PRACTICUM GUIDE

Physical Chemistry II

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PRACTICUM RULES

For students who program the Physical Chemistry II practicum course, they are required to take the Physical Chemistry II practicum and obey the applicable regulations at the Chemistry Laboratory, Faculty of Mathematics and Natural Sciences, Haluoleo Kendari University. Some of the things that every practitioner must know are as follows:

A. Registration

1. Every student who will do the Physical Chemistry II practicum must be registered in the student's Study Plan Card (KRS) as a participant in the Physical Chemistry II Practicum course.
2. Students are required to follow the requirements set by the lecturer and assistant supervisor of the Physical Chemistry II Practicum course.
3. Students are divided into several groups that apply during the Physical Chemistry II practicum activities in the semester in question

B. Trial Assistance

At the beginning of the practicum, the practitioner will be given instructions for each experiment that will be carried out in the semester in question. Students must complete the knowledge underlying the experiment from lecture materials and chemistry, physics, theory and experimental books.

C. Attendance

1. Practitioners are expected to arrive 15 minutes early before the practicum starts
2. Practitioners who are 10 minutes late without a valid/clear reason are considered absent and are not allowed to do practicums.
3. If the practitioner is unable to take part in the practicum (due to illness or other reasons), the head of the level must report it to the lecturer by bringing a certificate
4. Practitioners must participate in experiments for at least 80% of the number of experiments conducted.

D. Safety and Hygiene

1. Practitioners are required to wear a practicum suit complete with their own nameplate during the practicum.

2. Practitioners with long hair must tie their hair.
3. Practitioners are not allowed to smoke, eat, and drink in the laboratory during the practicum.
4. The borrowed tools must be accompanied by a tool voucher that has been approved or initialized by the assistant and after the practicum is completed, the tool must be returned intact and clean.
5. During the practicum, practitioners are not allowed to wear hats and sandals.
6. Each practitioner is required to bring a box containing coarse rags, fine rags, tube brushes, tissue rool, and soap.
7. Practitioners are expected to save on the use of chemicals, water and aquades.
8. After the practicum is over, materials such as tissue paper or spilled chemical residues should be disposed of in the place that has been provided.

E. Implementation of Praktimum

1. Before the practicum begins, the practitioner will respond according to the experiment that will be carried out for 15 minutes.
2. If you are using complicated equipment, ask your assistant to explain the working principle of the tool.

F. Practicum Report

1. The report of each experiment is already available in the practicum instructions, handwritten neatly and cleanly.
2. The report is checked by the practicum supervisor assistant no later than 2 days after the practicum is completed and each practicum brings a practicum control card to be paraphrased by the assistant.

G. Equipment Replacement

1. Practitioners are required to replace broken or damaged equipment before the next practicum accompanied by a memorandum of purchase.
2. The value of the Physical Chemistry practicum will not be distributed to students who are still related to the implementation of the Physical Chemistry II practicum.

EXPERIMENT 1

REACTION SPEED BETWEEN PEROXYDISULFATE AND IOD IONS

Meaning

Studying the kinetics of the reaction between peroxydisulfate and iodine ions

Purpose

1. To show how the reaction speed depends on the concentration of the reactant.
2. To show the assumptions used in differential methods
3. To determine the order of reaction and the setting of the reaction speed at a given temperature.

Theory

Usually the speed of a chemical reaction depends on the concentration of the reaction. This property can be determined by measuring changes in reaction velocity based on changes in the concentration of one of the reactionaries, while the concentration of the other reacts is fixed. The reaction between peroxydisulfate and iodine ions can be written as follows:

$$2\,I^- + S_2O_8^{2-} \rightarrow 2\,SO_4^{2-} + I_2 \qquad (1.1)$$

When the reaction is in progress, the concentration of the reagent will drop so that the reaction speed will change. This phenomenon can be explained in the following ways:

1. A certain amount of thiosulfate ions (S_{2O32-}) are added into the system for a fixed concentration of iod (I^-) ions. The thiosulfate ion reacts with the iodine (I_2) which is formed as a result of the reaction in equation (1). This reaction will form another I^- according to the reaction equation:

   $$I_2 + 2\,S_2O_3^{2-} \rightarrow S_4O_6^{2-} + 2\,I^- \qquad (1.2)$$

   so that the concentration of I^- is always fixed. Yods will be formed when$_{S2O32-}$ is exhausted and I2 can be detected from discoloration by the presence of starch in the system.
2. If the number of moles of S_{2O62-} that reacted is very small compared to the number of moles of S_{2O82-} that were present at the beginning of the reaction, then

only a small number of S_{2O82-} will react before the blue color appears, so the concentration is considered to be constant.

The reaction time is inversely proportional to the reaction speed, i.e. the shorter the time, the greater the reaction speed. The temperature of the experiment needs to be recorded.

Tools and Materials

Tools: 2 test tube racks, 20 test tubes, microburettes, pipettes (1.0 mL and 10 mL), and *stopwatch.*

Material: $Na_2S_2O_8$ 0.04 M, $Na_2S_2O_6$ 0.04 M, KI 0.10 M, 3% starch solution.

How It Works

A. Set up systems 1 through 10 as shown in Table 1. using the following methods:

1. Fill 10 clean, dry test tubes of 10, 9, 8, 7, 6, 5, 4, 3, 2, and 1 mL of S_{2O82-} solution using a Mohr pipette. Add the aqueduct to each tube (except for the tube containing S_{2O82-} as much as 10 mL) until the volume is exactly 10 mL (this solution is called solution A).
2. The other 10 test tubes were each containing 10 mL I^-, 1 mL $S_2O_3^{2-}$ of microburetta and 1 mL of starch solution (this solution is called solution B).
3. Mix the contents of the first tube of solution A with one of the tubes of solution B in the following way: insert the contents of the tube of solution A into tube B and pour it back into tube A as quickly as possible. Time measurement begins at the time of pouring the contents of tube A into tube B and ends at the moment of discoloration (color change occurs gradually).
4. For tubes 2 to 10, the treatment is the same as tube 1.

