

Effect on the Upper Consolute Temperature (UCT) of a Partially Miscible Phenol-water Solution with Addition of Ionic Compounds like NaCl, KCl, and Organic Compounds like C₁₀H₈ (Naphthalene), and C₁₀H₁₆O (Camphor) Producing a Ternary System

Anmol Mathur

Corresponding author email id: anmolyoyo2@gmail.com

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Abstract – Phenolic compounds are ubiquitous in today's modern pharmaceuticals with immense uses in the aviation to paint and to the cosmetics industries. Phenol's properties have been studied for centuries, particularly its partial miscibility with water. Generally, partially miscible liquids become more soluble with the increase in temperature and at a certain temperature they are completely miscible. This temperature is known as the critical solution temperature (CST) or consolute temperature. The temperature above which the phases of a system are completely miscible is known as the upper consolute temperature (UCT) and it gets affected by the addition of impurities. To find the miscibility temperature, the mixture was heated in a boiling tube until the turbidity disappeared and the final temperature was noted. Then, the mixture was cooled down and the temperature noted when the turbidity reappeared. Solutions of impurities of different concentrations were formed and their effect on the UCT of the ternary system created by the impurities, Phenol, and water was analyzed.

It was found that the ionic compounds, which get hydrated with water, show lesser increase in CST as they decrease the miscibility to a lesser extent. Further, at all concentrations, CST of phenol-water system containing Camphor as impurity is maximum and it increases with increasing concentration. Thus, Camphor is the most suitable substance to be added to increase the UCT of phenol-water system.

Keywords – Critical Solution Temperature/Consolute Temperature, Ionic and Organic Impurities in Phenol-Water System, Phenol-Water System, Ternary System.

I. INTRODUCTION

A homogenous mixture of a solute (dispersed phase) and a solvent (dispersion medium) is called as a true solution.¹ Various solutions are: solid-solid, solid-liquid, liquid-liquid, gas-liquid, etc. The liquid-liquid are of three types:

- Completely miscible liquid pairs - E.g. Water-Alcohol, Water-Sulphuric Acid
- Partially miscible liquid pairs - E.g. Ether-Water, Phenol-Water

¹Xamplified. "True Solution, Suspension and Colloidal Solution." *Colloidal Solution*. Xamplified, . Web. 14 Oct. 2015.

² UA, Waterloo. "Solutions." *Science.uwaterloo.ca*. University of Waterloo, . Web. 14 Oct. 2015.

³ Drugs.com. "Phenol Spray." *Drugs.com: Phenol Spray*. Drugs.com, 1 Jan. 2000. Web. 14 Oct. 2015.

⁴ Greener-Industry. "Phenol." *Greener-industry.org.uk*. Wwww.greener-industry.org.uk, . Web. 15 Oct. 2015.

⁵ Britannica Eyclopedia. "Bakelite." *Encyclopedia Britannica Online*. Encyclopedia Britannica, . Web. 15 Oct. 2015.

- Liquid pairs that are practically immiscible - E.g. Mercury-Water, Nitrobenzene-water.²

Many substances that we use every day contain partially miscible liquids like phenol-water, in which, the components of a system might not be miscible over the entire concentration range. If a component, say A, is added to component B, gradually, after a certain composition the system separates in two phases, one of which is a solution of A in B and the other is a solution of B in A. But increasing the temperature leads to a state of complete miscibility. This happens because the kinetic energy of the molecules increases due to the heat and they overcome the repulsion between A and B.

Most commercially available, over-the-counter, bottles of throat aid or spray contain phenol as a major ingredient. Phenol is also known as carboic acid or benzenol³. C₆H₅OH is the molecular formula of phenol and contains an alcohol group bonded to an aromatic ring. It is inexpensive and its mixture with water is thus used for many small-scale purposes, such as an antiseptic, in paint strippers, chemically resistant coatings⁴, in the aviation industry, preparation of plastics like Bakelite⁵, in the manufacture of cosmetics like sunscreens, hair color and in skin lightening⁶. Further, aqueous phenol is used in pharmaceuticals for the formation of microcapsules and in polymer coating methods⁷.

II. CRITICAL SOLUTION TEMPERATURE OR CONSULATE TEMPERATURE

When phenol and water are mixed, a certain amount of the two dissolves with the other due to hydrogen bonding and two conjugate layers of liquids are obtained. The upper layer of this system is water dissolved in phenol and the lower layer is phenol dissolved in water. The composition of these layers depends only on the temperature of the system and is independent of the quantities of the liquids

⁶ Vermerris, Wilfred, and Ralph L. Nicholson. "Chapter 7: Phenolic Compounds And Their Effects on Human Health." *Phenolic Compound Biochemistry*. Dordrecht, the Netherlands: Springer, 2006. 235-37. Print.

⁷ Jadhav, Rajendra S., Vishal Mane, Avinash V. Bagle, Dilip G. Hundiware, Pramod P. Mahulikar, and Gulzar Waghoo. "Synthesis of Multicore Phenol Formaldehyde Microcapsules and Their Application in Polyurethane Paint Formulation for Self-healing Anticorrosive Coating." *International Journal of Industrial Chemistry Int J Ind Chem* 4.1 (2013): 31. Springer. Web. 16 Oct. 2015.

mixed. For every temperature two solubilities can be obtained, one for phenol dissolved in water and the other for water dissolved in phenol.

Generally, for partially miscible liquids solubility increases with increasing temperature and the temperature at which they are completely miscible is termed as the Critical Solution Temperature (CST) or Consolute Temperature. The temperature above which the phases of a system are completely miscible is known as the Upper Consolute Temperature (UCT) or Upper Critical Solution Temperature (UCST). Similarly, the temperature below which the phases of a system are completely miscible is known as the Lower Critical Solution Temperature (LCST). For example, a hexane-nitrobenzene system has an UCST of 19°C and these substances are not miscible below this temperature. Similarly, for a tri-ethylamine-water system the LCST is 19°C i.e. the system is completely miscible below this temperature and not above it⁸. The phenol water system exhibits an upper critical solution temperature and this critical solution temperature can be used for testing the purity of the mixture.

