**Department of Chemical Engineering, Veer Surendra Sai University of Technology**

# Process Technology

# Lab Manual

**List of Experiments**

| Sl. No. | Name of the Experiment |
|---|---|
| 1 | To prepare soap and to determine the percentage yield of soap using a given oil sample |
| 2 | To determine the alkali content of a given soap sample |
| 3 | To determine the partition co-efficient of Iodine between an organic solvent and water |
| 4 | To determine the fat content in a given food stuff sample |
| 5 | To determine the ferrous iron concentration in Mohr's salt solution using $KMnO_4$ |
| 6 | To determine the dye concentration using UV- visible spectrophotometer |

# Experiment 1

**Aim:** To prepare soap and to determine the percentage yield of soap using a given oil sample

**Apparatus required:**

- Water heater bath
- Weighing balance
- Beaker: 250 cc
- Glass rod

**Chemical required:**

- Sunflower oil – 20 g
- About 3 cc of NaOH 25% (w/w)
- $Na_2CO_3$ – 4 g
- $Na_2SiO_3$ – 0.2 g
- NaCl – 0.4 g
- Water – 20 mL

**Procedure:**

1. Wash the beaker and dry it in hot air oven for 30 minutes
2. Take 20 g oil in the beaker
3. Fit the beaker in the insulating grip made up of thermocol
4. Add 6 mL of 25 % NaOH solution in beaker
5. Mix the contents thoroughly in the beaker with the help of a glass rod and keep it in a water bath at a temperature of 230-250 F with continuous stirring
6. Add water (20 mL) to keep the mixture in liquid state
7. Add $Na_2CO_3$ (4 g) to complete saponification reaction
8. Cool the mixture at room temperature.
9. Add about NaCl (0.2 g) to separate the foam layer after completion of the saponification reaction

10. Add $Na_2SiO_3$ (0.2 g) and mix the contents.
11. Separate the soap from the mixture.

**Calculation:**

- Wt. of sunflower oil = 20 g
- Wt. of $Na_2CO_3$ = 4 g
- Wt. of NaCl = 0.2 g
- Wt. of $Na_2SiO_3$ = 0.2 g
- Wt. of water = 20 g
- Total wt. oil mixture = 44.4 g
- After soap formation, total wt. = X g
- Wt. of container = Y g
- Wt. of soap formed = (X-Y) g
- % yield of soap = (X-Y)*100/44.4

**Result:**

The percentage yield of soap of the given oil sample was found to be ---------.

# Experiment 2

**Aim:** To determine the alkali content of a given soap sample

**Apparatus required**:

- Conical flask
- Beaker
- Separating funnel
- Glass rod
- Weighing balance
- Burette and Pipette

**Chemicals Required:**

- Diethyl ether
- NaCl (25 mL)
- $H_2SO_4$ (1N)
- NaOH (1N)
- Methyl orange indicator (4-6 drops)
- Distilled water

**Procedure:**

1. Weigh 5 g of soap sample accurately and transfer into a 250 mL beaker
2. Add 50 mL hot water to completely dissolve the soap
3. Add 4-6 drops of methyl orange indicator to the soap solution
4. Add 15 mL of 1N $H_2SO_4$ to the solution drop wise till the color changes from orange to pink
5. Add 20 mL of diethyl ether to the mixture using a test tube
6. Transfer the mixture into a separating funnel to separate the bottom layer and collect using a beaker
7. Add 25 mL of NaCl solution to the bottom layer and titrate with 1N NaOH solution

8. Record the initial burette reading
9. Add NaOH solution till the color of the solution changes from pink to orange
10. Note the final burette reading

**Tabulation**:

| SL. No. | Initial reading (IR) | Final reading (FR) | Difference |
|---|---|---|---|
| | | | |
| | | | |
| | | | |

**Calculation: (**For sodium soap**)**

Total alkali content in mass percentage = $[4.0*((V_1*N_1)-(V_2*N_2))]/M$

$V_1$ = volume of $H_2SO_4$ solution used in mL

$V_2$ = volume of NaOH solution used in mL

$N_1$ = Normality of $H_2SO_4$

$N_2$ = Normality of NaOH

M = Mass in grams of soap in g

**Tabulation**:

| Sl No | Initial reading (IR), mL | Final reading (FR), mL | Difference |
|---|---|---|---|
| | | | |
| | | | |
| | | | |

**Result:-**The alkali content of the given soap sample was found to be_____

# Experiment 3

**Aim:** To determine the partition co-efficient of Iodine between an organic solvent and water

**Apparatus required:**

1. Stopper glass bottle
2. Separating funnel
3. Beakers: 4 no.
4. Conical flask: 2 no.
5. Measuring cylinder: 1 no.
6. Burette
7. Stand

