a medium at constant surface temperatures gives.
$\frac{T_{0}-T_{2}}{T_{0}-T_{1}}=\frac{8}{\pi^{2}}\left(e^{-a 1 . N_{F o}}+\frac{1}{9} e^{-9 a 1 N_{F o}}+\frac{1}{25} e^{-25 a 1 N_{F o}}+\ldots\right)$
$T_{0}=$ constant average temperature of surface of slab $\left({ }^{\circ} \mathrm{C}\right)$
$T_{1}=$ initial temperature of slab $\left({ }^{\circ} \mathrm{C}\right)($ for $\mathrm{t}=0)$
$T_{2}=$ average temperature of slab at time for any distance and any time $\left({ }^{\circ} \mathrm{C}\right)$
$N_{F o}=$ Fourier number (defined as $\frac{\alpha t}{s^{2}}$ )
$s=$ one-half slab thickness
$a 1=\left(\frac{\pi}{2}\right)^{2}=2,4674$
In many cases, only the first term of the series in equation 13 is significant and the other terms may be ignored. Under these conditions situation with all except the first term of the series omitted, to give the slab
$\frac{T_{0}-T_{2}}{T_{0}-T_{1}}=0,8106 \cdot e^{\left(-2,467 \frac{\alpha \cdot t}{s^{2}}\right)}$


Figure 3. Average temperatures during unsteady state heating or cooling of a large slab, an infinitely long cylinder or a sphere.

## Pre-Experiment Questions

1. What are the temperature and heat?
2. Explain the heat transfer mechanism of conduction, convection and radiation with writing their equations.
3. What is the unsteady state heat transfer? Explain it with writing its equations.
4. What is the thermal diffusivity? What are the ways of determination of the thermal diffusivity?
5. What is the relationship between the thermal conductivity and thermal diffusivity?
6. Which data will be collected in the experiment? How can the theoretical equations associated with measurable quantities in the experiment?
7. If you wish to use another "X method" to solve for the thermal conductivity you will need to document in detail what technique you applied and how you applied it.
8. What are the parameters that affect thermal diffusivity?

## Experimental Procedure

In chambers of cylindrical apparatus, which made of brass alloys, include silicone oil. Silicone oil is heated before then it is allowed to cool. In this way, a time varying heat transfer is created in brass bar. The temperatures of different point of bar measure by 11 thermometers which placed on the bar with 9.5 cm interval. Changing of heat transfer with time and location examined by regularly reading of the thermometer in different locations. To carrying out the experiment:

1. Place the thermometer into the holes on the brass bar.
2. Insert the plug of heater (include silicone oil) at the same time and starts heating the silicone oil.
3. Read the temperature of thermometer once a minute and write to your notebook.
4. Unplug of heaters when outlet temperature in the end of thermometers reaches to $80^{\circ} \mathrm{C}$.
5. Continue to read and write of the temperature of the thermometer once a minute.
6. Seeing the end of the thermometer gets the highest value; then it begins to decrease.
7. The temperature measured in reference thermometer does not change at the beginning of the experiment, then begins to increase, the experiment ends when it starts to decrease.
8. Close the experimental setup, collect the thermometers and make the necessary cleaning.


Figure 4. Experimental setup of unsteady state heat transfer

Table 4. Temperature of the bar at various distances

| Time <br> $(\mathbf{m i n})$ | Temperature ( ${ }^{\circ} \mathbf{C}$ ( |  |  |  |  |  |  |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- | :--- |
|  | $\mathbf{0 , 4 6 5 m}$ | $\mathbf{0 , 3 8 0 m}$ | $\mathbf{0 , 2 8 5 m}$ | $\mathbf{0 , 1 9 0 m}$ | $\mathbf{0 , 0 9 5 0 m}$ | $\mathbf{0 m}$ | $\mathbf{0 , 0 9 5 0 m}$ | $\mathbf{0 , 1 9 0 m}$ | $\mathbf{0 , 2 8 5 m}$ | $\mathbf{0 , 3 8 0 m}$ | $\mathbf{0 , 4 6 5 m}$ |
| 0 |  |  |  |  |  |  |  |  |  |  |  |
| 1 |  |  |  |  |  |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |  |  |  |  |  |
| $\ldots$ |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |  |  |  |  |  |

## Final Report Should Involve

1. Calculate $\alpha$ value from $\mathrm{T} 0, \mathrm{~T} 1, \mathrm{~T} 2$ and S parameters.
2. Discuss comparing theoretical and experimental values of $\alpha$ for copper alloy.
3. Calculate the T2 temperature by using theoretical $\alpha$. Discuss comparing calculated T2 and experimental T2.
4. Draw the distance-temperature and time-temperature graphs.
5. Explain the any negative condition which prevents operation of the experimental setup/data collection/or reach the aim of the experiment with reasons.
6. Discuss, what you must do, if you wanted to try to determine the repeatability and accuracy of the measurements.

## Experiment 2: OXYGEN TRANSFER IN THE COLUMN REACTOR

## Aim and Theory

A biotechnological process is formed by 3 main steps:

1. Upstream processing - Preparation and pre-treatment of feedstock-raw material.
2. Cultivation - Fermentation or another biological reaction.
3. Downstream processing - Purification of end product.

Each step also has several subunits. The most important of these steps is reproduction of microorganisms in bioreactors.
Basic roles of a bioreactor can be summarized as follows:

- Uniformly mixing of microorganisms and nutrients within the reactor.
- Feeding the biomass with the appropriate amount of nutrients. In all aerobic processes, oxygen transfer occurs in the form of gas/liquid mass transfer. This is not easy due to low oxygen solubility.
- Removal of the heat produced as a result of microbial metabolism.

Performing these roles can only be achieved by thoroughly spreading the applied energy into the system.
Bioreactors are the basic elements of a biotechnological process. Providing microorganisms or other organisms such as animal and plant cells with optimum conditions, e.g. pH , temperature, substrate concentration, is executed by help of bioreactors. There are a lot of different bioreactor types, but these can be divided into two main groups: Mechanically stirring bioreactors and non-mechanically stirring bioreactors-one of which is bubble column reactor.
Below are some advantageous characteristics of bubble column bioreactors in comparison with the other types of bioreactors.

- It is easy to assemble and has no moving parts.
- It needs less space, less coolant, and less energy input, so it is more economic.
- It spreads energy more uniformly.

Some industrial applications of bubble column reactors are:

- Yeast culture.
- Single cell protein production.
- Citric acid production.
- Monoclonal antibody production by using hybridoma cells.

Many physical and chemical parameters such as liquid height, air flow rate, bubble size, liquid viscosity, ionic concentration, and density have effects on mass transfer in column reactors.