To Phenol–water system if impurities of ionic and covalent substances are added, a ternary system is formed.⁹ For a ternary system, there are three components co-existing in a system.¹⁰ The addition of a third substance to a partially miscible system to increase its miscibility is known as blending. This is also called as the ‘salting-out’ in pharmaceuticals and is used to select the best solvent for the drugs.¹¹ In this process, a salt is added to separate the aqueous phase from the organic phase.

If the added salt dissolves in one of the phases only, it results in an increase in the UCST, and a decrease in the LCST, thus decreasing the miscibility of the system. Further, if the added salt is soluble in both the phases, then the UCST is lowered and the LCST is raised, thus increasing the miscibility of the system. This increase or decrease in the CST depends on the nature and the mass of the added substance and the composition of the system.

II. FACTORS AFFECTING THE MISCIBILITY OF A LIQUID-LIQUID SYSTEM

The various factors affecting the solubility of a liquid-liquid system are: solute-solvent interactions, common ion effect, molecular size and mass and polarity¹². If the attraction between the added substance and the solvent particles is greater, higher miscibility can be observed in a system and vice versa. When a substance containing an ion, which is already present in the equilibrium state of the system, is added to that system, a decrease in miscibility is observed. This decreasing effect of the miscibility is known as the common ion effect. As the size and mass of the

molecule of the added substance increases, the miscibility of the system decreases and hence, the CST increases. Ionic solutes dissolve in polar solvents whereas organic solutes dissolve in non-polar solvents.

Thus, the addition of impurities like NaCl, KCl, Naphthalene and Camphor should reduce the miscibility of phenol and water. This is because of the nature of combining of the water molecules with these ions and hence, simple ions reduce the tendency of water to solvate phenol¹³. As a result, the addition of salt should always increase the critical solution temperature. Therefore, the aim of this research is:

How does the Upper Consolute Temperature (UCT)(in°C) of a partially miscible phenol-water solution changes with addition of varied concentrations of foreign ionic compounds like NaCl (Sodium Chloride), KCl (Potassium Chloride), and organic compounds like C₁₀H₈ (Naphthalene), and C₁₀H₁₆O (Camphor) producing a ternary system?

Research Presupposition

The CST is a characteristic feature of a partially miscible liquid solution and addition of the foreign substances, ionic like NaCl, KCl and organic like Naphthalene and Camphor in phenol-water system affect the CST. Since organic substances have large molecular mass, addition of organic compounds should affect CST of the system the most as compared to ionic substances.

III. RESEARCH VARIABLES

In a phenol-water system the concentration of phenol in water in mass percentage is the independent variable which is varied throughout the experiment from 75% to 30% and from 90% to 45% along with 0.2% to 0.8% solution of added impurities to determine the miscibility temperature. Miscibility temperature and thus the CST, depends on the concentration of phenol-water solutions and on the type of impurities added to the system. Thus, the CST is a dependent variable.

The concentration of all impurities added was kept constant. Also, two ionic (Sodium Chloride and Potassium Chloride) and two non-polar impurities (Naphthalene and Camphor), all of which are only soluble in one phase of the system, either phenol or water were used. Impurities like succinic acid (soluble in both water and phenol) were deliberately excluded because this research aimed to only study the upper critical solution temperature of the system.

It was important to ensure that the readings are taken at the precise moment of disappearance and reappearance of the turbidity of the mixture. Hence, a white card with a black cross was used, and it was suspended from the clamp stand, right behind the boiling tube. The mixture was very

⁸ Atkins, P. W., and Julio De Paula. "Equilibrium: Simple Mixtures." Atkins' Physical Chemistry. 8th ed. Oxford: Oxford UP, 2006. 180-82. Print.

⁹ Nelson, Stephen A., Prof. "Ternary Phase Diagrams." Tulane University. Prof. Stephen A. Nelson, 13 Jan. 2011. Web. 16 Oct. 2015.

¹⁰ DeVoe, Howard. "Phase Diagrams: Ternary Systems." UC Davis ChemiWiki. UC Davis ChemiWiki, . Web. 16 Oct. 2015.

¹¹ Turtinen, Lloyd W., and Brian D. Juran. "Protein Salting-Out Method Applied to Genomic DNA Isolation from Fish Whole Blood." *Protein Salting-Out Method Applied to Genomic DNA Isolation from Fish Whole Blood* 24.2 (1998): 238-39. *BioTechniques*. Web. 16 Oct. 2015.

¹² Clugston, M. J., and Rosalind Flemming. "Chemical Equilibrium." *Advanced Chemistry*. Oxford: Oxford UP, 2000. 182-84. Print.

¹³ Dhirendra, Singh. "Water-Phenol Miscibility Diagram." Academia.org. Academia.org, . Web. 18 Oct. 2015.

slightly stirred by a glass rod stirrer placed with the thermometer in the boiling tube and hence the rod moved up and down across a small height. To keep the stirring regular, it was moved once in approximately every 30 seconds. To prepare the solutions of different concentrations of ionic and organic compounds, only water was used as a solvent irrespective of their respective extent of solubility in water.

All experiments were carried out at R.T.P. since pressure can also affect the solubilities of different components. The standard state of all the four added impurities was the same, i.e. all exist as solids at R.T.P. This is because the change in state can also affect the solubility. While finding the UCST of various solutions of phenol-water and impurities, the mixtures were heated in the same water bath by a constant flame of a Bunsen burner to provide the mixture with constant heat.

IV. RESEARCH METHODOLOGY

Procedure for Calculating the CST of a Phenol-Water System (without added impurity)-

1. The hard glass boiling tubes were thoroughly cleaned and dried.
2. In one boiling tube, 75% phenol solution was prepared by weighing 9g Phenol and adding 3ml of water to it. (percentage from 75% was taken because for concentrations above this the turbidity reappears only if the solution is cooled down by putting it in an ice bath).
3. It was observed that turbidity appears.
4. The miscibility temperatures of the mixture prepared were found as follows:
 - A. The inner boiling tubes with the solutions were fitted in an outer jacket by means of a rubber cork. The inner tube was fitted with a cork, a thermometer and a stirrer.
 - B. This apparatus was then placed in a 500ml beaker containing water, and its temperature was raised slowly using a Bunsen burner, ignited on low flame.
 - C. The water bath was heated and the mixture was stirred at regular intervals of time.
 - D. The heating of the water bath was slowed by decreasing the flame of the burner, when the turbidity started disappearing.
 - E. The temperature from the thermometer when the turbidity disappeared completely was noted and the burner was turned off.
 - F. Further, set up was left to cool down so that the turbidity reappeared completely and this temperature was noted.
 - G. Using these temperatures, the miscibility temperature of the reaction mixture was calculated by the formula:

$$\text{Miscibility Temperature of a solution} = \frac{\text{Temperature at which turbidity disappears} + \text{Temperature at which turbidity reappears}}{2}$$

5. After adding 1ml water to the boiling tube after each trial, this procedure was repeated from step A to G; this helped to obtain the miscibility temperature of different percentage compositions of phenol in water.

