LAB 2: Two Component Systems Containing Liquid Phases (Part 1)

Introduction:

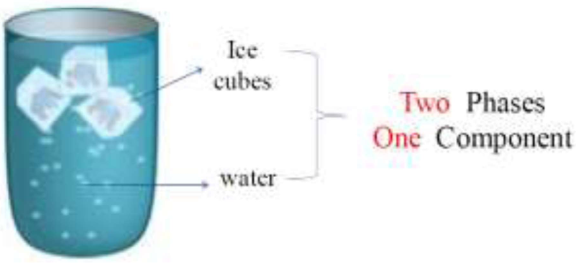
Preparation of pharmaceutical solutions involves mixing of 2 or more liquids (alcohol & water to form hydro-alcoholic solutions, volatile oils & water to form aromatic waters, volatile oils and alcohols to form spirits, etc.). Thus, it appears important to study the behavior of mixing liquids.

Definitions:

A "phase" may be defined as a homogenous, physically distinct portion of a system, which is separated from other portions of a system by a boundary.

A "component" is a substance in a system with a defined chemical structure.

- A gas or a gaseous mixture is a single phase.
- Totally miscible liquids constitute a single phase.
- A solution of sodium chloride in water is a single phase, two component system.
- In an immiscible liquid system, each layer is counted as a separate phase.
- A system containing water and its vapour forms a one component, two phase system
- A system composed of ice, water, and water vapour is a one component but a three phase system.



A phase may contain one or more component (e.g., ethanol water system is a one phase, 2 component system)

When dealing with 2 component liquid-liquid systems, regarding their miscibility, they are divided into 3 categories:

1. **Completely miscible systems**: such as alcohol & water, glycerin & water, glycerin & alcohol, benzene & carbon tetrachloride. Such systems are one liquid layer (1 phase) and both liquids are miscible in all portions.
2. **Completely immiscible systems**: such as mercury & water, castor oil & water. Such systems are formed from two liquid layers (2 phase system) which are completely immiscible regardless of the amounts of each present.

3. **Partially miscible systems**: such as phenol & water, ether & water, nicotine & water. Such systems appear as one or two liquid layers (1 or 2 phases) depending on the temperature and concentration (i.e., at certain ratios and at certain temperature, the two liquids are completely miscible and appear as one phase, and at other ratios and temperatures they appear as completely immiscible and appear as 2 phases).

Experimental work:

Objectives

1. To determine the miscibility of 2 partially miscible liquids (phenol in water)
2. Study the effect of temperature and concentration on miscibility of phenol and water.
3. Construct a phase diagram (a plot of the percent by weight of phenol against temperature) for the phenol in water liquid system.
4. Determine the composition of phases in a water-phenol system prepared at certain concentration at certain temperature.

Principle

A number of different mixtures of phenol and water are prepared and heated until complete miscibility is achieved. As the mixtures cool, two-phase systems form at certain temperatures which are recognizable by the appearance of turbidity. Plotting separation temperatures against percent compositions of the mixtures gives the binodal (separation curve).

Apparatus and Materials

- Chemicals: Phenol, water
- Glass-ware and equipment: boiling tubes, boiling tube rack, pipette, glass stirrer, and thermometer.
- Instruments: Balance, water bath

Procedure

1. Prepare (10 gm) mixtures of different concentration (%w/w) of phenol in water (2 %, 7%, 9 %, 11%, 24%, 40 %, 55 %, 63%, 70 % and 73 % w/w). This is done by weighing the respective phenol portions (as shown in table below) into appropriately numbered test tubes (use a beaker to support the test tubes and use the micro-burette to add the required quantity of water.)

