

Role of emulsifying agent

Aim

To study the role of emulsifying agents in stabilizing the emulsions of different oils.

Apparatus Required

• Test tubes : Six • Droppers : Five • Test tube stand : One • Glass rod : One • Stop watch : One

Material Required

1. Soap/detergent: 5g 2. Mustard oil, castor oil & coconut oil 10ml each.

Theory

Emulsion is a type of colloid in which, both the dispersed phase and the dispersion medium are liquids. Here the dispersed phase and the dispersion medium are distinguished by their relative amounts. The one, which is present in smaller proportion, is called **dispersed phase**, while the other, which is present in relatively large quantity, is known as the **dispersion medium**. When oil is shaken with water, a faint milky solution is often observed, which is unstable and is called an **emulsion of oil in water**. On standing, it gets separated into two layers, i.e. oil and water.

The stability of an oil and water emulsion is increased by the addition of a suitable emulsifying agent such as soap solution. Soap contains sodium salt of long chain aliphatic carboxylic acids with the carboxyl group as the polar group, which decreases the interfacial surface tension between oil and water. Hence oil mixes with water and emulsification takes place. In the presence of optimum amount of soap solution, oil in water emulsion is more stable and the separation of oil and water layers takes more time.

Procedure

- (i) Dissolve 1 g of soap/detergent in 10 mL of distilled water in a test tube with vigorous shaking and heat the content of the test tube if needed. Label it as 'A'.
- (ii) Take four test tubes. Mark these as B, C, and D and to each of the test tubes, add 5 mL distilled water followed by 10 drops of mustard oil in test tube B, , castor oil in test tube C and coconut oil in test tube D, respectively.
- (iii) Shake test tube B vigorously for five minutes, keep it in a test tube stand and simultaneously start the stopwatch. Record the time taken for the separation of the two layers.
- (iv) Repeat the same procedure with test tubes C and D and record the time for the separation of the layers in each case.
- (v) Now add two drops of soap/detergent solution from test tube 'A' into each test tube (B, C, and D). Shake each test tube for five minutes and record the time of separation of the layers in each case again.
- (vi) Record your observations in a manner detailed in Table 1.1

Note:- Rate each oil according to the time taken for separation in to two layers. The most stable emulsion takes the longest time.

Result:-The rating of the oils on the basis of the stability of emulsion formed is as follows

1. _____ oil (*Maximum time*)
2. _____ oil
3. _____ oil (*Minimum time*)

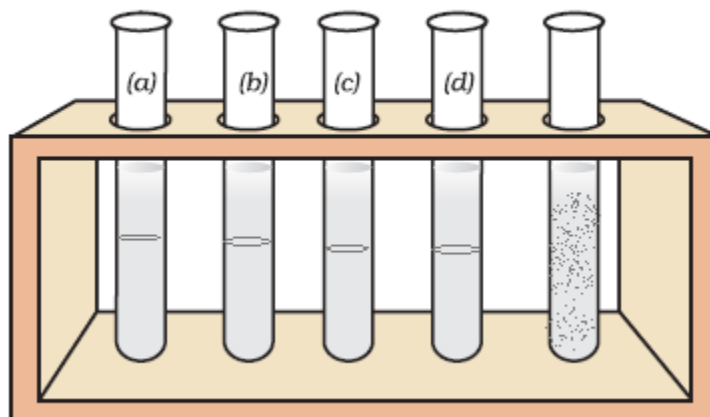
Observation

Table 1.1 : Emulsification of different oils by soap/detergent

Temperature:- _____K

Test tube specification	Name of oil used for emulsification	Time taken for the separation of layers	
		Without Soap/detergent	With Soap/detergent
B			
C			
D			

Diagram



Precautions

- Add equal number of drops of a soap/detergent solution to all the test tubes.
- To minimise the error in recording the time required for the separation of layers in different systems, shake all the test tubes for identical time span.
- Start the stopwatch immediately after shaking is stopped and stop it immediately when the two layers separate.

Discussion Questions

- Name a reagent other than soap, which can be used as an emulsifying agent in the *oil in water type emulsion*.
- Milk is said to be a stable emulsion. What provides stability to milk?
- Can two miscible liquids form an emulsion?
- Why do separation of layers of different oils forming an emulsion with water take different time?
- What are the points of similarity and dissimilarity among sol, gel and emulsion?
- Suggest a test to distinguish between *Oil in Water* and *Water in Oil* type of emulsions.
- Give some examples of emulsions that you come across in daily life.
- Dettol forms an emulsion in water. How does this emulsion get stabilised?