6. The temperature data obtained was recorded in data table.

Finding the Effect of NaCl on the CST of the Phenol-Water system-

7. Preparation of 1% NaCl solution

- 1 gram of Sodium Chloride (NaCl) salt was weighed by an electronic weighing machine.
 - This was dissolved NaCl in distilled water in the beaker and made the volume 100ml by using measuring cylinder: the solution obtained is 1% NaCl.
8. Then, 10ml each of 0.2%, 0.4%, 0.6%, and 0.8% of NaCl solutions were prepared by adding 2, 4, 6 and 8 ml of 1% NaCl to 8, 6, 4 and 2ml of distilled water respectively in 4 different 50 ml beakers, with the help of 10ml measuring cylinders.
 9. The prepared 10 ml of 0.2% NaCl solutions were added in the boiling tube containing 90% phenol solution prepared by adding 9g of phenol in 1ml water.
 10. The miscibility temperatures for the mixture prepared at different concentrations of Phenol were found by repeating steps 4 to 6.

11. The same is repeated for different concentrations of NaCl and the data obtained is recorded.

Finding the Effect of KCl on the CST of the Phenol-Water System-

12. Preparation of 1% KCl Solution

- This was done in the same way as in step 7 but instead of NaCl, KCl was taken.
13. Steps 8-11 were followed for calculating the effect of KCl on the miscibility temperature of two partially miscible liquids (Phenol-Water).

Finding the Effect of NaCl and KCl Mixed Together on the CST of the Phenol-Water System-

14. Preparation of 1% NaCl solution and 1% KCl solution

- The 1% NaCl and 1% KCl solutions prepared as per aforementioned procedure.
15. Then to obtain 0.2% concentration of this solution, 1ml each of 1%KCl and 1%NaCl solution were added to 8ml water.
 16. Similarly, the other concentrations were obtained, i.e. 0.4% by adding 2ml each of 1%KCl and 1% NaCl solution to 6ml water, 0.6% by adding 3ml each of 1%KCl and 1%NaCl to 4ml water and henceforth.

Finding the Effect of Naphthalene on the CST of the Phenol-Water System-

17. Preparation of Naphthalene solution-

- Since Naphthalene is almost insoluble in water thus its solution cannot be prepared as per other solutions.
 - And if alcohol is used for dissolving than it interferes with the ternary system of Phenol-Water as both Phenol and water can dissolve in alcohol.
 - Therefore, to prepare 0.2% Naphthalene solution 0.02g of Naphthalene was weighed and added directly along with 10 ml water to solution in boiling tube containing 90% phenol solution.
 - Similarly the 0.4%, 0.6%, 0.8% solutions were also prepared.
18. The miscibility temperatures for the mixture prepared at different concentrations of Phenol were found by

repeating steps 4 to 6.

19. The same is repeated for different concentrations of Naphthalene solutions for calculating the effect of Naphthalene on the miscibility temperature of two partially miscible liquids (Phenol-Water) and the data obtained is recorded.

Finding the Effect of Camphor on the CST of the Phenol-Water System-

20. *Preparation of Camphor Solution-*

- Like Naphthalene, Camphor is also almost insoluble in water, thus it was used via the same procedure as Naphthalene.
21. The miscibility temperatures for the mixture prepared at different concentrations were found and the data obtained was recorded.
22. Steps 8-11 were followed for calculating the effect of Camphor on the miscibility temperature of two partially miscible liquids (Phenol-Water).

V. GENERAL OBSERVATIONS

At RTP, phenol is solid, white crystalline and its odor is like that of a disinfectant. But, the crystals were found slightly hydrated and discolored. Phenol has moderate solubility in water - about 8.5 g per 100 g of water. On dissolving more of phenol in water, white turbidity appears and two layers of liquid are observed; the upper is a solution of phenol in water, and the lower a solution of water in phenol. When NaCl is added to the phenol-water system, the solution immediately shows turbidity. Organic compounds are readily soluble in phenol.

VI. RAW DATA TABLES

Note: Not all tables could be provided due to the limitations of the format. These tables are provided to give a basic understanding of the data collection process to the reader.

Table I: Phenol-Water System

S.No.	Percentage of Phenol Solution used per trial (% ± 0.001%)	Temperature at which turbidity disappears (°C ± 0.25°C)			Temperature at which turbidity reappears (°C ± 0.25°C)		
		Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3
1.	75.00	30.0	29.5	30.5	12.0	11.4	12.6
2.	60.00	63.0	62.0	64.0	55.0	54.5	55.5
3.	50.00	72.0	70.0	74.0	62.0	61.0	63.0
4.	42.85	68.0	69.0	67.0	60.0	58.5	61.5
5.	37.50	61.5	61.0	62.0	60.5	59.5	61.5
6.	33.33	60.0	59.5	60.5	54.0	52.0	56.0
7.	30.00	53.0	55.0	51.0	51.0	52.5	49.5

Table II: Effect of 0.2% NaCl on the miscibility of Phenol-Water System

S.No.	Percentage of Phenol Solution used per trial (% ± 0.001%)	Temperature at which turbidity disappears (°C ± 0.25°C)			Temperature at which turbidity reappears (°C ± 0.25°C)		
		Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3
1.	90.00	65.0	64.0	66.0	57.0	56.5	57.5
2.	81.81	68.0	66.0	70.0	59.0	58.0	60.0
3.	75.00	71.0	69.5	72.5	60.0	61.5	58.5
4.	69.23	74.0	76.0	72.0	62.0	61.0	63.0
5.	64.28	72.5	73.0	72.0	61.5	61.0	62.0