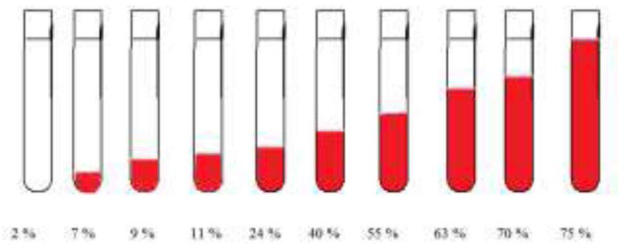
Test tube no.	phenol in water (%w/w)	Phenol weight (gm)	Water weight (gm)
1	2	0.2	9.8
2	7	0.7	9.3
3	9	0.9	9.1
4	11	1.1	8.9
5	24	2.4	7.6
6	40	4.0	6.0
7	55	5.5	4.5
8	63	6.3	3.7
9	70	7.0	3.0
10	75	7.5	2.5

2. Seal the test tubes with rubber stoppers and heat them in a temperature-controlled water bath at (25 °C) for 10 min. During heating remove the rubber stoppers from time to time to release excess pressure and shake the mixtures.
3. After the elapse of specified time, take out the test tubes, and observe them before their temperature has changed; record at which concentration you have two phases and which one has one phase. Tabulate the results for ease of observation.
4. Repeat the work and record observations at a higher temperature of 40 °C, 50 °C, and 70 °C.
5. Plot a phase diagram or mutual solubility curve for the phenol-water system (graph of temperature against % w/w phenol concentration). This can be done by assigning the points of concentrations showing 2 phases as (x) on the plot, then attach between these points to obtain a bimodal curve. Show the area of 2 phase and area of 1 phase in the graph.
6. Draw the tie line for each temp.
7. Mention the upper consulate temp.

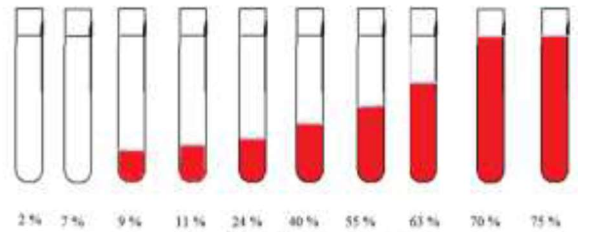
Results

Temperature (°C)	Concentration (w/w)									
	2 %	**7 %**	**9 %**	**11 %**	**24 %**	**40 %**	**55 %**	**63 %**	**70 %**	**73 %**
25										
40										
50										
70										

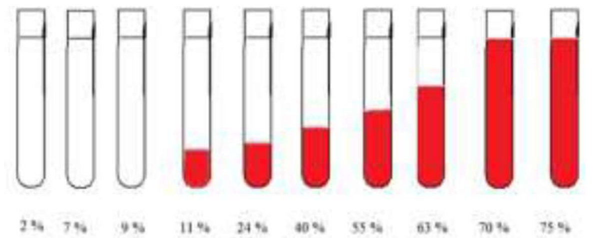
Number of phases obtained for varying (% w/w) concentrations of phenol in water at different temperatures