The parameters that affect mass transfer in column reactor are:

1. Liquid column height,
2. Ion concentration,
3. Liquid viscosity,
4. Bubble size
5. Air flowrate,
6. Density.

## Interfacial Area and Bubble Behavior

Specific interfacial area (a) is calculated based on liquid volume ( $\mathrm{V}_{\mathrm{L}}$ ); $a=A / V_{L}$

Here, A is the total interfacial area of gas-liquid dispersion. However, specific interfacial area (ad) is used in empirical correlation which is developed for mass transfer;
$\mathrm{ad}_{\mathrm{d}}=\mathrm{A} /\left(\mathrm{V}_{\mathrm{L}}+\mathrm{V}_{\mathrm{G}}\right)$
both specific interfacial areas depends on each other;
$a_{d}=\left(1-E_{G}\right) a$
and $E_{G}$ is represented as;
$\mathrm{E}_{\mathrm{G}}=\mathrm{V}_{\mathrm{G}} /\left(\mathrm{V}_{\mathrm{G}}+\mathrm{V}_{\mathrm{L}}\right)$
EG $_{G}$ is gas hold-up in gas-liquid dispersion. Specific interfacial area is a function of bubble size distribution and a is calculated with this equation;
$\mathrm{a}=6 \mathrm{Eg} /\left[\left(1-\mathrm{EG}_{\mathrm{G}}\right) \mathrm{d}_{\text {avg }}\right]$
davg is average bubble size and can be calculated with function of bubble size distribution. Generally, average diameter based on surface or average Sauter diameter is used;
$d_{\text {Sauter }}=\Sigma n_{i} d_{b, i}{ }^{3} / \Sigma n_{i} d_{b}, i^{2}$

Oxygen transfer rate is important factor in bioreactor used for aerobic culture. It depends on volumetric mass transfer coefficient ( $\mathrm{k}_{\mathrm{L}}$ ). Mass transfer coefficient $\left(\mathbf{k}_{\mathrm{L}}\right)$ is affected by process variations slightly. " a " is specific gas-liquid interfacial area. Because a can be easily changed by using convenient blower or changing power input applied for culture, it is important to find simple and fast method to measure a which controls oxygen transfer rate.
Since "a" strictly depends on liquid composition, chemical methods which requires solution with specific composition, are not appropriate for determine specific interfacial area a. In the present day, it is achieved with physical methods. Geometrical gas-liquid interfacial area a can be determined with Sauter average diameter (ds) of bubble in two-phased system and height of liquid-gas system (gas hold-up= $\mathbf{E}_{\mathbf{G}}$ ). When mass transfer coefficient is calculated, (a) which is average of all geometrical interfacial area is used as:
$\mathrm{k}_{\mathrm{L}}=\left(\mathrm{k}_{\mathrm{L}} \mathrm{a}\right) / \mathrm{a}$
By the equation below, "a" is calculated with ds and EG; accuracy of a depends on sensibility of ds and $E_{G}$ measurement.
$\mathrm{a}=\mathrm{a}^{\prime} / \mathrm{V}_{\mathrm{L}}=6 \mathrm{EG}_{\mathrm{G}} /\left[\mathrm{ds}\left(1-\mathrm{EG}_{\mathrm{G}}\right)\right] \quad \mathrm{d}_{\text {ort }}=\mathrm{dSauter}$

## Measurement of Partial Bubble Size

It is known that partial bubble size can be measured by these methods:
(a) photographically method
(b) electrical conductivity method
(c) electro-optic method
(d) light scattering method

It has been numerous measurement using methods from (a) to (c). Method (d) is generally conducted to organic liquid on which electrical conductivity measurement cannot be performed. Photographically method: Photographically method is usually used to measure bubble
diameter. Bubble diameter d , is identified as reflection of a bubble on a photograph and diameter of a circle which covers same surface area. For small spherical bubbles, this is equal to bubble diameter.

## Total average "gas hold-up"

Column height of liquid-gas system can be measured as gas volume rate for total dispersion volume. We need to measure liquid and gas volume in fermenter to determine gas average volume fraction. Gas volume in fermenter is the difference between measured gas-liquid two phased dispersion volume (Vdisp) and fermentation liquid volume (Vb). Gas hold-up is represented as;
$\mathrm{E}_{\mathrm{G}}=(\mathrm{Vdis}-\mathrm{Vb}) / \mathrm{Vdisp} \quad \mathrm{Vb}=\mathrm{V}_{\mathrm{L}} ; \mathrm{V}_{\text {disp }}=\mathrm{V}_{\mathrm{L}}+\mathrm{V}_{\mathrm{G}}$

Equation 4 is equal to equation 9 .

## Equations for Volumetric Mass Transfer Coefficient "Kla" in Bubble Columns

Gas enters through gas distribution system under reactor in bubble column reactor. If air rate is enough, reactor has heterogeneous flow regime, gas hold-up is a function of superficial gas velocity.
$\mathrm{E}_{\mathrm{G}}=0.6\left[\mathrm{vg}_{\mathrm{g}}(\mathrm{P} / \mathrm{P})\right]^{0.7}$
Mass transfer coefficient $k_{L a}$ is a parameter for scale-up while designing bioreactor. For mass transfer coefficient, this equation is tested for experimentally;
$\mathrm{k}_{\mathrm{La}}=0.32\left[\mathrm{vg}_{\mathrm{g}}\left(\mathrm{P}_{0} / \mathrm{P}\right)\right]^{0.7}$
In the experiment, oxygen transfer in the column reactor, solubility of oxygen gas in water will be examined by changing the parameters such as temperature, pressure, solution composition; and bubble size, gas hold-up will be determined. Aim of experiment is to examine the effects of air flowrate, pressure, temperature and solution composition on oxygen transfer in bubble column reactor.

## Pre-Experiment Questions

1. What is bioreactor?
2. What is bubble column bioreactor and where is it used?
3. What are the differences between designs of continuous and batch reactors?
4. What do you know about the solubility of gases in liquids?
5. Give detailed information about factors affecting oxygen solubility in a bubble column reactor.
6. Which equations are used to calculate oxygen transfer in a bubble column reactor?
7. Sort categories of bubble column reactors by oxygen requirement and medium flow.
8. What is fermentation?
9. What do you know about mass transfer?

## Experimental Procedure

The experiment will be conducted by using a column which is 1,5 meter long and has a diameter of 10 cm . There are 3 ports to make necessary measurements.

- Column is filled with water to a certain height.
- Air is pumped into the column from its bottom. After bubbles are formed by compressor changes the water level, new water level should be determined.
- Photography technique will be used to detect the bubble size. Therefore, a photograph of the column is taken and bubble size is detected by the help of a scale on the column.
- Under different conditions, the dissolved oxygen amount is detected by oxygen measuring device.


## Final Report Should Involve

- Draw the experimental setup in the laboratory and shortly explain its parts.
- Calculate the gas holdup.
- Calculate the mass transfer coefficient and volumetric mass transfer coefficient.
- What is the bubble size?
- Calculate gas-liquid interfacial area.
- Show correlation between oxygen solubility and the parameter that you studied during the experiment.
- Your report should also include the process flow diagram of the experiment.


## Experiment 3: ADSORPTION IN LIQUID PHASE

## Aim and Theory

Application of Freundlich and Langmuir equations on adsorption of acetic acid onto activated charcoal (carbon) is the aim of this experiment.
Adsorption, which is the oldest but least known engineering process, has found a wide application area in the last decades. This advancement is the result of (1) development of purification and separation processes using new types of adsorbents and (2) intensive studies on development of design of adsorption process, which works as a time variable process.
Almost all surfaces attract and accumulate gas molecules and particles dissolved in solutions. Adsorption is adhesion of molecules to the pore walls of a solid which is in contact with one of gas, vapor or liquid phases. Adsorption phenomenon occurs by interaction of molecules in fluid with molecules on solid surface in which intermolecular forces on solid surface are not offset. Adsorption is a surface phenomenon and this process differs from absorption which is used for diffusion of materials into the pores of solid material. Adsorption can be used for separating one molecule from a complex mixture or a solute from a solvent, simply (Fig. 5). Material adhered to the surface is adsorbate. Adsorbent is the surface, which adsorbate is adhered to. Mechanism is of adsorption depends on escaping tendency of solute from solvent and affinity of solute to the solid. Solute is adhered on the specific regions of adsorbent surface. These regions, that solutes are adhered, are called ligands.