Cell potential in Daniel cell

Aim

To study the variation in cell potential of the cell $\text{Zn}/\text{Zn}^{2+}||\text{Cu}^{2+}/\text{Cu}$ with change in concentration of electrolytes ($\text{CuSO}_4/\text{ZnSO}_4$) at room temperature.

Apparatus Required

- Zinc plate : One
- Copper plate : One
- Beaker (50 mL) : Six
- Voltmeter (Potentiometer) : One
- Salt bridge : One

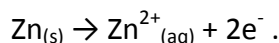
Material Required

1.0 M & 0.1M Zinc sulphate solution: 100mL each, 1.0M & 0.1M copper sulphate solution: 100mL each

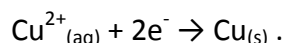
Theory

In the Daniel cell, copper and zinc electrodes are immersed in a solution of copper(II) sulfate and zinc sulfate respectively.

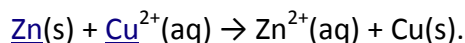
At the anode, zinc is oxidized per the following half reaction:



At the cathode, copper is reduced per the following reaction:



The total reaction being:



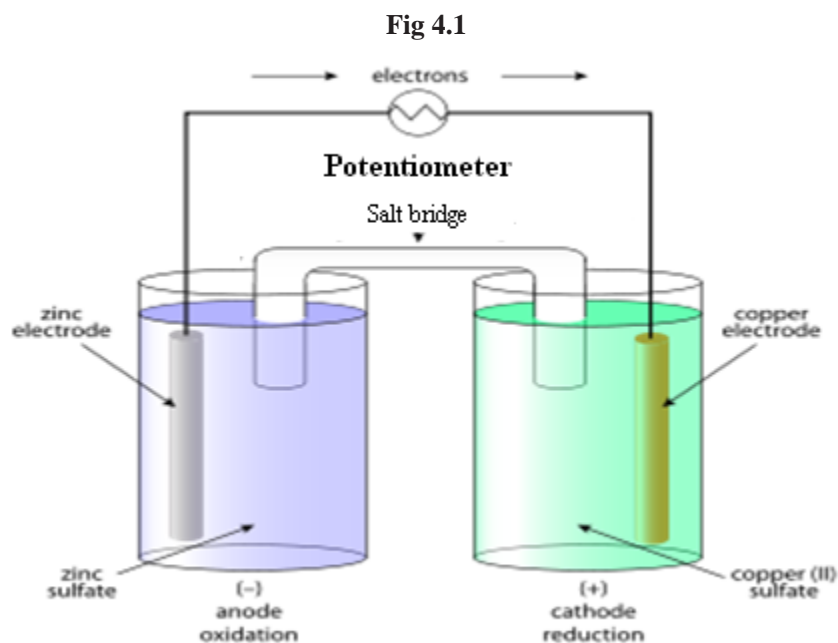
Procedure

- Set up the cell as given in Fig. 4.1, using 1.0 M ZnSO_4 and 1.0 M CuSO_4 solution.
- Measure the potential difference of the cell using potentiometer and record the cell potential
- Remove the salt bridge as soon as the cell potential measurement is over.
- Repeat the procedure as per different combinations of copper sulphate & Zinc sulphate as shown in observation table
- Calculate the cell potential using Nernst equation and record it in the given table.

Result

The cell potential varies with change in concentration of Copper sulphate & Zinc sulphate solution and values observed are in agreement with cell potential values calculated using Nernst equation

Diagram:



Observation:

Obs.No	Concentration of Solution		E° cell in Volts	E cell in Volts	
	Copper Sulphate	Zinc Sulphate	Std Cell Potential	Calculated	Observed
			$E^{\circ} = E^{\circ}_{\text{cathode}} - E^{\circ}_{\text{anode}}$		
1	1M	1M	1.10	1.10	
2	0.1M	0.1M	1.10	1.10	
3	1M	0.1M	1.10	1.1295	
4	0.1M	1M	1.10	1.0705	

Calculations:

$$1. E^0 = E^0_{\text{cathode}} - E^0_{\text{anode}}$$

$$E^0_{\text{cathode}} = +0.34$$

$$E^0_{\text{anode}} = -0.76$$

$$E^0 = 0.34 - (-0.76) = 1.10 \text{ volts}$$

2. To calculate E_{cell} for Obs.No 1

$$E_{\text{cell}} = E^0_{\text{cell}} - \frac{2.303RT}{nF} \log Q \frac{[\text{Zinc ions}]}{[\text{Copper ions}]}$$

$$\log Q = \frac{[\text{Zinc ions}]}{[\text{Copper ions}]} \times \frac{[\text{Copper}]}{[\text{Zinc}]} \quad \text{but} \quad \frac{[\text{Copper}]}{[\text{Zinc}]} = 1$$