B. Then prepare systems 11 to 20 as follows:

1. Fill into 10 clean, dry test tubes of 10mL each of S 2O82- solution using a Mohr pipette (this solution is called solution C).
2. Into the other 10 test tubes 10, 9, 8, 7, 6, 5, 4, 3, 2, and 1 mL of solution I- are inserted into the other 10 test tubes each by using a Mohr pipette and add aqueducts to each tube (except for the tube containing 10 mL) until the volume is exactly 10 mL then into each tube add 1 mL of S2O32- of microburetta and 1 mL of starch solution (this solution is called solution D).

3. Mix the contents of the first tube of solution C with one of the tubes of solution D in the same way as done in step A.3 above.

Observation Data

Volume of starch solution = ... mL; Mixture volume = ... mL

Initial molarity $S_2O_3^{2-}$ = ... mL; Temperature = ... ^{oC}

Early molarity I^- = ... mL

Data Calculation and Analysis

1. Calculate the concentrations of $S_2O_3^{2-}$ (peroxydisulfate ions) and I^- (iodine ions) for each system and enter the values into the calculation table.
2. Calculate 1/time for each system
3. Create a curve between the concentration of $S_2O_8^{2-}$ as a function of 1/time and I^- as a function of 1/time.
4. The equation for reaction speed can be written as follows:

$$-\frac{d[S_2O_8^{2-}]}{dt} = k[S_2O_8^{2-}]^x\,[I^-]^y \qquad (1.3)$$

Define x (the order of reaction to$_{S2O32-}$) and y (the order of reaction to I^-) of the curve. Also determine the order of total reaction.

Table 1. Data and Calculation Results

System	Tube 1		Tube 2		Time (seconds)	$S_2O_8^{2-}$	I^-	1/time ($^{sec-1}$)
	Volume $S_2O_8^{2-}$	Volume H_2O	Volume I^-	Volume H_2O				
1	5	0	10	0	…	…	…	…
2	4	1	10	0	…	…	…	…
3	3	2	10	0	…	…	…	…
4	2	3	10	0	…	…	…	…
5	1	4	10	0	…	…	…	…
6	10	0	5	0	…	…	…	…
7	10	0	4	1	…	…	…	…
8	10	0	3	2	…	…	…	…
9	10	0	2	3	…	…	…	…
10	10	0	1	4	…	…	…	…

EXPERIMENT 2

VISCOSITY

Meaning

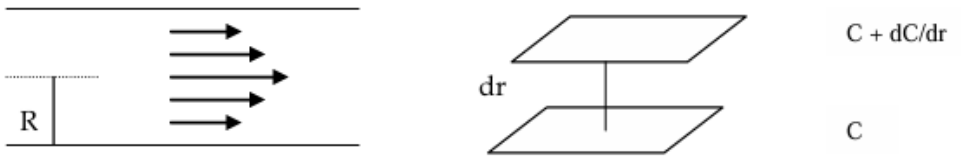
Studying the viscosity properties of liquids

Purpose

1. Determining the viscosity of liquids with *the Ostwald method*
2. Studying the relationship between viscosity and concentration

Theory

Each fluid (gas or liquid) has a property known as viscosity which can be defined as the resistance exerted by one fluid layer against another fluid layer. In laminar flow, the fluid in the pipe is thought to consist of layers of molecules that move on top of each other at different speeds. The speed profile of these various layers is in the form of a parabolic with the highest speed found in the layer in the middle of the pipe.



Speed profile on the flow of the laminar

Notice a layer at a distance *r* from the axis of the pipe moving at a certain speed *C*. The force *f* required to maintain the dC velocity difference between this layer and *the dr* layer above it is expressed as:

$$f = \eta A \frac{dC}{dr} \qquad (2.1)$$

where *A* is the cross-sectional area of the pipe and η is the viscosity coefficient. Based on equation (2.1) above, the viscosity coefficient unit in SI is $Nm^{-2}sec$, while in the cgs unit it is dyne $^{cm-1}$ or poise. The opposite of viscosity is called fluidity, $\Phi=1/\eta$, which is a measure of the ease with which a fluid flows.

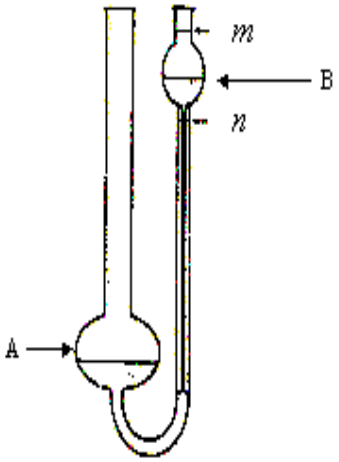
One way to determine the viscosity of a liquid is the capillary method from *Poiseuille*. In this method measured time, *t*, which is required for a volume of fluid *V*, flows through the capillaries under the influence of a fixed driving pressure. In

this case for liquids flowing with laminar flow, *the Poiseuille* equation is expressed as:

$$\eta = \frac{\pi R^4 pt}{8VL} \tag{2.2}$$

with R and L respectively being the radius and length of the capillaries. The *Ostwald* method is a variation of *the Poiseuille method*. The principle of this method can be learned from the following description:

A certain amount of liquid is put into A, then by sucking or blowing the liquid is brought to B until it passes the *m* line. Furthermore the liquid is allowed to flow freely and the time it takes to flow from the *m* to *n line* is measured. In the process of flowing through the capillary pipe the driving pressure is not fixed and at all times equal to ρgh, where h is the difference in the surface height of the liquid on the two arm of the tool, g is the acceleration of gravity, and ρ is the density of the mass of the liquid. Because in this method the flow of the liquid from *m* to *n is always taken into account* and uses the same viscometer, the viscosity of the liquid can be determined by comparing the measurement results of the time (t) and ρ of a particular liquid against *the t0* and ρ_0 of the comparator liquid whose viscosity has been known at the measurement temperature. The viscosity ratio of the two liquids can be stated as follows:



$$\frac{\eta}{\eta_0} = \frac{\rho\ t}{\rho_0\ t_0}$$
$$\eta = \eta_0 \frac{\rho\ t}{\rho\ t_0} \tag{2.3}$$

From equation (2.3) above, the viscosity of the liquid can be calculated by referring to the viscosity of the comparator liquid.