Figure 5. Separation based on adsorption
Ligand is a molecular structure that is grafted on the surface of an inert solid material. Affinity binding is a selective adsorption in which strong specific interactions are existing between molecules and ligands of these molecules. Adsorption must be a reversible process in which adhered material is released after a period of time. Release of adhered material from adsorbent is called as desorption and desorption takes place in reverse conditions of adsorption.
There are different forces that cause adsorption. Some of these forces are physical while the others are chemical. Adsorption is a selective process and given below are the factors affecting adsorption:

1. Structure of adsorbate (molecular weight, size and shape)
2. Shape of adsorption area or ligand
3. Polarity of molecules and adsorbent
4. Electrostatic charge on molecule and adsorbent
5. Structure of adsorbent (surface area, particle size)

## 6. Temperature

7. pH

Molecular size of adsorbate becomes a factor that affects adsorption, when intra-particle diffusion is rate limiting step. Increase in molecular size of adsorbate may cause a decrease in diffusion inside the pores and materials with large molecules are adsorbed slower than the materials with small molecules.
Maximum adsorption quantity is proportional to specific surface flow. Adsorbent that has higher pore volume and more granular structure increases adsorption. Pore structure is more important than total internal surface for adsorption. IUPAC (International Union of Pure and Applied Chemistry) classifies pore sizes according to their radius:

1. Macro pores ( $\mathrm{r}>25 \mathrm{~nm}$ )
2. Meso pores ( $1<\mathrm{r}<25 \mathrm{~nm}$ )
3. Micro pores ( $0,4<\mathrm{r}<1 \mathrm{~nm}$ )
4. Sub-micro pores (r $<0,4 \mathrm{~nm}$ )

Micro pores have large volume in adsorbate; therefore they cause high surface area. It makes these pores suitable for adsorption of small molecules. Meso and macro pores form suitable surface area for large molecules.

Solubility of adsorbate is a critical factor that controls adsorption equilibrium in adsorption from solution. Solubility of adsorbate and its adsorption are inversely proportional (Lundelius rule). Increase in solubility cause strong interaction with solute and adsorption decreases.
It was determined that temperature variations between 4 and $60^{\circ} \mathrm{C}$ change adsorption capacity and rate. Adsorption process is exothermic and diffusion processes are endothermic. Therefore, increase in heat causes increase in adsorption rate but, decrease in adsorption capacity. Higher temperatures directly affect ionization and solubility of adsorbate molecules.
pH value of solution affects adsorption capacity. pH affects ionization of solutes and adsorption is lower for ionized molecules.

## Some advantages of adsorption are

1. High selectivity in separation (ex. affinity adsorption)
2. Ability to separate solute from dilute solutions

## Disadvantages of adsorption:

1. It's a surface phenomenon so, adhesion of solute is limited with surface.
2. Adsorption is cyclic so, separation is made in batch mode.
3. Solute material may lose its biological activity.
4. Irreversible adsorption of biological materials may cause distortion of adsorbents

## Important applications of adsorption

1. Protein purification,
2. Nucleic acid purification,
3. Antibiotics purification,
4. Biomedical analyses,
5. Elution chromatography

Adsorption is a spontaneously occurring process and Gibbs free energy change is negative $(\Delta G<0)$. Adsorption causes a more ordered arrangement of molecules on the surface of solid in comparison with gas phase and degree of freedom decreases, which causes negative entropy change ( $\Delta \mathrm{S}<0$ ). According to the equation;
$\Delta \mathrm{G}=\Delta \mathrm{H}-\mathrm{T} \Delta \mathrm{S}$
enthalpy change $(\Delta \mathrm{H})$ is always negative at constant temperature (T). Therefore, adsorption is always exothermic and desorption is always endothermic. Adsorption heat is a measure of magnitude of the forces that causes interactions between adsorbent and adsorbate. When an adsorbent is in contact with a mixture, concentration of components of the mixture differs on adsorbent according to their adsorption energies. This phenomenon helps to separate gases from gases, liquids from liquids and solutes from solutions. It is impossible to completely separate two components with adsorption, since there is equilibrium in adsorption process and both of the components are adsorbed.
Physical adhesion of molecule on adsorbent is occurred depending on one or more non-covalent interactions given below:

1. Van der Waals forces,
2. Electrostatic interactions,
3. Hydrophobic interactions,
4. Hydrogen bonds,
5. Partitioning.

## Types of Adsorbents

Almost all solids may be accepted as an adsorbent but, mostly, granular adsorbents are used in adsorption processes. These adsorbents can be produced from natural or synthetic materials. Adsorbents suitable for industrial processes should;

- be available in large quantities,
- be reusable after regeneration,
- be physically stable (tough),
- be chemically inert,
- have high capacity for desired liquids and gases,
- be selective for a desired substance which will be separated from a mixture.

Most of the adsorbents have amorphous and microcrystalline structure. Today, various adsorptive purification and separation processes are made using specifically developed adsorbents. There are four classes of adsorbents:

1. Activated charcoal (carbon),
2. Active alumina,
3. Silica gel,
4. Molecular sieve zeolites

In biological separations, adsorbents made from cellulose, silica gel, synthetic resins, agarose or cross-linked dextran are mostly used. Granular adsorbents can be used in the forms of suspensions or packed bed.

## Adsorption Isotherms

Analysis of adsorption process depends on defining equilibrium between adhered and nonadhered solutes. Isotherm is the equilibrium equation between concentration of solute in liquid phase and concentration of solute on the surface of adsorbent under a given condition. There are three types of isotherms used in biological separations: linear, Freundlich and Langmuir (Fig. 6)


Figure 6. Adsorption isotherms

Adsorption isotherms are used for defining adsorption properties of adsorbents and determining their adsorption capacity. Isotherms are used;
(1) To choose most suitable adsorbent for the application,
(2) To determine the lifetime of adsorbent,
(3) To determine remaining adsorption capacity of adsorbent.