R=8.314J/K, T=298K and n=2

$$E = (E_R^0 - E_L^0) - \frac{0.059}{2} \log \frac{[\text{Zn}^{2+}_{(\text{aq})}]}{[\text{Cu}^{2+}_{(\text{aq})}]}$$

$$E_{\text{cell}} = 1.10 - \frac{0.0591}{2} \log \frac{[1]}{[1]}$$

$$\log 1 = 0$$

$$E_{\text{cell}} = 1.10$$

Similarly for **Obs.No 2** also

$$E_{\text{cell}} = 1.10 - \frac{0.0591}{2} \log \frac{[0.1]}{[0.1]}$$

$$E_{\text{cell}} = 1.10 \text{ volts}$$

For **Obs.No 3**

$$E_{\text{cell}} = 1.10 - \frac{0.0591}{2} \log \frac{[0.1]}{[1]}$$
$$\log \frac{[0.1]}{[1]} = \log 10^{-1} = -1$$

$$E_{\text{cell}} = 1.10 - \frac{0.0591}{2} \times -1$$

$$E_{\text{cell}} = 1.10 + 0.295$$

$$E_{\text{cell}} = 1.1295 \text{ volts}$$

For **Obs.No 4**

$$E_{\text{cell}} = 1.10 - \frac{0.0591}{2} \log \frac{[1]}{[0.1]}$$
$$\log \frac{[1]}{[0.1]} = \log 10^1 = +1$$

$$E_{\text{cell}} = 1.10 - \frac{0.0591}{2} \times +1$$

$$E_{\text{cell}} = 1.10 - 0.295$$

$$E_{\text{cell}} = 1.0705 \text{ volts}$$

Precautions

- Clean copper and zinc strips and connecting wires with sand paper before use.
- Place the salt bridge immediately in distilled water after its use.
- Carry out dilution of the solution to another concentration very carefully.

Discussion Questions????

- What is a Electrochemical cell?

Paper chromatography

Aim

Separation of pigments present in the leaves (spinach) and flowers (rose, marigold) by paper chromatography and determination of R_f value of components.

Theory

In paper chromatography, water molecules present in the pores of the filter paper act as the stationary phase and the moving phase can be a solvent like hexane, toluene, acetone or a mixture of solvents such as methanol-water mixture etc. As the moving phase passes through the spot on which sample has been adsorbed, it dissolves the components more or less readily; depending upon the solubility and carries them along with it while moving on the support.

At a given temperature and for a given solvent, it is possible to determine the characteristic rate of movement of each substance on the chromatographic paper, as the moving phase moves. This is represented by relative front or **retardation factor also called R_f value**. R_f values of different compounds are different even if the mobile phase (solvent) is same. Furthermore, R_f value of a compound may be different in different solvents. R_f values can be calculated by using the following expression:

$$R_f = \frac{\text{Distance travelled by the substance from reference line (cm)}}{\text{Distance travelled by the solvent front from reference line (cm)}}$$

Since solvent front moves faster than the compounds, the R_f value of a substance will always be less than one. Also note that R_f value has no unit.

If the compound is coloured then its position on the chromatographic paper may be easily located. However, if the substance is colourless, it may be treated with a reagent, which imparts it a characteristic colour. This reagent is given the name **developer**. Iodine is the most commonly used developer in paper chromatography. Several other techniques are available for locating the spots.

Material Required

Whatman's filter paper No.1 of size 4 cm × 17 cm : One • Gas jar of size 5 cm × 20 cm: One
• Rubber cork fixed with hook in the centre : One • Test tubes : As per need
• extract of leaves : As per need • Distilled water : As per need • Methanol/Acetone : As per need
• Petroleum ether boiling range (60–80°C) : As per need • Chloroform /Acetone : As per need

Procedure

- (i) Grind leaves in a mortar and transfer the paste into a test tube.
- (ii) Add small amounts of methanol or acetone in the crushed material. Close the test tube with an appropriate cork and shake it well. Filter it and collect the filtrate in a test tube and cork the test tube.
- (iii) Procure a Whatman filter paper No.1 of size 4 cm × 17 cm and mark a line at a distance of 3 cm from one of the ends of the paper with the help of a pencil [Fig. 5.1(a)].
- (iv) Using a finely drawn capillary put one spot 'a' for the extract of leaves. Allow these spot to dry as shown in Fig. 5.1 (a).
- (v) Hang the filter paper in a jar containing 20 mL mixture of petroleum ether (boiling range 60–80°C) and chloroform containing 19 mL petroleum ether and 1 mL chloroform or a mixture of petroleum ether (boiling range 60–80°C) and acetone in the ratio 9:1 (18 mL petroleum ether + 2 mL acetone) so that the solvent does not touch the reference line as given in Fig. 5.1 (b).
- (vi) Keep this jar as such till the mobile phase (solvent) rises up to 2/3 of the length of the paper [Fig. 5.1(c)].
- (vii) Remove the filter paper from the jar, mark the solvent front, outline the spots with the help of a pencil and allow the filter paper to get dry.
- (viii) Measure the distance travelled by the solvent front and the centre of different spots with respect to the reference line as given in Fig. 5.1 (d).