Tools and Materials

Tools: Ostwald *viscometer*, *stopwatch*, 25 mL measuring piper, and picnometer.

Material : Liquids with a specified viscosity (Glycerol): 5%, 10%, 15%, 20%, 25%, and 30%, and aqueducts.

How It Works

1. Use a clean viscometer
2. Place the viscometer in the thermostat in a vertical position
3. Insert the sample liquid into A so that when the liquid is brought to B, half of it is still left.
4. By sucking or blowing (through a piece of rubber hose) bring the liquid to B until slightly above the *m* line, then allow the liquid to flow freely, recording the time it takes for the liquid to flow from the *m* line to *the n* line. Do this work 3 times.
5. Perform procedures 3 – 4 for glycerol solutions of different concentrations, as well as the sample given by your assistant.
6. Determine the density of the liquid mass at the experimental temperature with a picnometer.
7. Do work 1 – 3 for the comparator fluid (aquaades), using the same picnometer.
8. Make a graph between viscosity versus concentration, what do you conclude. Determine the concentration of Glycerol x% given by your assistant.

Observation Table

Solution/solvent	Flow time (seconds)			Average flow time (seconds)
	T1	T2	T3	
Aquatics				
Glycerol 5%				
Glycerol 10%				
Glycerol 15%				
Glycerol 20%				
Glycerol 25%				
Glycerol 30%				
Glycerol x%				

Data Calculation and Analysis

1. Calculate the viscosity of each glycerol solution using an equation (2.3).
2. Determine the concentration of Glycerol x% from the graph by means of intrapolation.

EXPERIMENT 3

ISOTHERMAL ADSORPTION SOLUTION

Purpose

Quantitatively understand the adsorption properties of solutes of a solution on the surface of an adsorbent.

Theory

Adsorption is the event of absorption of a substance (gaseous or liquid) on the surface of an adsorbent. In the industry, the application of this adsorption principle is widely found, for example, impurities and colored materials contained in sugar and other organic products can be cleaned using adsorbents such as charcoal and other adsorbents. Gas masks contain adsorbents that function to absorb toxic gases that are harmful to humans.

Adsorption occurs on the surface of solids as a result of valence or other attractive forces of atoms or molecules on the surface of solids. When an atom or molecule is examined in a solid, it receives the force of attraction between atoms or molecules on the surface of the solid. The attraction experienced is not the same in all directions, so as compensation these atoms or molecules are adoptive to the adsorbate.

In general, adsorption is classified into two types, namely physical adsorption and chemical adsorption. In physical adsorption, the force that causes the adsorption is the same as the force that causes the condensation of a gas to form a liquid. The heat released is relatively small and this adsorption is reversible. Physical adsorption can form an adsorbate layer with the thickness of several molecular layers. Chemical adsorption only consists of one layer.

For adsorbents with a large surface, the adsorption is also getting bigger. The greater the concentration of solutes, the more solutes are adsorbed. The adsorption properties on the surface of solids are selective, meaning that in a mixture of various substances, only stu components are adsorbed by certain solids. If an adsorbent is allowed to come into contact with the solution, the amount of

adsorbent substances will gradually increase until a state of equilibrium is reached (Moore, 1974).

Several mathematical equations have been developed to study adsorption data. Two commonly used equations to study the adsorption of solutions in adsorbents are the Freudlich and Langmuir equations. In the Freundlich isotem equation, the effect of the concentration of the solution on adsorption can be expressed as follows:

$$\frac{x}{m} = k\,C^{n} \tag{3.1}$$

where x is the weight of the adsorbate, m is the weight of the adsorbent, C is the concentration of the adsorbate at equilibrium (the concentration of the adsorbate present in the solution), k and n are the empirical determinations. If the above equation is written in logarithmic form, a straight-line equation will be obtained, namely:

$$\log\frac{x}{m} = \log k + n\log C \tag{3.2}$$

From equation (3.2) we can determine *the k* of the intercept and the value *n* of the gradient (*slope).* The setting n is an indicator of the amount of energy and the variety of energy associated with the adsorption process, while *k* indicates the absorption capacity. The higher *the k value*, the greater the affinity of the adsorbent to the adsorbate.

Langmuir adsorption illustrates that on the adsorbent surface there are a number of active sites that are proportional to the surface area of the adsorbent. At each active site, only one atom or molecule is absorbed. The interaction between the adsorbed molecules at the adsorption result layer is negligible. This theory assumes that the bonds that occur do not depend on the bonds that have been formed at the active sites that are nearby.

According to the Langmuir isotrm equation, the effect of solution concentration on adsorption can be written as follows:

$$\frac{x}{m} = \frac{\alpha C}{(1+\beta C)} \text{ atau } \frac{C}{x/m} = \frac{1}{\alpha} + \frac{\beta}{\alpha}C \tag{3.3}$$

If adsorption involves a single absorption process (monolayer), then the curve $C/(x/m)$ versus C will be a straight line. From these curves, the values of □ and □ can be determined from their *slopes* and intercepts.

Tools and Materials

Tools: Analytical scales, Erlenmeyers, burettes, measuring flasks, measuring pipettes, and funnels.

Material: 1.0 M acetic acid solution, 0.5 M NaOH standard solution, activated carbon/activated clay, pp indicator, filter paper, and aquades.

Procedure

1. From a solution of acetic acid of 1.0 M, 5 samples of acetic solution were made with a successive concentration of 0.8; 0,6; 0,4; 0,2; and 0.1 M, each as much as 50 mL. To find out the true concentration of each solution, each of these acetic acid solutions needs to be titrated again with a standard NaOH solution.
2. For each acetic acid solution, 10 mL was taken and then titrated with NaOH 0.5 M with the pp indicator.
3. A mixture of 25 mL of acetic acid (1) is taken from each solution then put into the erlenmeyer, 1.0 g of activated carbon is added each then stirred with a magnetic stirrer for a few moments, then covered with filter paper and left for 30 minutes.
4. The above mixtures are then filtered. It is taken 10 mL then titrated with a standard solution of NaOH 0.5 M to determine the concentration of residual acetic acid present in the solution.