## Linear Isotherm

When the concentration of solute is very low, most isotherms are linear. Therefore, using linear isotherm is generally suitable, if adsorption of very dilute solutes is investigated.
$\mathrm{q}_{\mathrm{e}} \quad=\mathrm{KC}_{\mathrm{e}}$
$\mathrm{q}_{\mathrm{e}} \quad=$ Quantity of substance adsorbed on the adsorbent ( $\mathrm{mg} / \mathrm{g}$ )
$\mathrm{C}_{e} \quad=$ Quantitiy of non-adsorbed substance which remains in solution ( $\mathrm{mg} / \mathrm{l}$ )
$K=$ Linear equilibrium constant (-)

## Freundlich Isotherm

This isotherm is shown using the equation:
$\mathrm{q}_{\mathrm{e}} \quad=\mathrm{K}_{\mathrm{F}} \mathrm{Ce}^{1 / \mathrm{n}}$
$K_{F} \quad=$ Freundlich constant which shows the magnitude of adsorption capacity depending on temperature, adsorbent and adsorbate;
$\mathrm{n} \quad=$ Freundlich constant which shows the degree of adsorption depending on magnitude of adsorption;
n is smaller than 1 . Freundlich isotherm does not predict the saturation of adhesion surface with solute. Adsorption of antibiotics, steroids and hormones mainly comply with Freundlich isotherm. Reversed phase and hydrophobic interaction also comply with Freundlich isotherm. $\mathrm{K}_{\mathrm{f}}$ and n constants are calculated experimentally (Fig. 7) by using linearized Freundlich equation:
$\log \mathrm{q}_{\mathrm{e}}=\log \mathrm{K}_{\mathrm{f}}+1 / \mathrm{n} \log \mathrm{C}_{\mathrm{e}}$


Figure 7. Determination of Freundlich isotherm constants.

## Langmuir Isotherm

This isotherm is defined by the equation:

$$
\frac{1}{q_{e}}=\left(\frac{1}{Q_{0} b}\right) \frac{1}{C_{e}}+\frac{1}{Q_{0}}
$$

$\mathrm{Q}_{0}$ : saturation constant ( $\mathrm{mg} / \mathrm{g}$ )
b: affinity constant for Langmuir isotherm ( $1 / \mathrm{g}$ )
Langmuir isotherm is generally applied when there is strong interaction between solute and adsorbent. Adsorption types, such as ion exchange and affinity, are generally comply with Langmuir isotherm. This isotherm predicts the saturation of adsorption regions with solute molecules and shows single-layer formation. $\mathrm{Q}_{0}$ and b are the constants and determined experimentally (Fig. 8)


Figure 8. Determination of Langmuir isotherm constants

## Industrial Applications of Adsorption

## Examples of liquid adsorption;

1. Deodorization, drying
2. Removal of dissolved organic substances from drinking water,
3. Removal of odor, taste and coloring substances,
4. Decolorization of raw sugar,
5. Decolorization of vegetable oils,
6. Purification of waste water or industrial waste,
7. Separation of paraffins and isoparaffins.

## Examples of gas adsorption;

1. Recovery of organic solvent vapors,
2. Dehumidification of gases,
3. Removal of odor and toxic substances from air,
4. Separation of organic solvents from air (hydrocarbons, alcohols, etc.)
5. Removal of carbon dioxide and sulphur compounds from natural gas,
6. Separation of paraffins and isoparaffins.

## Pre-Experiment Questions

- What is adsorption?
- In which industrial areas is adsorption used?
- How can I apply solid-liquid adsorption?
- Is adsorption affected from chemical or physical factors?
- Which materials are used as adsorbent?
- How is activated charcoal produced?
- Why is activated charcoal one of the mostly used adsorbents?
- What are the types of activated charcoal according to its shape?
- What is the relationship between adsorption isotherms?
- How can I apply Freundlich and Langmuir equations on data obtained from experiment?
- What are the safety precautions before and during experiment?


## Experimental Procedure

## Materials

* 12 erlenmeyers ( 250 mL ),
* 6 volumetric flasks ( 100 mL ),
* Burette,
- 1 N acetic acid,
* 0.1 N NaOH ,
* Activated charcoal
* Phenolphthalein solution


## Method

Add $50,25,10,5,2.5$ ve 1 mL of 1 N acetic acid to each volumetric flask and dilute each of them to 100 mL . Pour these solutions to separate erlenmeyers and then add 1 g of activated charcoal to each of them. Stir the solutions gently for 1 h . Take samples from clear solutions ( 5 mL from $1^{\text {st }}$ solution, 10 mL from $2^{\text {nd }}$ solution and 25 ml from rest) and add 2-3 drops of phenolphthalein solution to each erlenmeyer. Titrate each sample with 0.1 N NaOH solution. Calculate the concentration of acetic acid in 100 mL solution before and after adsorption. Difference between these values gives the quantity of adsorbed acetic acid. Calculate $\log \mathrm{q}_{\mathrm{e}}$, $\log C_{e}, 1 / q_{e}$ and $1 / C_{e}$ values to draw Langmuir and Freundlich isotherms.

## Final Report Should Involve

- Calculate the quantity of material adsorbed on the solid surface by using data obtained from titrations for each sample.
- Calculate Freundlich and Langmuir constants using isotherm graphics.
- Compare the values of constants with values in literature.
- Determine the suitability of experimental setup according to the comparison of experimental and literature values.
- How could you determine the repeatability and accuracy of experimental results?
- How is adsorption kinetics investigated?


## Experiment 4: SHELL-TUBE HEAT EXCHANGER

## Aim and Theory

One of the most important and common process uncounted in the engineering applications is the heat exchange among two or more fluids at different temperatures. The apparatus that are used to make such changes are called heat exchangers which are, in practice, used in many places, such as thermal power plant, chemical industries, heating, air conditioning refrigeration installations, vehicles, electronic devices, application of alternative energy sources, heat storage etc.
Heat exchangers are widely used in the process industries so their design has been highly developed. Most exchangers are liquid-to-liquid but gas and non-condensing vapors can also be treated in them. The fluids in heat exchangers are generally diverged without mixing with one another by a metallic material surface where heat is directly transferred. For the design of the heat exchangers, material and energy balances are primarily established. The required heat transfer area is calculated by using all heat transfer coefficients and the average temperature difference.
The goal of this test is to recognize shell-tube type heat exchanger and to examine various properties which characterize them.

## You are expected:

- To calculate heat transfer coefficient by using the data obtained from testing apparatus and the basic heat transfer equations as well as associate the coefficient with heat transfer rate,
- To examine the relationship between flow rate/hot fluid temperature and heat transfer coefficient,
- To specify the necessary measurements by the present testing apparatus in order to calculate the heat transfer coefficient,
- To screen out suitability of testing apparatus,
- To report in written the testing period.


## Pre-Experiment Questions

- What are heat and temperature?
- What do I know about heat conduction mechanism?
- What I know about heat exchanger and application area?
- How do I use the basic heat transfer equation?
- What are the important issues that should be considered design of heat exchangers?
- What is the coefficients used in the calculations of heat exchangers?
- What are the factors that change total heat transfer coefficient in a heat exchanger?
- What are the safety precautions that should be taken into consideration during the experiment?
- What are the variables that I should change in order to determine the parameters affecting heat transfer coefficient during the experiment?


## Experimental Procedure

All the heat transfer coefficient and heat loss are calculated by following the below procedures step by step in the experiment.

- Open the thermostat button and set the value of the temperature.
- Close to valve of the tank and fill the tank distilled water up to line you marked.
- Wait for the fluid to flow at a certain flow rate. Also determine the flow rate of water by measuring the time of detention for water in volumetric cylinder.
- Wait for the system to become stable then measure and record Tha: Hot fluid inlet temperature, Thb: Hot fluid outlet temperature, Tca: Cold fluid inlet temperature, Tcb: Cold fluid outlet temperature.
- Repeat the experiment at three different flow rates and thermostat temperatures.
- Close to experimental apparatus and do cleaning.
- Record the experimental data in your notebook.