- (x) Ascertain the number of pigments, which are present in the extract of leaves
- (xi) Calculate the R_f value of different spots with the help of the expression mentioned earlier.
- (xii) Record your observations as in Table 5.1.

Result

(i) R_f values of components of flower are _____.

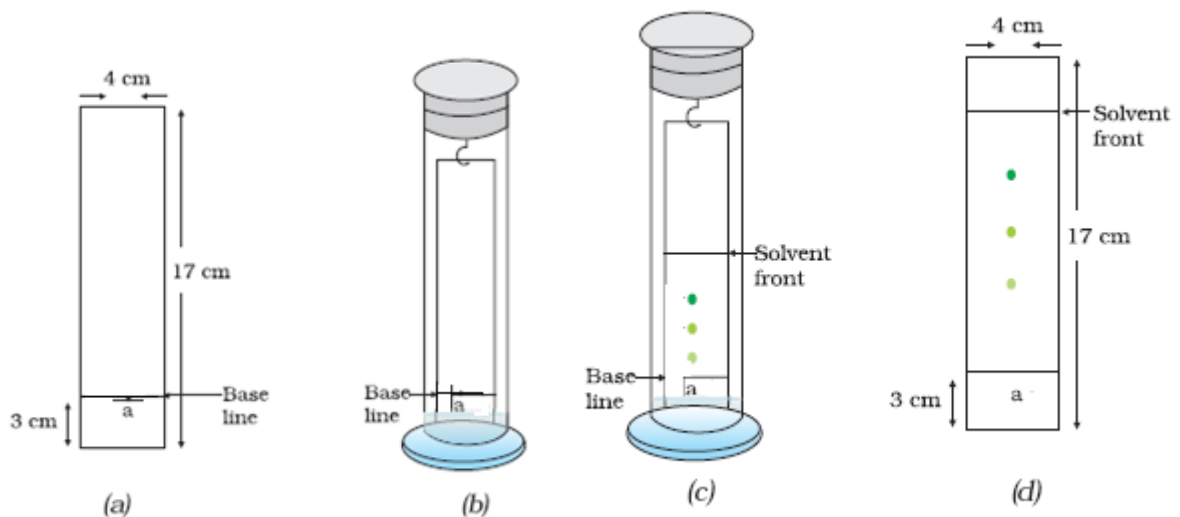


Fig. 5.1 : (a) Marked paper; (b) Dipping the filter paper in the solvent; (c) Developing chromatogram; and (d) Developed chromatogram

Table 5.1: Separation of pigments of leaves

Sl. No.	Name of the extract	Colour of the spot	Distance travelled by the components of the spot 'a' from the reference line in cm	Distance travelled by the solvent from reference line in cm	R_f value
1.					
2.					
3.					
4.					

Precautions

- (b) Dip the paper strip in the solvent in such a way that the spot of the mixture is above the solvent level and the movement of the solvent front is not zig-zag.
- (c) While spotting the test solution on the paper, do not allow the spots to spread. Use finely drawn capillary to put the spot on the paper.
- (e) Once the experiment is set, do not disturb the jar as long as the chromatogram is being developed.
- (f) Keep the jar covered with the lid when the chromatogram is being developed.

PREPARATION OF INORGANIC COMPOUNDS

Aim

To prepare double salt: ferrous ammonium sulphate (Mohr's salt).

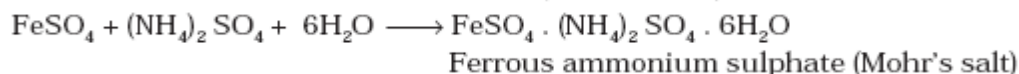
Material Required

Beaker (50 mL) : One • Conical flask (50 mL) : One • Trough : One • Glass rod : One • Tripod stand : One
• Funnel : One • Wire gauze : One

• Ferrous sulphate : As per need • Ammonium sulphate : As per need • Dilute sulphuric acid : As per need
• Ethanol : As per need

Theory

When a mixture containing equimolar proportions of ferrous sulphate and ammonium sulphate is crystallized from its solution, a double salt is formed. The formation of double salt may be shown as follows:



Fe^{2+} and Al^{3+} ions undergo hydrolysis, therefore, while preparing aqueous solutions of ferrous sulphate and aluminium sulphate in water, 2-3 mL dilute sulphuric acid is added to prevent the hydrolysis of these salts.