**Chemicals required:**

1. Saturated solution of $I_2$ in $CCl_4$
2. $Na_2S_2O_3$ solution (N/10 and N/100)
3. Starch solution
4. Potassium Iodide (KI)

**Procedure (A):**

1. Take four glass stopper bottles of about 150 mL capacity
2. Wash these bottles with water and allow drying. Name the bottles as No. 1, 2, 3 and 4
3. Add 50 mL of distilled water in each of these bottles and add the saturated solution of iodine in benzene and benzene as per Table 1

**Observation Table 1**

| Bottle no. | Volume of Iodine in mL | Volume of Benzene in mL |
|---|---|---|
| 1 | 10 | 40 |
| 2 | 20 | 30 |
| 3 | 30 | 20 |

| | | |
|---|---|---|
| 4 | 40 | 10 |

**Procedure (B):**

1. Shake each bottle (closed) vigorously for 20 minutes (shaking of each bottle is necessary for getting accurate results)
2. Keep these bottles undistributed for some time and allow to separate the two layers
3. Pour the contents of these bottles separately into a separating funnel and collect benzene and water layers of each bottle in separately numbered beaker
4. Pipette out exactly 5mL of benzene layer from the separated layers of bottle no. 1 into a conical flask
5. Add 1 g of potassium iodide and 5 drops of the starch solution
6. Do titration for the solution against (N/10) $Na_2S_2O_3$ from the burette
7. Continue the titration, till the blue color get disappear
8. Repeat three times to get concordant readings
9. Repeat this process with benzene layers of other bottles also in the same manner
10. Pipette out 10 mL of aqueous layer of bottle no. 1 into a conical flask
11. Add 1 g of potassium iodide and 5 drops of the starch solution
12. Titrate this solution against (M/100) $Na_2S_2O_3$ from the burette
13. Continue the titration till the blue color get disappear
14. Repeat three times to get concordant readings
15. Repeat this process with aqueous layers of other bottles in the same way
16. Note the room temperature using thermometer

**Observation Table 2**

| Bottle number | Aqueous layers ($C_1$) | | | | Benzene layers ($C_2$) | | | |
|---|---|---|---|---|---|---|---|---|
| | Burette Reading in mL | | | | Burette Reading in mL | | | |
| | I.B.R | F.B.R | Volume used | Mean value | I.B.R | F.B.R | Volume used | Mean value |

| | | | | | | | | |
|---|---|---|---|---|---|---|---|---|
| 1. | 1.<br>2.<br>3. | | 10 | $V_1$ | | | 5 | $V_5$ |
| 2. | 1.<br>2.<br>3. | | 10 | $V_2$ | | | 5 | $V_6$ |
| 3. | 1.<br>2.<br>3. | | 10 | $V_3$ | | | 5 | $V_7$ |
| 4. | 1.<br>2.<br>3. | | 10 | $V_4$ | | | 5 | $V_8$ |

**A) For Aqueous layer**

Volume of aqueous layer taken for titration = 10 mL

Mean value of (N/100) $Na_2S_2O_3$ solution = $V_1$

10 mL of $N_1$ iodine = $V_1$ mL of (N/10) $Na_2S_2O_3$

or $N_1 = V_1 \times (1/100) \times (1/10) = (V_1/1000)$

Thus, the concentration of $I_2$ in water layer = $127 \times (V_1/1000)$ gram equivalent / liter

(Since equivalent of $I_2 = 127$) = $C_1$

**B) For Benzene layer**

Volume of benzene layer taken for titration = 5 mL

Mean value of (N/10) $Na_2S_2O_3$ solution = $V_5$ mL

5mL of $N_2$ iodine = $V_5$ mL of (N/10) $Na_2S_2O_3$

or $N_2 = V_5 \times (1/10) \times (1/5) = (V_5/50)$

Thus the concentration of $I_2$ in benzene layer = $127 \times (V_5/50)$ gram equivalent/liter= $C_2$

Partition co-efficient (K) = $C_1/ C_2$

$= [127 \times (V_1/1000)]/ (127 \times V_5/50)$

$= 127 \times (V_1/1000) \times (50/127 \times V_5)$

$= (V_1 \times 50)/ (V5 \times 1000) = (V1)/ (V5 \times 20)$ ---------- (I)

The value of the partition coefficient (K) is calculated in the same way for other bottles by using the simplified formulae as given under.