## Final Report Should Involve

- Tabulate each temperature level and the temperature and the temperature level calculated for flow rate.
- Plot a graph for heat transfer rate versus mean heat transfer coefficient.
- Plot a graph indicating the changes on heat transfer coefficient depending on the flow rate when the inlet temperature of hot water is constant.
- Plot a graph indicating the changes on the heat transfer coefficient by the inlet temperature of the hot water when the flow rate is constant.


## For the shell- tube heat exchanger:

- What are the design parameters? Which parameters can change the heat transfer in shelltube heat exchanger? Explain which parameters were observed to have effect? How can the design parameters (used in the design of the heat exchanger) be changed by:
- Changing flow rate of cold fluid when flow rate of hot fluid and the other parameters are kept constant,
- Changing flow rate of hot fluid when flow rate of cold fluid and the other parameters are kept constant,
- Changing temperature of hot fluid when flow rates of fluids are kept constant,
- Increasing pass number in the system, was the system that you used single pass or multiple pass heat exchanger?
- Examine your test results by comparing them with those reported in the literature. Discuss the possible reasons why the experimental errors have occurred and how to correct them.
- Discuss what you would need to do if you wanted to determine repeatability and accuracy of the test results.
- In your opinion, what are the points that you need to take into consideration in a testing apparatus?


## Experiment 5: SEPERATION OF COMPOUNDS WITH DISTILLATION - ASTM(D 86-61) DISTILLATION

The Aim of The Experiment

With this test method the distillation characteristics of engine and aircraft petrol, turbo-jet fuels, naphtha, kerosine, distillate fuel oil, gas oil and similar petroleum products are determined. Determination of the requirements for storage and use of petroleum products and the determination of whether a foreign substance is added in the products shall be provided by the ASTM boiling curve.

## Theoretical Information

## Oil

Crude oil with very dark colored, slightly or very peculiar odor and a density ranging from 0.8 to 0.95 is a mineral oil that almost all consisting of paraffinic, naphthenic and aromatic hydrocarbons and usually sulfur organic compounds and traces of oxygen and nitrogen compounds.
Also included are inorganic metallic elements in the form of emulsified inorganic salts, as well as salts of fatty acids and naphthoic acids, as well as organic complexes of these elements.

## Distillation

The distillation term is sometimes used for operations (eg water distillation) in which a single component is evaporated from the solution. However, this term is used generically and in situ for the desired operations in which one or more components of the mixture having more than one component in the vapor phase constituted after evaporation of a homogeneous liquid mixture (for example, to separate components of the alcohol-water mixture)
The essential condition for the distillation to be achieved is that in the vapor-liquid system which has reached equilibrium, the vapor phase has a different composition than the liquid phase. If the vapor and liquid phase compositions are the same, no sufficient separation occurs in distillation. Theoretically, completely pure components can not be produced by distillation operation; with it is possible to produce materials of sufficient purity from the economic direction.
Distillation is practically done by the application of one of the two essential methods. In the first method, steam is produced by boiling the liquid mixture containing the desired components to be separated, the steam is condensed by passing through a condenser and immediately taken out. This intensifying new mixture is not allowed to come into contact with the vapor phase by sending it to the distillation unit. In the second method, while some of the condensed steam is taken out as the head product of the distillation column, the other part is sent to the distillation column to make contact with the vapor phase. The second method, which has a very important place in the chemical industry, is known under the name of rectification.


Figure 9. Distillation column

## Distillation of Crude Oil

All of the hydrocarbons have a different boiling point. Above the $380^{\circ} \mathrm{C}$, they undergo breakdown of molecules called cracking. The purification of crude oil is done in 4 ways.
a)Distillation

Normal distillation (products: gasoline, kerosene, diesel)
Vacuum distillation (products: lubrication oils, bitumen, asphalt)
b)Cracking
c)Extraction
d)Chemical Purification

Table 5. Distillation temperatures of various fuels

| Fuel | Distillation temperatures |
| :--- | :--- |
| Petroleum gases | $0-35$ |
| Aircraft fuel | $35-150$ |
| Vehicle gasoline | $35-200$ |
| Gas oil -kerosene | $150-260$ |
| Diesel fuel(thin) | $175-290$ |
| Diesel fuel (Standard) | $200-370$ |
| Heavy Fuels | $370-550$ |

Distillation of oil is based on this difference between boiling points. Crude oil brought to the crude oil tank is sent to the heating oven from here and the temperature is raised to about $380^{\circ} \mathrm{C}$. Crude oil's hydrocarbons, become vapor which boiling points are below this temperature. Heavy hydrocarbons remain liquid.
The inside of the distillation column consists of trays and boiling caps. The vaporized hydrocarbons rise to the top of the column, while the liquid ones descend to the bottom of the column. As the rising vapors go up, they get cold and begin to condense respectively. Condensed liquids fill the trays and when the level reaches the overflow level of first tray, they flow into the other lower tray. Because the lower tray is warmer than the previous one, some of it evaporates again. In this way, hydrocarbons that boiling in specific temperatures will be accumulated respectively in trays according to their boiling points. The liquids taken from these trays are passed through the coolers and the mixtures that we call gasoline, kerosene and diesel are obtained. This means that these three fuels are fundamentally the same thing, and the only difference is that the boiling points or molecular weights of hydrocarbons in the inner of them are different.
The liquid and heavier hydrocarbons that have accumulated underneath the column are conveyed to another distillation column for "Vacuum Distillation" after a second heating. The working principle of this column is the same as the first one. However, a vacuum was formed in the column. This is because the hydrocarbons forming crude oil are exposed to an event called cracking at temperatures above $380^{\circ} \mathrm{C}$.


Figure 10. Tray and Boiling Heads in the Distillation Tower
Distillation is that the heavy molecules break down and become lighter molecules. This is for the purpose of obtain gasoline from the engine, but it is obvious that cracking must be prevented in order to obtain lubricating oils. From various parts of the vacuum distillation column, oils of various weights, or more precisely, various viscosities, are obtained. Bitumen or asphalt remains in the bottom of this column.


Figure 11. Purification and petroleum products of crude oil

## Definitions

Initial Boiling Point (IBP): It is a thermometer reading when the first condensate drop falls to the distillate boiling tray.

End Point / Final Boiling Point (FBP): It is the highest thermometer reading during the experiment which is generally achieved as the moment all the liquid evaporates in the bottom of the balloon. For the same purpose, "the maximum temperature" phrase is often used as a synonym.

Dry Point: The reading of the thermometer corresponding to the evaporation moment of the last liquid droplet in the balloon. In practice, the drops of liquid that are likely to be on the edges of the balloon are not considered.
Note: In general, 'end point' definition is used instead of dry point. The dry point is used, in addition to the boiling point, for special naphthas, such as in the dye industry. Likewise, in accordance with the definitions used today, it is possible to use the dry point in samples which cannot determine of the exact end point.

Degradation point: The temperature recorded in the thermometer reading of the liquid at which the first signs of thermal degradation occur in the balloon.

Amount of collected substance: Volume of liquid drawn in milliliters at temperatures read in correspond to the amount of condensate, accumulated in a 100 ml measure.

Yield percentages: The maximum percentage of liquid that can be withdrawn.
Amount of total yield: The sum of the collected substance volume and the remaining liquid volume in the balloon as milliliter.