Procedure

Preparation of Double Salt: Ferrous Ammonium Sulphate

- (i) Dissolve 3.5 g of ferrous sulphate and 1.7 g of ammonium sulphate (weighed separately), in 5 mL of distilled water contained in a 50 mL conical flask by heating. Add about 0.5 mL of dilute sulphuric acid to the flask and concentrate the solution by heating till the crystallization point is reached.
- (ii) Allow the mixture to cool to room temperature slowly.
- (iii) On cooling, light green crystals of ferrous ammonium sulphate separate out.
- (iv) Decant the mother liquor and wash the crystals by shaking with very small amounts of 1:1 cold water and alcohol mixture to remove sticking mother liquor.
- (iv) Separate the crystals by filtration wash with alcohol, dry between the folds of a filter paper and record the yield.

Result

Yield of Mohr's salt is _____ g.

Precautions

- (a) Cool the solution slowly to get good crystals. Avoid rapid cooling.
- (b) Do not disturb the solution while cooling.
- (c) Avoid prolonged heating while preparing crystals of ferrous ammonium sulphate, as it may oxidise ferrous ions to ferric ions and change the stoichiometry of the crystals.

Discussion Questions?????

- (i) Why do we take equimolar quantities of reacting compounds in the preparation of double salts?
- (ii) In the preparation of ferrous ammonium sulphate, can concentrated sulphuric acid be used in place of dilute sulphuric acid? Explain.
- (iii) What is the difference between iron compounds; $\text{K}_4[\text{Fe}(\text{CN})_6]$ and $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$?
- (vi) What is the difference between a complex compound and a double salt?

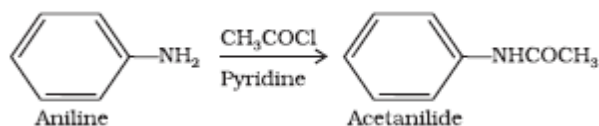
PREPARATION OF ORGANIC COMPOUNDS

Aim

To prepare acetanilide.

Theory

The replacement of one hydrogen atom of the —NH_2 group of aniline by $\text{CH}_3\text{CO—}$ group in the presence of glacial acetic acid. Gives acetanilide. In the laboratory, acetylation is usually carried out with acetic anhydride. Acetyl chloride may also be used for the purpose of acetylation if acetic anhydride is not available. Acetylation with CH_3COCl is usually carried out in the presence of pyridine.



Material Required

Funnel : One • Round bottomed flask (100 mL) : One • Beaker (250 mL) : One • Air condenser : One
• Sand bath : One • Clamp and iron stand : One • Pumice stone : As per need • Melting point assembly : One

• Aniline : 5 mL • Acetic anhydride /Acetyl chloride : 5 mL • Acetic acid / Pyridine : 5 mL

Procedure

- (i) Take 5 mL of aniline in a 100 mL round bottom flask and add acetylating mixture containing 5 mL acetic anhydride and 5 mL glacial acetic acid. Alternatively, you can use 5 mL of acetyl chloride and 5 mL of dry pyridine as the acetylating mixture.
- (ii) Fit an air condenser on the mouth of the round bottom flask after adding a few pumice stones and reflux the mixture gently for 10-15 minutes on a sand bath.
- (iii) Cool the reaction mixture and pour it slowly in 150-200 mL of ice cold water with stirring.
- (iv) Filter the solid, wash it with cold water and recrystallise a small amount of sample from hot water containing a few drops of methanol or ethanol.
- (v) Report the yield and the melting point of the compound.

Result

- (a) Yield of acetanilide _____ g.
- (b) Melting point of acetanilide is _____ °C.

Precautions

- (a) Handle acetic anhydride and acetyl chloride carefully as they cause irritation to the eyes and acetyl chloride also strongly fumes in air.
- (b) Store acetylchloride under dry conditions.
- (c) Handle pyridine with extreme caution. Dispense it in an efficient fume cupboard and wear disposable glasses while using it.
- (d) Distil pyridine before use because it absorbs moisture and the reaction does not take place under moist conditions.
- (e) Wash the solid 2-3 times with cold water till the filtrate is neutral to litmus.
- (f) Determine the melting point of perfectly dried and recrystallised sample.