Observations

Adsorbent weight (m) : ..... g

Concentration CH_3COOH early (M)	Volume CH_3COOH titrated (mL)	Required NaOH volume (mL)		Average NaOH volume required (mL)
		Titration 1	Titration 2	
0,8				
0,6				
0,4				
0,2				

0,1				

Data Calculation and Analysis

1. Calculate the final concentration of CH_3COOH at eachinitial concentration.
2. Calculate the initial and final CH_3COOH weights.
3. Calculate the weight of the adsorbed CH_3COOH
4. Complete the following table based on the results of your calculations.

Concentration CH_3COOH early, M	Concentration CH_3COOH end, M	Weight CH_3COOH early, mg	Weight CH_3COOH end, mg	Adsorbate weight (x), mg	x/m	Log(x/m)
0,8						
0,6						
0,4						
0,2						
0,1						

5. Graph the relationship between Log(x/m) and C(final CH_3COOH concentration)
6. Specify *the n* and *k* of the graph you created.

EXPERIMENT 4

ACETONE IONIZATION REACTION SPEED

Meaning

Studying the reaction rate of acetone iodition in acid-catalyzed water solution

Purpose

Determining the law of reaction velocity of acetone iodine in acid-catalyzed water solutions

Theory

Reaction between acetone and iodine in a water solution,

$$CH_3\text{-}CO\text{-}CH_3 + I_2 \rightarrow CH_3\text{-}CO\text{-}CH_2I \quad (4.1)$$

runs slowly without a catalyst. In an acidic atmosphere this reaction takes place rapidly,

$$CH_3\text{-}CO\text{-}CH_3 + I_2 + H+ \rightarrow CH_3\text{-}CO\text{-}CH_2I + HI \quad (4.2)$$

and its reaction rate law can be expressed as:

$$\frac{d[I_2]}{dt} = k[aseton]^a[I_2]^b[H^+]^c \quad (4.3)$$

By using excessive amounts of acetone and acid, the above equation (4.1) can be changed to:

$$\frac{d[I_2]}{dt} = k[I_2]^b \quad (4.4)$$

with k'=k $[acetone]^a[H^+]^c$.

The reaction (4.2) can be monitored by determining the concentration of I_2 as a function of time. From this data, *a value of b can be determined*, which is the order of reaction to yod. The order of reaction to acetone (*a*) and acid (*c*) can be determined by changing the initial concentration of the two substances.

Tools and Materials

<u>*Tools*</u> : Erlenmeyer 300 mL, measuring pipette (5, 10 and 25 mL), measuring cup 10mL, burette 50 mL, measuring flask 200 mL, thermometer, and stopwatch.

Ingredients : Acetone, iodine solution (0.05 M in 10% KI solution), 0.01 M sodium thiosulfate solution, 1 M sulfuric acid solution, 10% sodium acetate solution and 1% amylum solution.

How It Works

A. - Put 25 mL of acetone and 10 mL of sulfuric acid solution in a measuring flask and dilute with acups to 200 mL.

- Transfer this solution to a 300 mL erlenmeyer (covered) and let it come to room temperature.
- Pipette 25 mL of iod into the above solution, shake vigorously, while the stopwatch is running.
- As soon as it starts to react, take 25 mL of solution and put it in an erlenmeyer flask containing 10 mL of sodium thiosulfate (to shut down the reaction) and then titrated with a thiosulfate solution with an amylum solution as an indicator (10 mL).
- The next footage was taken 4 minutes apart until the reaction mixture became colorless.

B. Repeat procedure A by taking 10 mL of acetone, snippets taken every 10 minutes.

C. Repeat procedure A by taking 5 mL of sulfuric acid solution, snippets taken every 10 minutes.

Observation Table

Procedure A					
Vol. Acetone (mL)	Vol. HCl (mL)	Vol. I2 (mL)	Time (minutes)	Vol. Titrated solution (mL)	Vol. Nattiosulfate (mL)
25	10	25	0		
			4		
			8		
			12		
			16		
			20		
			24		
			28		
Procedure B					

10	10	25	0		
			10		
			20		
			30		
			40		
			50		
			60		
			70		
Procedure C					
25	5	25	0		
			10		
			20		
			30		
			40		
			50		
			60		
			70		

Data Calculation and Analysis

1. From the data obtained in experiment A, calculate the concentration I_2 as a function of time, then graph the relationship between $[I_2]$ and time (t). From the graph you have created, determine the order of reaction to I_2 and the speed setting k'.
2. From the data obtained in experiment B, determine the speed setting k' and by paying attention to the results obtained in procedure A determine the order of reaction to acetone.
3. From the data obtained in experiment C, determine the speed setting k' and by paying attention to the results obtained in procedure A determine the order of reaction to H^+.

EXPERIMENT 5

EFFECT OF TEMPERATURE ON REACTION RATE

Meaning

Knowing the effect of temperature on the reaction rate.

Purpose

1. Studying the effect of temperature on the reaction rate of sulfur colloidal deposition.
2. Calculate activation energy using the Arrhenius equation.

Theory

One of the factors that affect the rate of a chemical reaction is temperature. The relationship between temperature and the reaction rate (*k) is* expressed according to the Arrhenius equation, namely:

$$k = Ae^{-E_a/RT} \quad (5.1)$$

or

$$\ln k = lnA - \frac{E_a}{RT} \quad (5.2)$$

with A being a preexponential constant or an Arrhenius constant, *Eais the* activation energy. If a graph is made of the relationship between 1/T and ln k, a straight line will be obtained with the tangent α/slope = *Ea*.

In this experiment, what was observed was the reaction of colloidal sulfur deposition formed when thiosulfate was reacted with acid. What was measured in this experiment was the time it took for the colloidal sulfur to reach a certain intensity. The sulfur cementation reaction can be written as follows:

$$S_2O_3^{2-}(aq) + 2H^+(aq) \rightarrow S(s) + SO_2(g) + H_2O(l) \quad (5.3)$$

By measuring time, the reaction rate of colloidal sulfur deposition in equation (5.3) can be calculated because the rate or rate constant is inversely proportional to time.