Amount of Loss: 100-amount of total yield.
Amount of residue: Amount of total yield minus amount of the collected substance or if directly measured, the amount of residue remaining in the balloon as a milliliter.

Amount of evaporated substances: It is the sum of the amount of collected substance and the amount of loss.

## Materials Required For The Experiment Devices

## DEVICES

Astm distillation apparatus
Astm thermometer
Standard 100 ml measure (measure cylinder)
Filter paper
Astm distillation balloons
Standard balloon supports
Bathroom thermometer
A Beaker ( $600 \mathrm{~cm}^{3}$ )

## Experimental Procedure

- Take personal precautions for both personal and experimental setup.
- 100 ml sample will be gouged to its specification and distilled under specified conditions. Systematic thermometer readings and condensate volume measurements are made. Experimental results will be calculated from this information.
- For this purpose, heat will be applied to the distillation ball and sample.
- When the first boiling point is seen with the liquid droplet coming out, the inner wall of the measuring cup will be connected to the cooler's mouth and the heating will be continued homogenously. Any distillation that does not meet these conditions will be repeated.
Between the first boiling point and the end of distillation, distillate fractions corresponding to the temperatures or temperatures corresponding to the percentage of distillate leaving will be recorded.
- When the temperature reaches $370^{\circ} \mathrm{C}$ or the break point is detected, the heating will be interrupted.
- When you are doing your experiment / experiments, don't forget to process the necessary records into your personal laboratory notebook.


## The Principles Required To Be Learned Before The Experiment

- What safety precautions should I take into account before the experiment and during the experiment?
- What is distillation?
- What are the types of distillation?
- In what areas is the distillation used in industry?
- What are the products obtained as distillation of petrol?
- Which separation methods do you know apart from distillation?
(Note: The theoretical information of the final report must consist of these questions too.)


## Requirements For The Final Report

- Temperatures corresponding to the substances collected in the amounts of $5 \%, 10 \%$, $20 \%, 30 \%, 40 \%, 50 \%, 60 \%, 70 \%, 80 \%, 90 \%$, of the initial boiling point recorded during the test, Amount of collected substances and total yield, amount of loss, amount of residue and Amount of evaporated substances in the given sample.
- Draw an ASTM distillation chart with correspond the amount of collected substance to each respective temperatures to the abscess and the correspond temperatures to the ordinance.
- If there is a negativity prevents you from reaching the purpose of experiment that you have not foreseen at the preliminary stages of experiment which the operation of the experimental setup or data collection please explain these situations in your report with their reasons.
- Discuss what you need to do if you wanted to determine the repeatability and accuracy of your experiment measurements.
- Discuss relevant issues that are not written in the paper and are appropriate for you.
- Avoid quoting directly from the literature when preparing your report.


## Experiment 6: SOLID-LIQUID EXTRACTION

## Aim and Theory

Extraction is the process of transferring one or more components from one phase (mostly solid and liquid) to another (mostly liquid and gas). At the end of this process, the phase in which the substance we are interested in is transferred is called "extract". Extracts are mostly a solution of the substance of interest and other impurities. Extraction as an efficient separation process is used in many areas of industry, such as gold, pharmaceutical, oil, cosmetics, foods etc. At constant temperature and pressure, separation of a desired substance from a mixture is called the extraction.

In the mixture the distribution of a substance between two phases is in equilibrium concentrations. Extractions use two immiscible phases to separate the substance from one phase into the other. It may refer to liquid-liquid extraction, and solid phase extraction. In solid-liquid extraction, the liquid contains the solid substances as solute which is capable of dissolving highly in this liquid. After the phases separated the solid is recovered by removal from any means of fluid substance.

With this experiment, we intent to solid-liquid extraction and find the parameters that affect the yield of extraction process. For oil extraction, we use Soxhlet extractor because it was originally designed for the extraction of lipid from solid material. This experiment explains the principle of Soxhlet device and how any desired component is extracted from solid compound with any solvent.

## You are expected;

- How different parameters influence the amount of extracted substance. Please comment on it. The current experimental setup should be established for obtaining the maximum amount of the substance as yield.
- You need to collect the data and calculate it to determine the amount of end product.
- Refractometer result depends on the amount of substance that needs to extract. During the experiment, you have to correlate between refractometer result and the amount of the mixture (solvent + substance).
- Compare your obtained data with literature one.
- According to the results of this comparison comments on your result.
- Write a report about the steps of the process that you have done.


Figure 12. Soxthlet Extraction


Figure 13. Rotary Evaporator

## Pre-Experiment Questions

1. What is extraction? How many ways of extraction process can be?
2. In which areas of industry extraction process is used?
3. What is refractive index?
4. What amounts of substance are used in this analysis?
5. Which phases occur in the extraction process?
6. What is the basis of separation principle in the extraction process?
7. Which types of solvents are used in Solid-liquid extraction?

## Experimental Procedure:

Soxhlet method is used for determining the extracts or oils. In this method, desired sample is extracted semi continuously with an organic solvent. Solvent, which is heated and volatized, is condensed above the sample. Solvent drips on to the sample to extract. To carry out this experiment;

- Pour the $250-300 \mathrm{~mL}$ solvent round-bottom flask or balloon of Glass Soxhlet with boiling chips. Then place on the eclectic heater.
- Weigh approximately 5 g precisely crushed sample (note that weight for yield calculation) and wrapped in filter paper or placed in a cartridge then put into a siphon extraction chamber or reservoir of extraction apparatus.
- Place the siphon extraction reservoir on the top of the flask.
- Place this siphoning back into the open mouth of the extraction chamber after cooling sited static foot is connected to all devices.
- Make the reverse cooling water inlet and outlet connections.
- Start the extraction procedure for 3-5 siphons.
- Drain all solvent extraction chamber after doing so is discharged from the balloon should be filled up to a maximum of $3 / 4$ reputations.
- After the beginning of extraction, two siphon extractions sample is taken from the container, density or refractive index is determined.
- Density or refractive index remains constant, extraction is terminated.
- Extracted after maintaining this condition all the solvent contained in the reservoir allowing it to siphon into the flask and the heater is turned off.
- Evaporation is carried out in a rotary evaporator to evaporate the solvent remaining in the round-bottom flask or balloon of Glass Soxhlet.
- After the extraction and evaporation process take the filter paper from the cartridge and dried it in the oven for overnight at $40-60^{\circ} \mathrm{C}$.
- Weight the dried sample for calculating how much solvent is evaporated to separate from extract.

$$
\text { Yield of Extraction } \%=\frac{\mid \text { Initial-Final } \mid}{\text { Initial }} \times 100
$$

- The difference between the most 10 mg descriptions exceed state occurs if deemed constant mass and fat percentage of the sample is calculated. By taking into consideration the moisture percentage of the sample, the dry sample is calculated by fat percentage.


## Final Report Should Involve

- Please explain extraction what the process of desired extract.
- Examine the parameters that affect the efficiency of the experiment.
- If any unexpected and unpredictable problem occurred during the experiment that effect the experimental setup / data collection / results, please mention the reasons in your report.
- Discuss what you should do if you need to testify your experiment or/and to get same result again by this experiment.
- Please mention if something is not written in this literature and comments on it.
- Avoid quoting directly from the literature during preparing your report. Copying the sentences from others is academically unethical.