For bottle no. 2, $K= C_1/ C_2= (V_2)/ (V_6 \times 20)$

For bottle no. 3, $K= C_1/ C_2= (V_3)/ (V_7 \times 20)$

For bottle no. 4, $K= C_1/ C_2= (V_4)/ (V_8 \times 20)$

The mean value of K obtained after using the relationships (I), (II), (III) and (IV) as given above gives the value of the partition coefficient (K) of iodine between water and benzene.

**Conclusion:**

The partition co-efficient of iodine between benzene and water was found to be ____ at ___ $^oC$.

# Experiment 4

**Aim:** To determine the fat content of a given food stuff sample

**Apparatus required:**

- Soxhlet apparatus
- Weighing balance
- Thimble

**Chemicals required:**

- n-hexane
- Petroleum ether
- Ethanol
- Food stuff

**Theory:**

The fat or extractive content in food stuff can be extracted completely by using solvents such as n-hexane, ethanol and petroleum ether

**Description of Soxhlet apparatus:**

The apparatus is consisting of three parts such as a round bottom flask, an extraction section and a condensing section. A thimble filled with sample is placed in the extraction section. The three parts of the apparatus are connected and placed over heating mental at a constant temperature. Cold water is circulated in the condenser (top section of the apparatus) to condense the evaporated solvent. The condensed solvents falls drop wise on the thimble where frequent contact between the feed and the solvent occurs and the extractives get solubilized in the solvent. The solution gets transfer to the round bottom flask after reaching a particular level above the thimble. This process should be continued for 3 to 4 h to extract maximum soluble.

**Procedure:**

1. Record the empty weight of the thimble ($w_o$)
2. Ground the given peanuts (12 g) into very small size, place inside the thimble and note the weight of the filled thimble accurately ($w_1$)
3. Put the sample filled thimble in the extraction section of the Soxhlet apparatus
4. Fill the round bottom flask with the solvent (400 mL) and connect all the section of the apparatus above a heating mental
5. Start the heating process at a constant temperature (boiling point of the solvent) and keep circulating cold water in the condenser section
6. Continue the process of extraction for 3 h and observed the change in color of the solution in the thimble section.
7. Stop the process of heating when no change in the color is observed.
8. Remove the thimble very carefully so that the sample remaining in the thimble will not fall outside and then dry and weigh it accurately.
9. Note the final weight of the thimble is $w_2$
10. Similarly, follow all the steps by taking different solvents to determine the removal efficiency

**Calculations:**

% fat/extractives in the given sample = $((w_1-w_2)/ (w_1-w_0)) \times 100$

**Conclusion:**

The removal efficiency of fats/extractive from _____ sample was found to be ____ % using ____ solvent.

# Experiment 5

**Aim:** To determine the ferrous iron concentration in Mohr's salt solution using $KMnO_4$

**Chemicals required:**

- Standard oxalic acid solution
- Sulphuric acid
- Potassium permanganate solution
- Ferrous ammonium sulphate

**Apparatus required:**

- Burette
- Pipette (10 mL)
- Beaker
- Conical flask
- Hot water bath
- Thermometer

**Procedure:**

Standardization of potassium permanganate

1. Rinse burette with water and with $KMnO_4$ solution
2. Fill the burette with $KMnO_4$ solution
3. Pipette out 10 mL of standard sodium oxalate solution into a conical flask
4. Add 10 mL of 2N $H_2SO_4$ solution to it
5. Heat this solution at 70 $^oC$ using a hot water bath for 15 minutes
6. Titrate the solution against $KMnO_4$ from the burette till the solution becomes permanent pink color
7. Repeat three times to get concordant readings

**Observation Table 1**

| No. of obs. | Volume of oxalic acid + volume of $H_2SO_4$ in mL | Burette reading in mL | | | Remarks |
|---|---|---|---|---|---|
| | | IBR | FBR | Difference | |
| 1 | 10+10 | | | | |
| 2 | 10+10 | | | | |
| 3 | 10+10 | | | | |
| 4 | 10+10 | | | | |

**Calculation:**

Vol. of $KMnO_4$ consumed ($V_1$) = X mL

Vol. of oxalic acid ($V_2$) = 10 mL

Strength of $KMnO_4$ ($S_1$) =

Strength of oxalic acid ($S_2$) =

$S_1=V_2*S_2/V_1$

**Estimation of iron:**

1. Pipette out 10 mL of Mohr's salt [$FeSO_4(NH_4)_2SO_46H_2O$] solution into a conical flask
2. Add 10 mL of (2N) $H_2SO_4$ solution to it
3. Titrate the solution against $KMnO_4$ from the burette till the solution becomes permanent pink color
4. Repeat the procedure to get three concordant readings