Tools and Materials

Tools : measuring cup, stopwatch, erlenmeyer, thermometer, water bath, volume pipette.

Material : $Na_2S_2O_4$ and HCl

Procedure

1. Put 10 mL of 0.5 M Potasium-thiosulfate solution in a measuring cup, then dilute until it reaches 50 mL by volume
2. Take 2 mL of HCL 1 M, put it in the test tube, place the measuring cup and the test tube in a water bath with a temperature of ± $30^{oC.}$ Let the two solutions sit for a while, until they reach equilibrium temperature. Measure the temperature using a thermometer and take notes.
3. Add the acid to the thiosulfate solution, and at the same time turn off the stopwatch. The solution is stirred and then place the measuring cup on top of the black cross. Record the time it takes until the cross is no longer visible when viewed from above.
4. Repeat the steps above for various temperatures up to 60^{oC} (do it for 4 different temperatures).

Observation Data

Temperature (^{oC})	Time (seconds)

Data Calculation and Analysis

1. The reaction rate is defined as 1/time. Complete the table of calculation results.

$T(^{oC})$	T(K)	Time, t (seconds)	1/time (1/second)	$1/T$ ($^{K-1}$)	*ln* (1/time)

2. Create a reaction rate curve as a function of temperature (^{oC}).
3. Create a curve *ln* reaction rate as a function of 1/T (1/K).
4. Comment on the curve shape obtained.

EXPERIMENT 6

KINETICS OF ADSORPTION

Intent of the Experiment

Studying the adsorption kinetics of solids in solution

Purpose of the Experiment

Studying the adsorption kinetics of activated carbon in acetic acid solutions

Theory

One of the important properties of the surface of a substance is adsorption. Adsorption is used to state that there are other substances that are absorbed into the substance, for example activated carbon can absorb acetic acid molecules in the solution.

Activated carbon is usually made by burning coconut shells or wood with a limited supply of air (oxygen). Each adsorbent particle is surrounded by molecules that are absorbed due to the tensile interaction. The size of adsorption is influenced by: (1) the type of adsorbent, (2) the type of substance adsorbent, (3) the concentration of the adsorbent and the adsorpted substance, (4) the surface area, (5) the temperature and (6) the pressure.

Generally, adsorbents are specific, that is, they only absorb certain substances. The adsorption rate of activated charcoal against the acetic acid molecule in its solution at a fixed pressure and temperature depends on the concentration of acetic acid. Adsorption and desorption (discharge) are equilibrium. The adsorption speed before equilibrium is reached can be learned from the equation:

$$-\frac{dC}{dt} = k\,C^{n} \tag{6.1}$$

dC/dt is the speed of adsorption, *k* is the constant of adsorption, *C* is the concentration, and *n* is an order of adsorption velocity.

In adsorption kinetics, the relationship between species concentration and time change can be studied. The adsorption kinetics of activated carbon against acetic acid can be determined by measuring changes in the concentration of acetic

acid as a function of time and analyzing it by price *analysis k* (adsorption constant) or by graph.

Analysis of adsorption kinetics

a. Order One

$$-\frac{dC}{dt} = k\,C \tag{6.2}$$

$$\int_{C_0}^{C} -dC = k\int_{0}^{t} dt \tag{6.3}$$

$$\ln \frac{C}{C_0} = -\,kt \tag{6.4}$$

$$\ln\, C = \ln\, C_0 - kt \tag{6.5}$$

Graphs ln *C* Opponent *t* is a straight line with *Slope* = *k* and *Intersept* = ln C_0

b. Order Two

$$\int_{C_0}^{C} -\frac{dC}{C^2} = k\int_{0}^{t} dt \tag{6.6}$$

$$\frac{1}{C} - \frac{1}{C_0} = kt \qquad \text{atau} \qquad \frac{1}{C} = \frac{1}{C_0} + kt \tag{6.7}$$

Graphics $\frac{1}{\mathrm{C}}$ versus *t* is a straight line with *Slope* = *k* and *Intersept* = $\frac{1}{C_0}$

c. Order Three

$$\int_{C_0}^{C} -\frac{dC}{C^3} = k\int_{0}^{t} dt \tag{6.8}$$

$$\frac{1}{2\,C} - \frac{1}{2\,C_0} = kt \qquad \text{atau} \qquad \frac{1}{C^2} = \frac{1}{C_0^2} + 2kt \tag{6.9}$$

Graph $\frac{1}{C^2}$ versus *t* is a straight line with *slope* = *2k* and *intersept* = $\frac{1}{C_0^2}$

Tools and materials

Tools : Burette 50 mL, erlenmeyer 250 mL, glass funnel, measuring cup 25 mL, *Material* : CH_3COOH solution 1 N and 0.5 N, standard solution NaOH 0.5 N, phenolphthalein (PP) indicator, and filter paper.

How It Works

1. Preparing 5 pieces of erlenmeyer 200 mL
2. Five erlenmeyers were filled with a solution of 0.5 N acetic acid of 25 mL each
3. Weigh 2 grams of activated carbon 5 times.
4. Each 2 grams of activated carbon is put into an erlenmeyer containing an acetic acid solution, then whisked for 1 minute, after which it is left at time intervals of 15 minutes, 30 minutes, 45 minutes, 60 minutes and 75 minutes, respectively.
5. Filtered, each filtrate was measured in volume and then titrated with NaOH 0.5 N.

Observations

Contact time (minutes)	Titrated CH_3COOH volume 0.5 N (mL)	Required NaOH volume 0.5 N (mL)
15		
30		
45		
60		

Data Calculation and Analysis

1. Calculate the final concentration of CH_3COOH at each contact time.
2. Create a graph using equations (6.5), (6.7), and (6.9) to determine the order (n) and the reaction rate constant (k). Determine the equation that matches the observation data or calculations in your experiment.