## Experiment 7: FLUID FLOW

## Aim and Theory

In most basic physical operations of engineering applications, flow of liquids and gases is observed. Fluid flow is the term which describes the motion of fluids, like liquids and gases, in an isolated environment such as pipes and ducts. Mass balance, energy balance, and mechanical energy loss resulted from fluid friction are important points to be considered in the analysis of fluid flow. Liquids are incompressible fluids and while analyzing their flow, mechanical and chemical properties of the elements (pipe, valve, elbow fitting, bracket, and many more) that are used in the flow line should be known well.
The aim of this experiment is to analyze the correlation between flow rate and pressure drop during the flow of a fluid through a straight pipe and various fittings. Your responsibility involves;

- Correlating flow rate or fluid velocity with pressure drop.
- Comparing pressure drop for different elements -orifice meter, valves, and elbows- by analyzing the pressure change while flow of the fluid through those elements.
- Determining the equations to calculate the head loss.
- Evaluating the correctness of the experimental setup by carefully examining it.
- Submitting a written report of your entire experiment.


## Pre-Experiment Questions

- What do you know about the flow types? How are flow types determined?
- Can you compare friction coefficient with Reynolds number and surface roughness?
- What do you know about Bernoulli equation, its mathematical expressions, and area of use?
- How many parts is frictional pressure drop divided into? Give detailed information about them.
- What do you know about these flow meters: Orifice meter, venturimeter, and pitot tube?
- What do you know about tube (pipe) fittings and their types?
- What are the safety measures that you have to follow during the experiment?
- What is the first thing to do after you decide the element you will study on?


## Theoretical Information

Fluid current can be divided into three basic types: Laminar flow, transition flow (laminar + turbulence), and turbulent flow. Type of the flow is mainly (but not only) dependent on the velocity of the fluid. For all liquids, the Reynolds number, which is dimensionless, is used to distinguish between laminar flow and turbulent flow.
$\operatorname{Re}=\frac{D u \rho}{\mu}$
D: diameter of the pipe (m)
u : fluid velocity ( $\mathrm{m} / \mathrm{s}$ )
$\rho$ : fluid density $\left(\mathrm{kg} / \mathrm{m}^{3}\right)$

During the flow of a fluid, there is a loss of energy due to friction between the fluid and the flow system, which is called "Friction Losses" or "Head Losses". Friction losses can be divided into two subgroups: Skin Friction and Form Friction Losses (the latter is also known as minor loss). Friction losses manifest themselves in the system with pressure drop.

- Skin friction losses occur between the fluid and the inner surface of the flow pipe. Such friction losses vary depending on the velocity, density and viscosity values of the fluid and the length, diameter and roughness of the pipe.
The roughness in the inner walls of the pipe is absolute roughness ( $\varepsilon$ ), the ratio of absolute roughness to internal diameter is called relative roughness. The Fanning friction factor $(f)$ depends on;

$$
\begin{array}{ll}
\text { For laminar flow } & f \rightarrow \operatorname{Re} \\
\text { For transition flow } & f \rightarrow\left(\operatorname{Re}, \frac{\varepsilon}{D}\right) \\
\text { For fully developed turbulent flow } & f \rightarrow \frac{\varepsilon}{D}
\end{array}
$$

The relationship between the skin friction and the flow conditions in a circular straight pipe is given by Fanning Equation.

$$
\begin{aligned}
& H_{s y}=2 . f \frac{L \cdot u^{2}}{D} \\
& H_{s y}=2 . f \frac{L \cdot u^{2}}{g_{c} \cdot D}
\end{aligned}
$$

(for SI system)
(MKS system)
Because friction losses manifest themselves in the system as pressure drop, if the relationship between pressure drop and friction loss $\left(H_{s y}=\frac{\Delta P}{\rho}\right)$ is combined with Fanning equation, $H_{s y}=\frac{\Delta P}{\rho}=2 . f \frac{L \cdot u^{2}}{g_{c} \cdot D}$ is obtained and Fanning factor is found as $f=\frac{g_{c} \cdot D \cdot \Delta P}{2 \cdot L \cdot u^{2} \cdot \rho}$ from that equation.
$\Delta \mathrm{P}$ : Pressure drop, L: pipe length, D: pipe diameter, $\rho$ : specific weight, $u$ : fluid velocity, $f$ : Fanning friction factor)

In a laminar flow, Hagen-Poiseuille equation is used to calculate the skin friction. As stated above, for a turbulent flow $f$ is related to relative roughness also but this is not the case for a laminar flow. In laminar flows, the equation $H_{s y}=\frac{\Delta P}{\rho}$ is combined with Hagen-Poiseuille equation to calculate skin friction and the following equation is obtained.

$$
H_{s y}=\frac{\Delta P}{\rho}=\frac{32 \cdot L \cdot u \cdot \mu}{g_{c} \cdot D^{2}}
$$

The above equation is then combined with the Fanning friction equation.

$$
H_{s y}=2 . f \frac{L \cdot u^{2}}{g_{c} \cdot D}=\frac{32 \cdot L \cdot u \cdot \mu}{g_{c} \cdot D^{2}} \quad \text { and then, } \quad f=\frac{16 \cdot \mu}{D \cdot u \cdot \rho}=\frac{16}{\operatorname{Re}} \text { (Darcy friction factor) can be }
$$ obtained. So, we can safely say;

the Fanning friction factor $=4 x$ Darcy friction factor
$f$ can be found with the help of the Moody diagram (or Moody chart) given in Fig. 3.3.
In addition to the equations described above, the Reynolds number values between 2100 and 3500 , i.e the transition flow (which can be laminar or turbulent according to the flow conditions in the region), the relationship between Re and $f$ is defined by various equations in the literature. Most of these equations are in the form $f=a+b \cdot \operatorname{Re}^{n}$, where $\mathrm{a}, \mathrm{b}$, and n are constant.
Among the equations given in the literature, the most commonly used equations are the ones developed by Koo.

1. For straight clean pipes made from glass, lead and copper,
$f=0,00140+0,125 \mathrm{Re}^{-0,32}$
2. For steel and iron clean pipes,
$f=0,00307+0,1886 \operatorname{Re}^{0,38}$
In addition to the ones above, the friction loss coefficient can be calculated for smooth pipes from $f=\frac{0,3164}{\sqrt[4]{\mathrm{Re}}}$ if $\mathrm{Re}<100.000$.