6.	60.00	71.0	70.5	71.5	60.0	59.0	61.0
7.	56.25	69.0	70.0	68.0	59.0	58.0	60.0
8.	52.94	67.0	66.0	68.0	58.0	56.0	60.0
9.	50.00	66.0	65.5	66.5	56.0	55.0	57.0
10.	45.00	65.0	64.0	66.0	54.0	52.0	56.0

Table III: Effect of 0.2% KCl on the miscibility of Phenol-Water System

S.No.	Percentage of Phenol Solution used per trial (% ± 0.001%)	Temperature at which turbidity disappears (°C ± 0.25°C)			Temperature at which turbidity reappears (°C ± 0.25°C)		
		Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3
1.	90.00	64.0	65.0	63.0	61.0	62.0	60.0
2.	81.81	66.0	66.7	65.3	62.0	61.5	62.5
3.	75.00	67.0	67.0	67.0	63.0	63.0	63.0
4.	69.23	68.0	67.5	68.5	63.0	64.0	62.0
5.	64.28	70.0	71.0	69.0	62.0	61.0	63.0
6.	60.00	69.0	70.0	68.0	62.0	60.0	64.0
7.	56.25	68.0	68.5	67.5	62.0	61.5	62.5
8.	52.94	67.2	67.2	67.2	61.2	61.2	61.2
9.	50.00	66.0	65.5	66.5	60.0	60.0	60.0
10.	45.00	65.0	64.0	66.0	59.0	60.0	58.0

Table IV: Effect of 0.2% NaCl + KCl on the miscibility of Phenol-Water System Similarly, data was collected for Camphor and aphenalene too.

S.No.	Percentage of Phenol Solution used per trial (% ± 0.001%)	Temperature at which turbidity disappears (°C ± 0.25°C)			Temperature at which turbidity reappears (°C ± 0.25°C)		
		Trial 1	Trial 2	Trial 3	Trial 1	Trial 2	Trial 3
1.	90.00	66.0	67.0	65.0	61.0	61.5	60.5
2.	81.81	67.0	66.0	68.0	65.0	64.0	66.0
3.	75.00	68.5	68.0	69.0	65.5	66.0	65.0
4.	69.23	69.0	68.0	70.0	66.0	66.0	66.0
5.	64.28	70.0	69.0	71.0	62.4	61.4	63.4
6.	60.00	71.0	70.0	72.0	60.0	59.0	61.0
7.	56.25	70.5	70.0	71.0	57.5	57.0	58.0
8.	52.94	69.0	68.0	70.0	57.0	56.0	58.0
9.	50.00	68.0	67.0	69.0	56.0	55.0	57.0
10.	45.00	65.0	64.0	66.0	55.0	54.0	56.0

VII. PROCESSED DATA TABLES

To find the CST for each system created by different concentrations of impurities, a graph (using Vernier's *LoggerPro 3.8.6.1*) was plotted, taking the y-axis to be the miscibility temperatures and the concentration of impurities in the x-axis. The CST for each case was obtained from the highest peak of each graph. Further, the standard deviation was found for assessing the accuracy of the results. These calculations were done on an Excel Workbook. Uncertainty in T_1 and T_2 (ΔT) was $\pm 0.25^\circ\text{C}$. To calculate the uncertainty in the miscibility temperature (T_3), the following formula was used:

$$\begin{aligned} \text{Uncertainty in } T_3 &= \pm(2 \times \Delta T) \\ &= \pm(2 \times 0.25^\circ\text{C}) \\ &= \pm 0.5^\circ\text{C} \end{aligned}$$

The standard deviation for the data was calculated using the following formula:

$$\sigma = \sqrt{\frac{\sum(T - \bar{T})^2}{N - 1}}$$

Where T is the temperature recorded, \bar{T} is the average reading (T_1 or T_2), and N is the number of trials.

An example of standard deviation calculation is as follows:

From Raw Data Table 1, for 75% Phenol, Calculating the mean,

$$\begin{aligned} \bar{T} &= \left(\frac{30.0 + 29.5 + 30.5}{3} \right)^\circ\text{C} \\ \bar{T} &= 30.0^\circ\text{C} \end{aligned}$$

For calculating standard deviation,

$$\begin{aligned} \sum (T - \bar{T})^2 &= (30.0 - 30.0)^2 + (29.5 - 30.0)^2 \\ &\quad + (30.5 - 30.0)^2 \text{ } ^\circ\text{C}^2 \\ &= 0.25 + 0.25 \text{ } ^\circ\text{C}^2 \\ &= 0.5 \text{ } ^\circ\text{C}^2 \\ \sigma &= \sqrt{\frac{0.5}{3-1}} \text{ } ^\circ\text{C} \\ \sigma &= 0.5 \text{ } ^\circ\text{C} \end{aligned}$$

Table V: Phenol-Water System

Percentage of Phenol Solution used per trial (% = 0.001%)		75.00	60.00	50.00	42.85	37.50	33.33	30.00
Average Temperature Readings	Turbidity disappears (°C ± 0.25°C) (T ₁)	30.0	63.0	72.0	68.0	61.5	60.0	53.0
	Turbidity disappears (°C ± 0.25°C) (T ₂)	12.0	55.0	62.0	60.0	60.5	54.0	51.0
Standard Deviation	Turbidity disappears (°C ± 0.25°C)	0.50	1.0	2.0	1.0	0.5	0.28	2.0
	Turbidity disappears (°C ± 0.25°C)	0.60	0.28	1.0	1.25	0.76	2.0	1.75
Miscibility Temperature (°C ± 0.50°C) (T ₃)	$T_3 = \frac{T_1 + T_2}{2}$	21.0	59.0	67.0	64.0	61.0	57.0	52.0