EXPERIMENT 7

DETERMINATION OF *POLYMER* $\overline{M_V}$ USING THE VISCOMETRY METHOD

Purpose

Determining *the* $\overline{M_V}$ of a polymer

Theory

The viscosity of a dilute polymer solution is a function of its molecular mass and the dimensions of the solute. The ratio of the viscosity of a dilute solution (η) to the viscosity of a pure solution (η_0) is expressed as the relative viscosity (η_r) and is a comparison between the flow time of the solution (t) and the solvent (t_0) measured under the same experimental conditions.

$$\eta_r = \frac{\eta}{\eta_0} = \frac{t}{t_0} \tag{7.1}$$

If the concentration (C) of the solution is close to zero or the solution is very dilute, then the price of η_r will be close to one. In this state the viscosity of the solution is expressed as

Specific viscosity (η_{SP}):

$$\eta_{sp} = \eta_r - 1 = \frac{\eta - \eta}{\eta_0} \tag{7.2}$$

This reality illustrates the increase in the viscosity of solvents due to the presence of solutes. Since η_{sp} is a concentration function, it is defined as reduced viscosity (η_{red}) which is expressed as:

$$\eta_{red} = \frac{\eta_{sp}}{C} \tag{7.3}$$

By knowing η then the molecular mass and macromolecular dimensions can be calculated. When a polymer is dissolved, the size (dimensions) of the macromolecular chain will depend on its interaction with the solvent. In a good solvent, the molecular chain will open, as a result of which the solvent interacts easily. Whereas in an ugly solvent, macromolecules tend to retain their dimensions.

According to *Flory* and *Fox's theory*, the intrinsic viscosity and dimensions of polymer chains are connected by equations:

$$[\eta] = \phi\alpha^3\beta^3 \frac{M^{\frac{1}{2}}}{M_0^{\frac{3}{2}}} \qquad (7.4)$$

with ϕ is the universal constant *of Flory* (2.86 x 1023)

Solvents can interact with polymers. This situation occurs when $\alpha > 1$. Conversely, if $\alpha > 1$ there is no interaction between the polymer and the solvent. At $\alpha = 1$ is called the *critical point* of polymer solubility. In this state the temperature is called the temperature ϕ and the solvent is called *the solvent* ϕ. Since M_0 and β are constants of a given polymer, equation (7.4) above can be written as:

$$[\eta] = K M^{\frac{1}{2}} \alpha^3 \text{ dan} \qquad [\eta]_\phi = K M^{\frac{1}{2}} \qquad (7.5)$$

since α is a function *of M*, then

$$[\eta] = K M^a \qquad (7.6)$$

The equation (7.6) above is known as the *Mark-Houwink equation*. By knowing the value of $[\eta]$ and entering the prices K and a for a particular polymer-solvent system, then the price *of* $\overline{M_V}$ polymer can be calculated.

Tools and Materials

Tools : Viscometer, water bath at $30^{oC,}$ 100 mL measuring flask, and *stopwatch*.

Material : Polyvinyl alcohol (PVA), acetone, ether, and aquades.

How It Works

1. Weigh 1.0 grams of PVA, put it in a 100 mL measuring flask and dissolve it little by little with aquades, strain as needed. The concentration of this solution is *C*.
2. Put 15 mL of aquaades into the viscometer. Measure the flow time several times until a constant value is obtained.
3. Rinse the newly used viscometer with a solution that will measure the flow time (for thorough workmanship it is recommended to wash and dry the viscometer after use)

4. Measure the flow time of each PVA solution with a concentration of 1*C*, 3/4 *C*, 1/2 *C*, and, 3/8 *C*. (for each measurement put 15 mL of solution into the viscometer and the measurement starts with the thinnest solution).
5. Calculate the viscosity of each solution at a temperature of $30^{oC.}$
6. Create a $\frac{\eta_{sp}}{C}$ chart as a function *C*. [η] expressed in mL/g.
7. From the *Mark-Houwink equation*, determine *the* $\overline{M_V}$ of the polymer solution, the price *K* = 15.6 x 10-5 and a = 0.76.

Observations

Solution/solvent	Flow time (det me)			Average flow time (seconds)
	T1	T2	T3	
Aquatics				
PVC 1C				
PVA 3/4 C				
PVA 1/2 C				
PVA 3/8 C				

Data Calculation and Analysis

1. Calculate the relative viscosity (η_{rel}), specific viscosity (η_{spe}), and reduction viscosity (η_{red}) each PVA solution uses equations (7.1) to (7.3)
2. Define [η] from the graph by extrapolating.
3. Calculate the average molecular weight of PVA using the equation (7.6)

EXPERIMENT 8

ACTIVATED CLAY AND ACTIVATED CHARCOAL AS ADSORBENTS

Purpose

Studying the adsorption process using activated clay and activated charcoal

Theory

Clay.

Clay is a natural material in the form of crystals consisting of alumina silica particles that are less than 2□ in size. The basic structure of clay minerals consists of a silicate layer composed of silicon-oxygen tetrahedral sheets and aluminum-oxygen-hydroxy sheets. The four oxygen atoms on the tetrahedral sheet bond with four Al-Mg atoms on the octahedral sheet forming two layers. The structure structure of monmorilonite clay is in the form of a 2:1 ratio (composed of two tetrahedral sheets and one octahedral sheet) with a molecular formula of $Al_{4Si8}(OH)_{4O20}$. The unit cells in the attached monmorilonite are linked by van der Waals bonds and possible cations to neutralize the lack of charge in the clay structure. These bonds are not strong enough that the space between the layers can expand or separate if water molecules or other polar molecules enter or be absorbed.

Activated Charcoal.

Activated carbon is activated carbon, so it has high absorbency against adsorbate such as dyes, odorous substances, and toxic substances. The high absorption is due to a very large surface area of activated carbon, which is 300-2500 m^2/g. Activated carbon is amorphous, black, insoluble in water, acids, bases, and organic solvents. Some of the basic materials that have been used to make charcoal are wood, coconut shells, pecan shells, cashew husks, sago dregs, and other materials containing cellulose and lignin. Carbon activation can be done by physical means, namely heat treatment (thermal) or chemical, namely the addition of chemical substances (activators).

Materials and Tools

Tools: 20D Spectronic, magnetic stirrer, glass tools Material: Clay, activated carbon, dyestuffs, aquades.