- Form friction losses can be categorized as sudden expansion loss, sudden contraction loss, and fittings friction losses. These losses generally are the result of the changes in the geometry and type of the environment (pipe fittings such as elbows, valves, fluwmeters etc) that contains the fluid.
- Connection friction losses can be calculated in two ways. The length of a straight pipe with the same internal diameter as a pipe fitting, which causes a skin friction equal to the friction loss in the fitting, is called the "equivalent pipe length ( $L_{b}$ )" of that fitting. The $L_{b}$ value can be found in the charts.

$$
H s b=2 . f \frac{u^{2} \cdot L_{b}}{g_{c} \cdot D} \text { is the equation to calculate friction loss in such scenarios. }
$$

The second way is to find Kb friction loss constants from tables/charts and using them in the following formula:
$H s b=K_{b} \frac{u^{2}}{2 . g_{c}}$
Table 6. Kb constants of some connectors.

| Fitting | $\mathbf{K}_{\mathbf{b}}$ Constant |
| :--- | :---: |
| $45^{\circ}$ elbow | 0,4 |
| $90^{\circ}$ elbow, standard diameter | 0,9 |
| T connection | 1,8 |
| Nipple | 0,8 |
| Return pipe | 2,2 |
| Angle valve, fully open | 5,0 |
| Ball valve, fully open | 10,0 |

- Sudden contraction friction losses are losses that occur during the flow of fluid from a wide pipe to a narrower pipe. The sudden contraction friction loss coefficient is $\mathrm{K}_{\mathrm{d}}$ and depending on this coefficient, the sudden constriction friction loss (Hsd) is calculated by the following formula.

$$
\mathrm{H}_{\mathrm{sd}}=K_{d} \frac{u^{2}}{2 g_{c}}
$$

u: fluid velocity

For laminar flows

$$
\mathrm{K}_{\mathrm{d}}=0,1
$$

For turbulent flows $\mathrm{K}_{\mathrm{d}}=0,5 .\left(1-\frac{S_{1}}{S_{2}}\right)$, S1 and S2 being the cross-sectional areas of the narrow and wide pipes, respectively.

- Sudden expansion friction losses are losses that occur during the flow of fluid from the narrow pipe to the wider pipe. Sudden expansion friction loss coefficient is $\mathrm{K}_{\mathrm{g}}$ and depending on this coefficient, sudden expansion friction loss (Hsg) is calculated by the following formula.

$$
\mathrm{H}_{\mathrm{sg}}=K_{g} \frac{u^{2}}{2 g_{c}} \quad \text { u: fluid velocity }
$$

$\mathrm{K}_{\mathrm{g}}=\left(1-\frac{S_{1}}{S_{2}}\right)^{2}$
$S_{1}$ and $S_{2}$ : the cross-sectional areas of the narrow and wide pipes, respectively.

## When we put friction losses into the total mechanical energy equation;

$\Sigma H s=\Sigma H_{s y}+\Sigma H_{s b}+\Sigma H_{s d}+\Sigma H_{s g}$
For a laminar flow in such a system, all friction losses can be calculated from the following equation.

$$
\Sigma H s=\Sigma \frac{32 \cdot \mu \cdot u \cdot L}{g_{c} \cdot D^{2} \cdot \rho}+\Sigma \frac{32 \cdot \mu \cdot u \cdot L_{b}}{g_{c} \cdot D^{2} \cdot \rho}+\Sigma K_{d} \frac{u^{2}}{2 \cdot g_{c}}+\Sigma K_{g} \frac{u^{2}}{2 \cdot g_{c}}
$$

Another relationship that applies to both laminar and turbulent flows and including all friction losses;

$$
\Sigma H s=\Sigma 2 \cdot f \frac{u^{2} \cdot L}{g_{c} \cdot D}+\Sigma 2 \cdot f \frac{u^{2} \cdot L_{b}}{g_{c} \cdot D}+\Sigma K_{d} \frac{u^{2}}{2 \cdot g_{c}}+\Sigma K_{g} \frac{u^{2}}{2 \cdot g_{c}}
$$

If the connection head loss is required to be written using the connection friction loss coefficient $K_{b}$, the formula above takes the form below.

$$
\Sigma H s=\Sigma 2 \cdot f \frac{u^{2} \cdot L}{g_{c} \cdot D}+\Sigma K_{b} \frac{u^{2} \cdot L_{b}}{g_{c} \cdot D}+\Sigma K_{d} \frac{u^{2}}{2 \cdot g_{c}}+\Sigma K_{g} \frac{u^{2}}{2 \cdot g_{c}}
$$

If the velocities are equal to each other, the relationship transforms into following equation.

$$
\Sigma H s=\left(\Sigma 4 . f \frac{L}{D}+\Sigma K_{b}+\Sigma K_{d}+\Sigma K_{g}\right) \frac{u^{2}}{2 . g_{c}}
$$

If it is to be used in the Bernoulli equation, head losses are written to the Hs part of the Bernoulli equation.

$$
\frac{p_{a}}{\rho}+\frac{g \cdot Z}{g_{c}}+\frac{u_{a}^{2}}{2 g_{c}}=\frac{p_{b}}{\rho}+\frac{g \cdot Z_{b}}{g_{c}}+\frac{u_{b}^{2}}{2 g_{c}}+\sum H S
$$

## Flow Rate Measurement Methods - One Example

In order to control the physical and chemical processes, the quantities of the substances entering and leaving the process must be known. It is very important to measure the flow velocity of a liquid flowing through a pipe or a duct, as the materials are more easily transported in the liquid state. There are different methods for measuring the flow rate and the most commonly used one is the calculation of the average linear velocity by measuring the pressure drop generated in any part of the flow line. Different devices such as orifice, venturimeter and pitot tube were developed for flow rate measurements based on this foundation.

- Orifice consists of a thin plate which is placed in the flow area and narrows the section to accelerate the flow and thereby reduce the static pressure. Bernoulli and continuity equations are used to measure the average velocity with the orificemeter shown below.


Figure 14. A typical orifice
When the average velocity in the orifice is shown as $u_{0}$ and orifice diameter as $\mathrm{D}_{0}$, the orifice equation can be given as follows. The orifice equation $\mathrm{C}_{0}$ is the dimensionless orifice correction coefficient.

$$
u_{0}=\frac{c_{0}}{\sqrt{1-\left(\frac{D_{0}}{D_{1}}\right)^{4}}} \cdot \sqrt{\frac{2 . g_{c}\left(P_{1}-P_{2}\right)}{\rho}}
$$

## Experimental Procedure

Fluid flow experiment will be completed in four steps. In each step, relationship between flow rate and pressure drop will be determined. In order to conduct the experiment;

- Take necessary safety measures for the experimental setup and yourselves.
- When flow rate regulating valve is fully open, connect the two ends of the manometer to entrance and exit of orifice meter and read the pressure change. Repeat it for elbow, sleeve, pipe fittings, and straight pipe, respectively.
- Repeat the steps above for the half open and closed states of valve.
- Turn off the setup and clean up whatever necessary.

Remember to record data that you acquired throughout the experiment.


Figure 15. Schematic diagram of the experimental setup
Circulation pump (1), Pipe for flow measurement (2), Orifice flowmeter (3), Check valve (4), Gate valve (5), Elbows (6), Pipe couplings (7), Straight pipe (8), Tank (9)

## Final Report Should Involve

- A table showing the readings from manometer for all flow rates.
- Draw graphics of Reynolds number-head loss, pressure drop-flow rate, and square root of pressure drop-flow rate, in Excel. Compare your results with theory.
- Calculate the orifice constant and set up an equation for orifice meter, based on your results.
- Compare the head loss you calculated with values taken from the literature or calculated values from the equation given in the literature. Draw the fluid velocity-friction coefficient graphic in Excel. Find the analytical statement of the line of the graphic.
- Calculate the equivalent lengths for gate valve, check valve, elbows, and sleeves.
- What would happen if both of the valves were half open?
- Discuss what you would do if you were to verify the repeatability and correctness of the results.
- In your opinion, what is the most important point of the experiment to take into consideration?
- Discuss if there is a need to add another element to the experimental setup.


Figure 16. Moody Chart