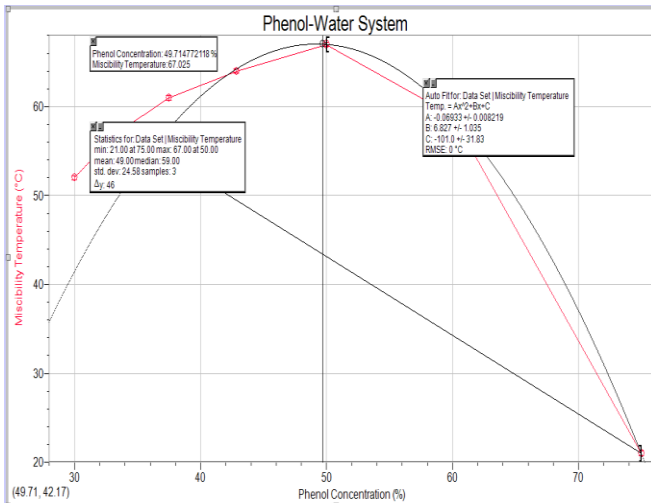


Fig. 1. Phenol Concentration vs. Miscibility Temperature for Phenol-Water System

Table VI: Effect of 0.2% NaCl on the miscibility of Phenol-Water System

Percentage of Phenol Solution used per trial (% = 0.001%)		90.0	81.8	75.0	69.2	64.2	60.0	56.2	52.9	50.0	45.0
Average Temperature Readings	Turbidity disappears (°C ± 0.25°C) (T ₁)	65.0	68.0	71.0	74.0	72.5	71.0	69.0	67.0	66.0	65.0
	Turbidity disappears (°C ± 0.25°C) (T ₂)	57.0	59.0	60.0	62.0	61.5	60.0	59.0	58.0	56.0	54.0
Standard Deviation	Turbidity disappears (°C ± 0.25°C)	1.0	2.0	1.5	2.0	0.5	0.5	1.0	1.0	0.5	1.0
	Turbidity disappears (°C ± 0.25°C)	0.28	1.0	1.5	1.0	0.5	1.0	1.0	2.0	1.0	2.0
Miscibility Temperature (°C ± 0.50°C) (T ₃)	$T_3 = \frac{T_1 + T_2}{2}$	61.0	63.5	65.5	68.0	67.0	65.5	64.0	62.5	61.0	59.5

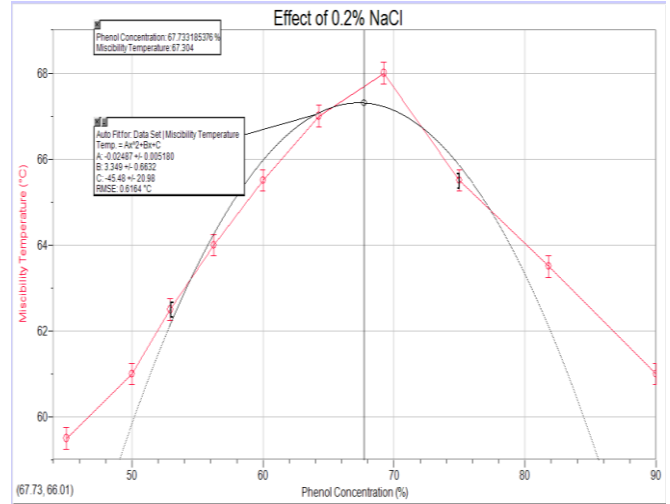


Fig. 2. Phenol Concentration vs. Miscibility Temperature with added 0.2% NaCl

From Fig. 2, we get that CST of phenol-water system with added 0.2% NaCl is 67.3°C.

Table VII: Effect of 0.4% NaCl on the miscibility of Phenol-Water System

Percentage of Phenol Solution used per trial (% = 0.001%)		90.00	81.81	75.00	69.23	64.28	60.00	56.25	52.94	50.00	45.00
Average Temperature Readings	Turbidity disappears (°C ± 0.25°C) (T ₁)	67.5	68.5	70.0	71.0	69.0	69.0	68.0	67.0	66.0	65.5
	Turbidity disappears (°C ± 0.25°C) (T ₂)	62.5	65.5	67.0	68.0	68.0	67.0	66.0	65.0	64.0	61.5
Standard Deviation	Turbidity disappears (°C ± 0.25°C)	0.5	0.5	0.0	1.0	1.0	0.5	0.76	1.0	2.0	1.0
	Turbidity disappears (°C ± 0.25°C)	1.5	0.5	1.0	0.0	0.0	2.0	0.5	1.0	0.5	0.5
Miscibility Temperature (°C ± 0.50°C) (T ₃)	$T_3 = \frac{T_1 + T_2}{2}$	65.0	67.0	68.5	69.5	68.5	68.0	67.0	66.0	65.0	63.5

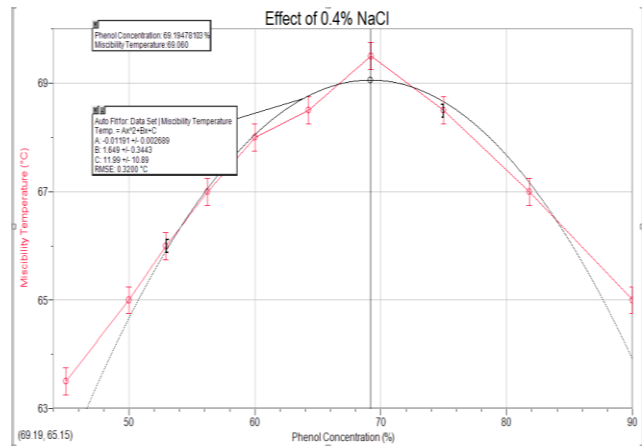


Fig. 3. Phenol Concentration vs. Miscibility Temperature with added 0.4% NaCl.

From Fig. 3, we get that CST of phenol-water system with added 0.4% NaCl is 69.0°C. Similarly, data was processed for all other impurities with varying concentrations.

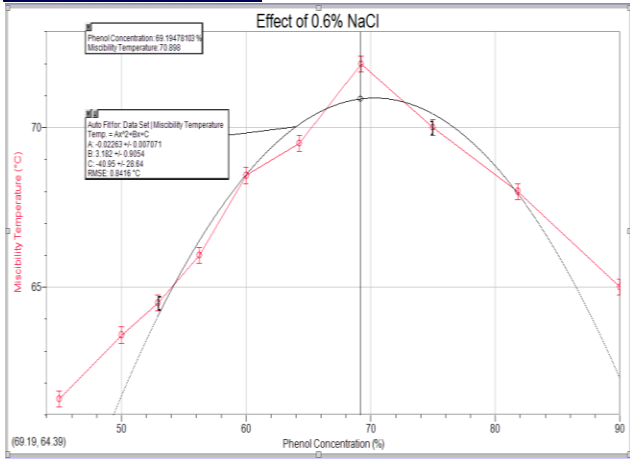


Fig. 4. Phenol Concentration vs. Miscibility Temperature with added 0.6% NaCl

From Fig. 4, we get that CST of phenol-water system with added 0.6% NaCl is 70.9°C.