How It Works

Adsorption of dye :

a. Add 10 mg of adsorbents to 25 mL of 10 mg/L dye solution.
b. Stir the mixture for 1 hour.
c. Separate the filtrate and adsorbent solids after stirring is complete by filtering.
d. Measure the absorbance of the filtrate (dye after adsorption) at the maximum wavelength of the dye solution.

Spectrometry determination of dye concentrations

a. Determine the maximum wavelength of the dye solution using a spectrophotometer.
b. Make a calibration curve for the standard solution of dyestuffs: 0, 2, 5, 10, and 15 mg/L.
c. Determine the concentration of the dye after adsorption using a standard solution calibration curve.

Observations

Volume of dye : ..... mL

Before adsorption		After adsorption		Weight of adsorbed dye (mg)
Concentration of dyestuff, ppm	Absorbance	Concentration of dyestuff, ppm	Absorbance	
0				
2				
5				
10				
20				

Data Calculation and Analysis

1. Calculate the weight of the dye before and after the adsorption process.
2. Calculate the weight of the dye adsorbed by the clay or activated carbon.
3. Calculate the percentage (%) of the dye that is adsorbent.

EXPERIMENT 9

DETERMINATION OF WEAK ACID IONIZATION STATUS BY CONDUCTTOMETRIC METHOD

Purpose

1. Measuring the conductivity of weak acid electrolytes
2. Determining the ionization setting of acetic acid by measuring conductivity

Theory

The equilibrium constant of weak acid dissociation HA can be written in simple form as:

$$HA(aq) \rightarrow H^+(aq) + A^-(aq)$$

$$K_a = \frac{[H]^+[A]^-}{[HA]} \qquad (9.1)$$

If the stoichiometric concentration (total) HA is equal to *c*, and the degree of dissociation, α then :

$$K_a = \frac{\alpha^2 c}{1-\alpha} \qquad (9.2)$$

One method to determine K_a is to measure the pH of the solution, from that measurement we get $[H^+] = \alpha c$, and then we can calculate K_a. However, this method is not very accurate because there is a large error in the calculation of $[H^+]$ in the logarithmic relationship between pH and $[H^+]$. The best method to determine K_a is by electrolyte conductivity.

For weak acids, the degree of ionization is small, for example a solution of 0.1 M, the concentration of ions is very small. The conductivity of the molar, Λ, depends on the concentration, but for very low concentrations of ions we can assume that the conductivity of an individual ion is equal to its conductivity at infinite dilution. The molar conductivity at infinite dilution is the sum of the conductivity of individual ions at infinite dilution, and is called Λ_o. The Λ_o value for weak acids can be calculated directly from the Λ_o value of compounds containing the same ions. For example if the weak acid is acetic acid, HAc we can write:

$$\Lambda_o(HAc) = \Lambda_o(NaAc) + \Lambda_o(HCl) - \Lambda_o(NaCl) \quad (9.3)$$

The value of Λ_o at 25°C is presented in table 5.1. If we assume that the concentration of ions is small (meaning c is large), we can write:

$$\alpha = \frac{\Lambda}{\Lambda_o} \quad (9.4)$$

Equation (9.4) shows that the fraction of the ionized HA molecule must be equal to the observed conductivity divided by the conductivity if the HA is 100% ionized. Molar conductivity [HA] = *c* can be calculated from the measurement of conductivity type:

$$\Lambda = \frac{L}{c} \quad (9.5)$$

Thus, the measurement *of L* at the total concentration *of c* yields α of equations (9.4) and (9.5) then K of equations (9.2). Measurements of conductivity at different concentrations will show that α decreases with increasing concentration, but K_a constant.

If the value of Λ_o is unknown, the second method to determine α and Λ_o simultaneously is with the "Ostwald Law of Dilution" below. From the equation (9.2)

$$\frac{\alpha^2 c}{K_a} = 1 - \alpha \quad (9.6)$$

Substitution equation (5.4) and rearranged yields:

$$\Lambda = \Lambda_o - \frac{c\Lambda^2}{\Lambda_o K_a} \quad (9.7)$$

Equation (9.7) shows that the plot Λ to c Λ^2 will produce a straight line with the interception at $c = 0$ being equal to Λ_o and the *slope* of $1/K_a \Lambda_o$ thus, we can calculate Λ_o and Ka.

This method is very accurate for electrolyte solutions that are not ideal despite the very low ion concentration but can still be determined. In the measurement of K_a, we need to consider that the value of K_a increases slowly and systematically with

concentration. This is in accordance with the fact that K_a must be described in activities other than concentration.

$$K_a = \frac{a_{H^+}\, a_{A^-}}{a_{HA}} = \frac{\gamma_+ [H^+]\gamma_- [A^-]}{[HA]} \tag{9.8}$$

In equation (9.8) we have used $\square_{HA} = 1$. According to the definition:

$$\gamma_{\pm}^2 = \gamma_+ \gamma_- \tag{9.9}$$

then obtained:

$$K_a = \frac{\gamma_{\pm}^{\,2} [H^+][A^-]}{[HA]} \tag{9.10}$$

for low concentrations the average coefficient of ion activity according to *Debye-Huckel's law:*

$$\log \gamma_{\pm} = A z_+ z_- I^{1/2} \qquad I = \tfrac{1}{2} \Sigma c_i z_i^2 \tag{9.11}$$

I is called the ion force. At 25oC A = 0.509. For 1:1 electrolytes such as NaCl, HAc, and so on, $I = c$. Equations (9.9) and (9.10) show that if K_a varies with concentration, its value at infinite dilution can be obtained from plotting pK_a against *c1/2* and extrapolating it at $c = 0$.

Table 9.1. Molar conductivity limits, Λ_o (Sm2mol-1) some acids, bases and salts.