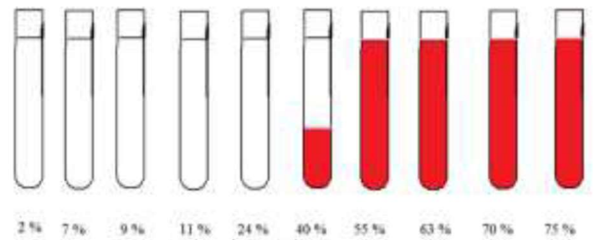
At 25 °C



At 40 °C



At 50 °C



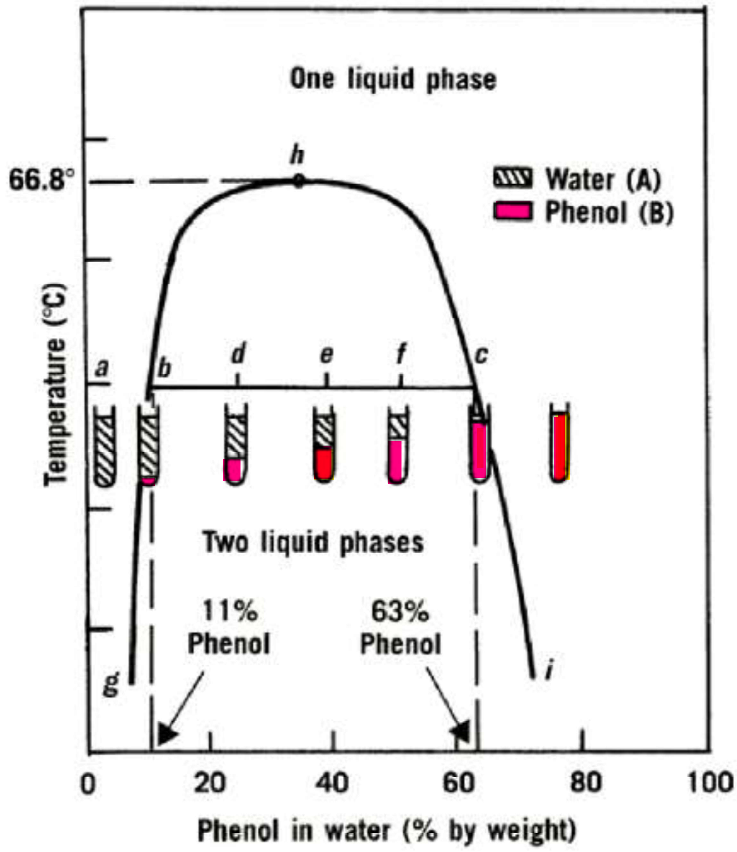
At 70 °C

Constructing a Phase Diagram

A ***phase diagram*** (also called equilibrium, solubility diagram or binodal curve) is a plot or diagram of one system variable versus another. It summarizes the relationship between specific phases and the combination of variables (e.g., temperature, concentration…) at equilibrium. At any point on the phase diagram boundaries, the phases are in dynamic equilibrium.

- For phenol-water system a phase diagram is obtained by plotting the percentage of phenol in water (represented on x-axis) against different temperatures (represented on y-axis).
- The number of phases obtained at different concentrations and temperatures is represented on the phase diagram. This can be done by representing concentrations showing the beginning of 2 phases at certain temperature by a point (●), then by attaching between these points, a binodal curve would be apparent (i.e, the curve of the graph shows the limits of the temperature and concentration within which two liquid phases exist in equilibrium).
- Binodal curve will separate 2 phase area from one phase area. All concentrations lying inside the curve represent 2 phase systems while those lying outside the curve represent 1 phase systems.

If a series of tubes containing varying, but known, percentages of phenol and water are just to the point of formation of a homogeneous solution, and the temperature at such points is noted, upon plotting the results a curve is obtained similar to that in the figure below.



Starting at the point **a,** equivalent to a system containing 100% water (pure water) at 50 °C, the addition of known increments of phenol to a fixed weight of water, the whole being maintained at 50°C, will result in the formation of a single liquid phase until the point **b** is reached, at which a minute amount of a second phase appears.
As we prepare mixtures containing increasing quantities of phenol, that is, as we proceed across the diagram from point **b** to point **c**, we form systems in which the amount of the phenol-rich phase continually increases. At the same time, the amount of the water-rich phases decreases. Once the total concentration of phenol exceeds 63%, at 50 °C, a single phenol-rich liquid phase is formed.

- The maximum temperature on this curve is called the ***critical solution temperature*** (**CST** or ***upper consolute temperature (UCT)***), it is defined as the temperature above which a homogeneous completely miscible, one-phase liquid system exists, regardless of the composition of the mixture (or: it can be defined as the maximum temperature at which the two-phase region exists).

    - In the case of phenol and water, the mutual solubility increases with an increase in temperature, and the critical solution temperature occurs at a relatively high point and is known as Upper Consolute Temperature
    - In a certain number of cases, the mutual solubility increases with decrease in temperature, and the critical solution temperature occurs at a relatively low value. Most of the substances that show lower critical solution temperatures are amines such as, for example, triethylamine with water.
    - There are pairs of liquids that show both upper and lower critical solution temperatures, and the mutual solubility curve is of the closed type. An example of this type of liquid pair is found in the case of nicotine and water. Mixtures of nicotine and water represented by points within the curve will separate into two layers, but mixtures represented by points outside of the curve are perfectly miscible with each other

The information which can be obtained from phase diagram include:

1. The number of phases present at certain concentration and temperature
2. The upper consolute temperature of the system
3. The relative fraction of each phase
4. Composition of each phase