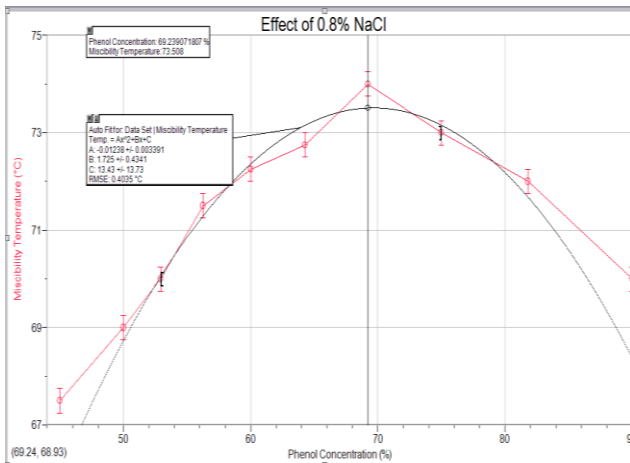


Fig. 5. Phenol Concentration vs. Miscibility Temperature with added 0.8% NaCl

From Fig. 5, we get that CST of phenol-water system with added 0.8% NaCl is 73.5°C.

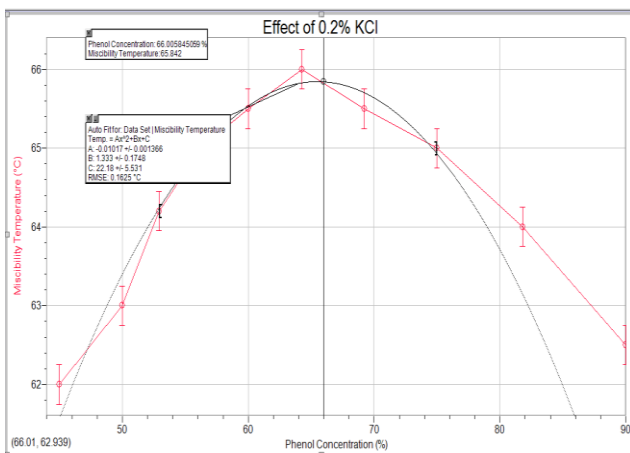


Fig. 6. Phenol Concentration vs. Miscibility Temperature with added 0.2% KCl

From Fig. 6, we get that CST of phenol-water system with added 0.2% KCl is 65.8°C.

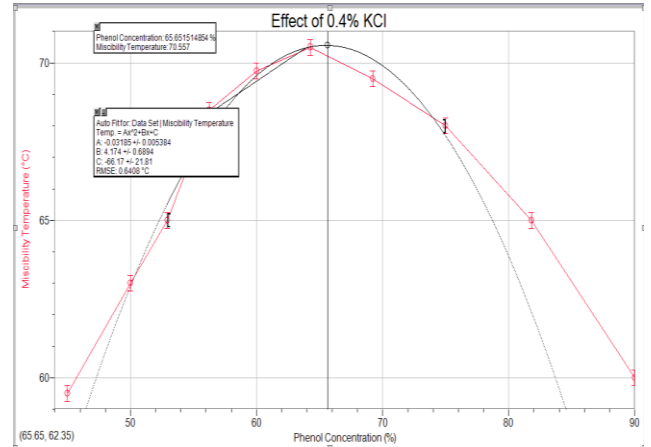


Fig. 7. Phenol Concentration vs. Miscibility Temperature with added 0.4% KCl

From Fig. 7, we get that CST of phenol-water system with added 0.4% KCl is 70.6°C.

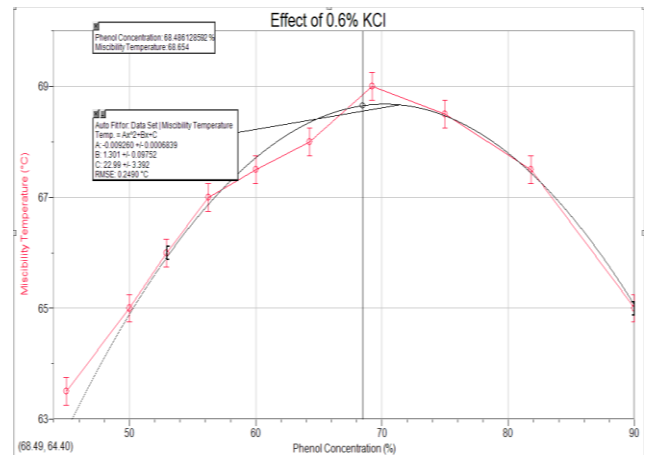


Fig. 8. Phenol Concentration vs. Miscibility Temperature with added 0.6% KCl

From Fig. 8, we get that CST of phenol-water system with added 0.4% KCl is 70.6°C.

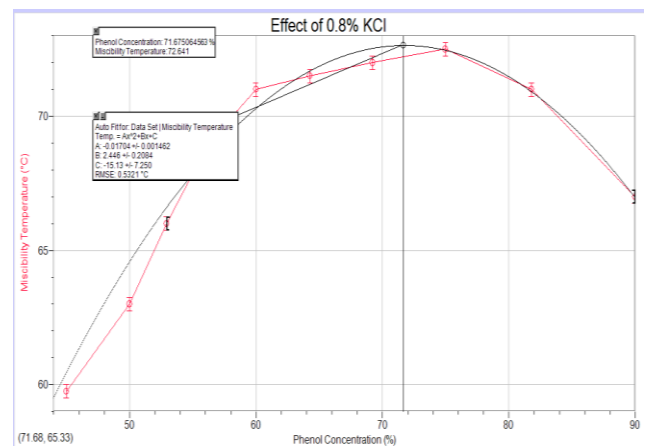


Fig. 9. Phenol Concentration vs. Miscibility Temperature with added 0.8% KCl

From Fig. 9, we get that CST of phenol-water system with added 0.4% KCl is 70.6°C.