Compounds	**Λ_o x10-2**	**Compounds**	**Λ_o x10-2**
HCl	4,262	NaCl	1,266
HBr	4,281	KCl	1,499
NaOH	2,480	NaAc	0,910
KOH	2,720	Na_2SO_4	2,598
$Ca(OH)_2$	5,170	$MgSO_4$	2,657

How It Works

a. Solution manufacturing

Make a stock solution of 0.1 M acetic acid of 250 mL, HAc. The standardization of this solution by titration uses a standard solution of NaOH 0.1 M and phenolphthalein as indicators to determine the titration endpoint. With accurate

dilution of the stock solution, make a 100 mL HAc solution with concentrations of 0.025, 0.01, and 0.0025 M, respectively.

b. Transmittance measurement

The following are instructions for the Oakton conductometer:

Rinse the electrodes twice with aqueducts and measure their conductivity. Record the transmittance of the aquifers. Next, measure the conductivity of the acetic acid solution that has been made starting from the lowest concentration. Before measuring the conductivity of the next solution, it is recommended that the electrodes be rinsed with aqueducts and then dried with a tissue. Note the conductivity of each acetic acid solution. Make sure the transmittance value is stable, wait a few seconds. Do not leave the electrodes in a 0.1 M acetic acid solution for more than 5 minutes. Once the measurement is complete, rinse the electrodes several times with aqueducts and dry/wrap them with a tissue.

c. Calculations.

Remember that the Oaktonmeter calibration procedure is electronically set where the cell constant is 1.00 $S.cm^{-1}$ or 100 $S.m^{-1}$. So, if the conductivity of HAc solution is shown on the display as G(S), the conductivity of the solution type (*L*) is equal to Gx1.00 $S.cm^{-1}$ or G x 100 $S.m^{-1}$.

1. From equation (5) and the measurement value L, calculate Λ each solution including the stock solution. Don't forget the unit correction.
2. Using the value of Λ_o in table 1, calculate Λ_o(HAc) and α of each solution. Calculate K_a for each solution.
3. As an alternative to the calculation in number 2 above, calculate the value of $c\Lambda^2$ of each solution and make a graph Λ against $c\Lambda^2$. of the slope and intercept calculate the values Λ_o(HAc) and K_a.

d. Tools and Materials

- Oakton conductometer and conductivity cells
- Erlenmeyer 250 mL
- Buret 50 mL
- Pipette 25 mL

- Pumpkin measuring 100 and 250 mL
- Phenolphthalein solution
- Acetic acid
- Standard solution NaOH 0.1 M

e. Manufacture of standard alkaline solutions

1. Dissolve about 1 g of NaOH solids into 250 mL of aqueducts
2. Carefully weigh about 0.15 g of oxalic acid dihydrate in a 250 mL erlenmeyer
3. Fill the burette with NaOH solution and titrify it with oxalic acid to the end point using phenolphthalein as an indicator.
4. Repeat steps 3 and 4 and calculate the average NaOH concentration

Observations

HAc Solution Concentration (M)	Transmittance, *L* (S.m-1)
0,0250	
0,0100	
0,0025	
0,0010	

Data Calculation and Analysis

1. Create a table containing the concentration, conductivity type, molar conductivity, ionization degree and calculation value K_a for each solution, using the value Λ_o in Table 9.1. in the calculation. Calculate ΔGo in the process of dissociation of HAc.
2. Plot Λ against $c\Lambda^2$ and use the methods described in theory to obtain Λ_o and Ka. Compare the values with the Λ_o and Ka that have been obtained in the calculation above.

EXPERIMENT 10

DEGRADATION OF DYES USING METAL OXIDE CATALYSTS

Purpose

Understand the principle of photocatalysis degradation using semiconductor materials.

Introduction

Photocatalyst is defined as a combination of photochemical processes and catalysts, which are chemical transformation processes that use photons as an energy source and catalysts as a speed accelerator of transformation. The process is based on the dual ability of a semiconductor material (such as TiO_2, ZnO, Fe_2O_3, CdS, ZnS) to absorb photons and perform transformation reactions at the interface of the material simultaneously.

TiO_2 is a semiconductor material that has *a band-gap energy* of 3.2 eV. When a colloidal TiO_2 particle is photoexcited (λ < 380 nm) electrons, electrons and a positive charge are produced called *holes*, $h+$ according to the following reaction equation:

$$TiO_2 + h\nu \rightarrow e^- + h^+ \qquad (10.1)$$

Sequentially, electrons and *holes* allow reductive and oxidative reactions to take place at the interface of semiconductor materials. In water solvents, dissolved oxygen acts as an electron capturer, while water will react with *holes* to produce OH radicals as follows:

$$H_2O + h^+ \rightarrow \bullet OH + H^+ \qquad (10.2)$$

OH radicals have a fairly high reduction potential of ~2.8 V, so they are highly oxidative. This allows OH radicals to immediately react with organic or inorganic compounds that are in the water (photodegradation). The total degradation of these organic pollutants will result in CO_2, H_2O and minerals (perfect mineralization)

$$\text{Polutan} + O_2 \xrightarrow{TiO_2, h\nu} CO_2 + H_2O + \text{asam mineral.} \qquad (10.3)$$

Tools and Materials

Tools : UV-lit cabinet, 20D Spectronic, magnetic mixer and glassware.

Material: Metal oxide (semiconductor), dye (methyl orange).

How it works

Photocatalysis Studies

a. In a dark room, put 0.05 g of metal oxide in 50 mL of dye solution at each concentration and stir for 30 minutes.
b. Shine the solution with a UV lamp for 1 hour.
c. After irradiation, filter the photocatalyst solids using *Whatman filter paper*.
d. Measure the absorbance of methyl orange solution before and after photocatalysis at λ_{max} of the dye.

Determination of the concentration of dye

a. Determine the λ_{max} of the dye solution.
b. Make a calibration curve of the standard solution of the dye : 0, 5, 10, 15, 20 ppm.
c. Determine the concentration of the dye using the calibration curve method.

Observations

Before photocatalysis		After photocatalysis		Weight of degraded dye (mg)
Concentration of dyestuff, ppm	Absorbance	Concentration of dyestuff, ppm	Absorbance	
0				
5				
10				
15				
20				

Data Calculation and Analysis

1. Calculate the weight of the dye before and after the photocatalysis process.
2. Calculate the weight of the dye degraded by the metal oxide catalyst.
3. Calculate the percentage (%) of the dye degraded.