**Observation Table 1**

| No. of obs. | Volume of Ferrous ammonium sulphate + volume of $H_2SO_4$ in ml. | Burette reading in ml. | | | Remarks |
|---|---|---|---|---|---|
| | | IBR | FBR | Difference | |
| 1 | 10+10 | | | | rough |
| 2 | 10+10 | | | | |
| 3 | 10+10 | | | | |
| 4 | 10+10 | | | | |

**Calculation:**

Strength of $KMnO_4$ = Z

1000 ml of (1N) $KMnO_4$ = 55.85 g of $Fe^{+2}$ ion

Y ml of Z(N/10) $KMnO_4$ = 55.85*Y*Z/(1000*10) = A g of $Fe^{+2}$ ion

10 ml of supplied solution contains = A g of Fe ion

1000 ml of supplied solution contains = (A*1000)/(10) g of $Fe^{+2}$ ion

= B*1000 mg of

= _____ mg of $Fe^{+2}$ ion

**Conclusion:**

The amount of Ferrous iron present in the Mohr's salt is _____ ppm.

# Experiment-6

**Aim:** To determine the dye concentration using UV- visible spectrophotometer

**Apparatus required:**

- UV-visible spectrophotometer
- Volumetric flask
- Pipette
- Measuring cylinder

**Materials required:**

- Dye
- Distilled water

**Theory:**

Beer-Lambert's law states that there is a linear relationship between the concentration and the absorbance of the solution. The concentration of a solution can be calculated by measuring the absorbance of the sample.

According to Beer's Law, $A = \varepsilon bc$, where A is absorbance, ε is the molar absorptive with units of L $mol^{-1}$ $cm^{-1}$ (formerly called the extinction coefficient) b is the path length of the sample, usually expressed in cm and C is the concentration.

The molar extension coefficient is given as a constant for each molecule. The absorbance does not carry any unit. Since the absorbance and path length is known the concentration of any unknown sample can be determined.

**Procedure:**

1. Prepare 100 mL of 0.001M $KMnO_4$ standard stock solution
2. Prepare six standards in 100 mL volumetric flask with concentration of 0.0004M (solution-1), 0.0002M (solution-2), 0.0001M (solution-3), and 0.00005M (solution-4) by diluting the stock solution prepared in step-1
3. Rinse one cuvette with distilled water and fill with water
4. Put the cuvette in the sample compartment of UV-visible spectrophotometer. This is the reference solution.
5. Set the wavelength to 400 nm and set the absorbance to zero
6. Rinse the second cuvette once with distilled water and once with standard solution-1(0.001M) $KMnO_4$
7. Then fill the standard solution 1 in the cuvette
8. Place the cuvette in the sample compartment and measure the absorbance at 410 nm
9. Repeat the procedure (step-3 and 4) above for the two cuvette as wavelength 420,440,450, 500, 550, 600 nm.
10. Sett the absorbance to 0 for the cuvette with water .measure absorbance for the cuvette with 0.001M $KMnO_4$ and record the absorbance at each wavelength in the data Table
11. Prepare a graph of absorbance (A) vs. wavelength $\lambda$ and determine the maximum wavelength ($\lambda$)
12. Check the maximum $\lambda$ in the UV visible software. Place the cuvette with the distilled water in the same compartment and again set the absorbance to zero. Measure and record the absorbance of each of 6 standard solutions starting with the most dilute standard. After each measurement rinse the cuvette with next standard not with the

distilled water. Draw a plot having x-axis as concentration in moles/lit and y-axis as absorbance at maximum λ

13. Using Beer-Lambert's law to calculate the concentration £ for $KMnO_4$ by assuming the width of cell or path length to be 1cm.

**Observation Table 1**

| Solution | Concentration of solution (in M) | Volume of stock solution added in mL |
|---|---|---|
| 1 | 0.0004M | |
| 2 | 0.0002M | |
| 3 | 0.0001M | |
| 4 | 0.00005M | |

**Observation Table 2**

| Concentration | Absorbance | | | | |
|---|---|---|---|---|---|
| | 430nm | 440nm | 450nm | 500nm | 600nm |
| 0.0004M | | | | | |
| 0.0002M | | | | | |
| 0.0001M | | | | | |
| 0.00005M | | | | | |

Absorbance of different concentration $KMnO_4$ at different λ has to be performed to determine the maximum absorbance of $KMnO_4$ solution. Then plot a graph of absorbance $V_s$ wavelength.

**Conclusion:** The concentration of unknown sample was found to be_____