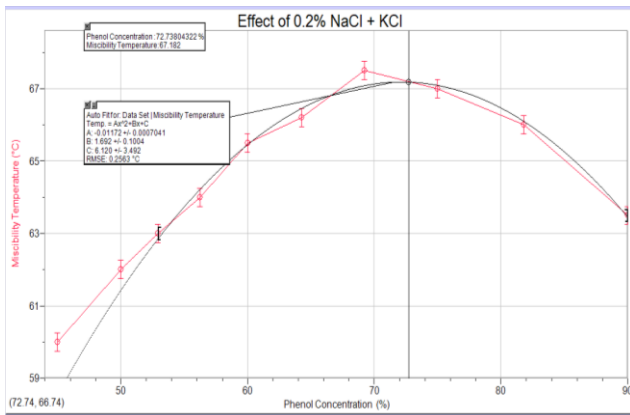


Fig. 10. Phenol Concentration vs. Miscibility Temperature with added 0.2% NaCl + KCl

From Fig. 10, we get that CST of phenol-water system with added 0.2% NaCl + KCl is 67.2°C.

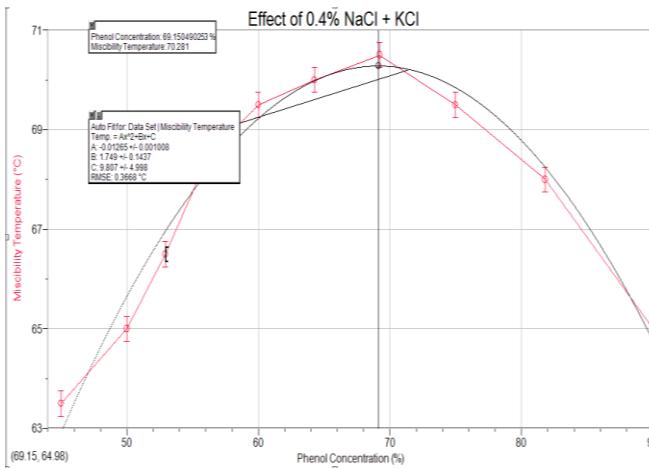


Fig. 11. Phenol Concentration vs. Miscibility Temperature with added 0.4% NaCl + KCl

From Fig. 11, we get that CST of phenol-water system with added 0.4% NaCl + KCl is 70.3°C.

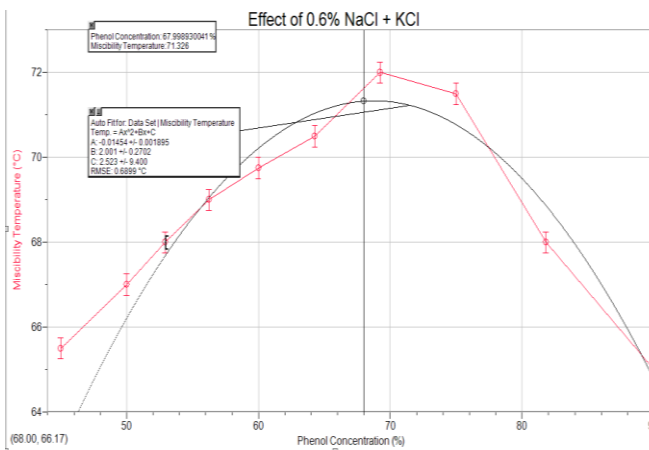


Fig. 12. Phenol Concentration vs. Miscibility Temperature with added 0.6% NaCl + KCl

From Fig. 12, we get that CST of phenol-water system with added 0.6% NaCl + KCl is 71.3°C.

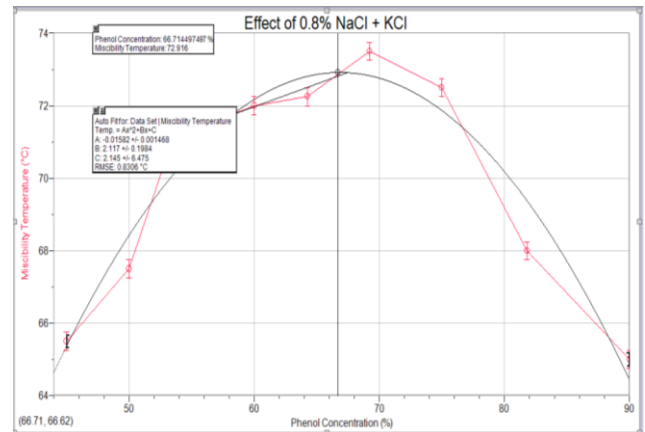


Fig. 13. Phenol Concentration vs. Miscibility Temperature with added 0.8% NaCl + KCl

From Fig. 13, we get that CST of phenol-water system with added 0.8% NaCl + KCl is 72.9°C.

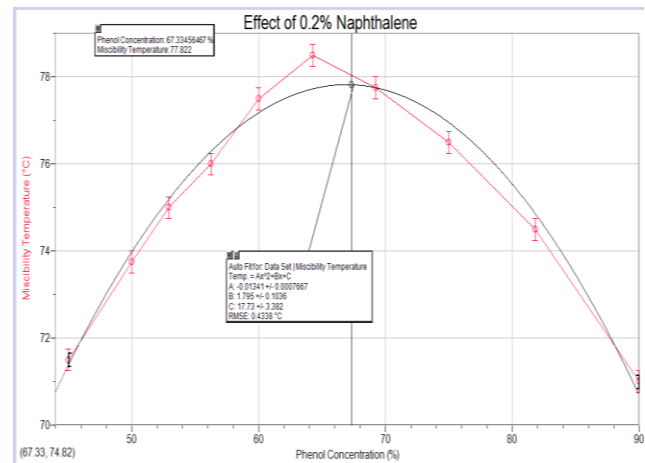


Fig. 14. Phenol Concentration vs. Miscibility Temperature with added 0.2% Naphthalene

From Fig. 14, we get that CST of phenol-water system with added 0.2% Naphthalene is 77.8°C.

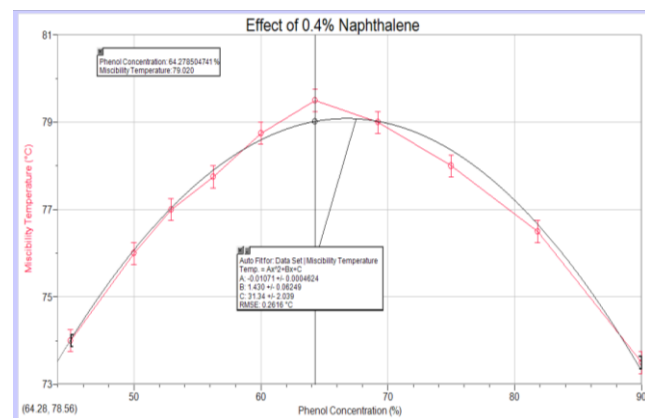


Fig. 15. Phenol Concentration vs. Miscibility Temperature with added 0.4% Naphthalene

From Fig. 15, we get that CST of phenol-water system with added 0.4% Naphthalene is 79.0°C.

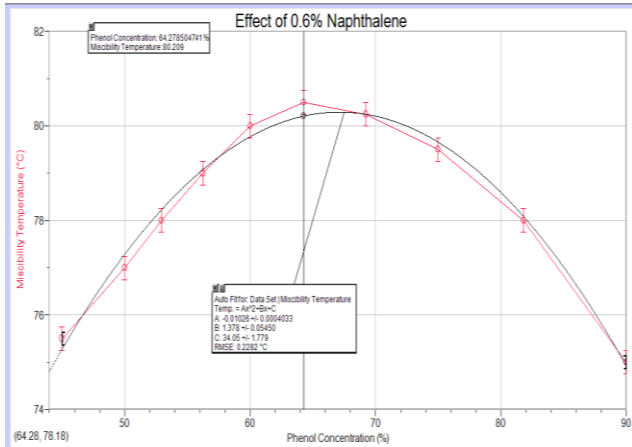


Fig. 16. Phenol Concentration vs. Miscibility Temperature with added 0.6% Naphthalene

From Fig. 16, we get that CST of phenol-water system with added 0.6% Naphthalene is 80.2°C.

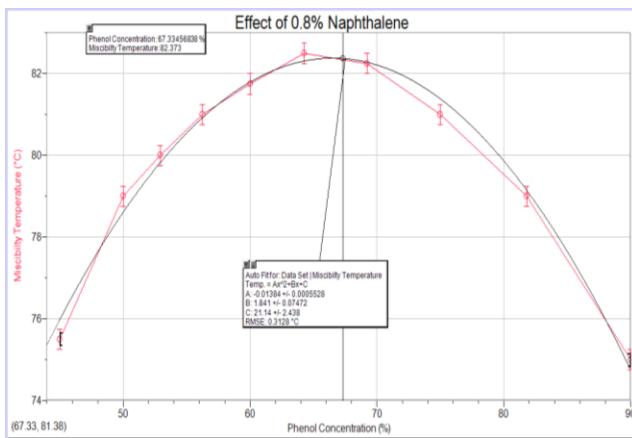


Fig. 17. Phenol Concentration vs. Miscibility Temperature with added 0.8% Naphthalene

From Fig. 17, we get that CST of phenol-water system with added 0.8% Naphthalene is 82.4°C.

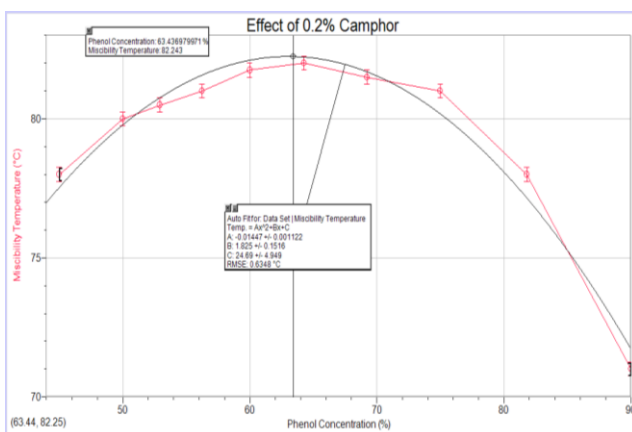


Fig. 18. Phenol Concentration vs. Miscibility Temperature with added 0.2% Camphor

From Fig. 18, we get that CST of phenol-water system with added 0.2% Camphor is 82.2°C.

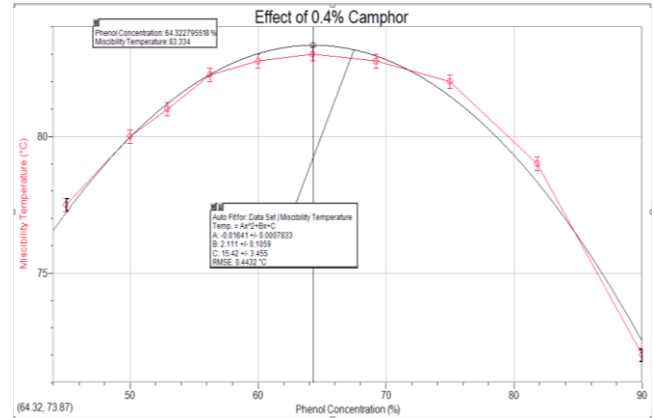


Fig. 19. Phenol Concentration vs. Miscibility Temperature with added 0.4% Camphor

From Fig. 19, we get that CST of phenol-water system with added 0.4% Camphor is 83.3°C.

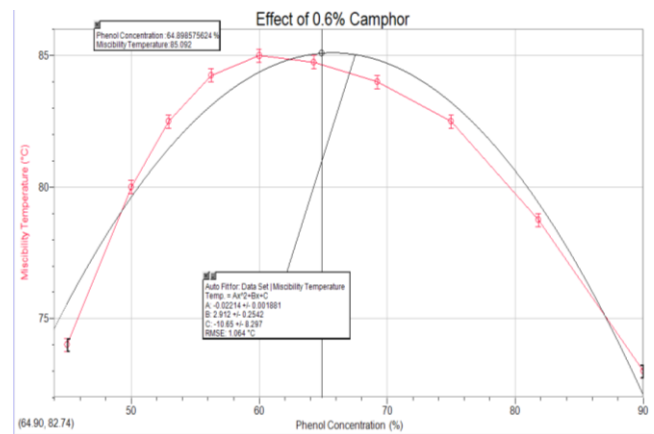


Fig. 20. Phenol Concentration vs. Miscibility Temperature with added 0.6% Camphor

From Fig. 20, we get that CST of phenol-water system with added 0.6% Camphor is 85.1°C.

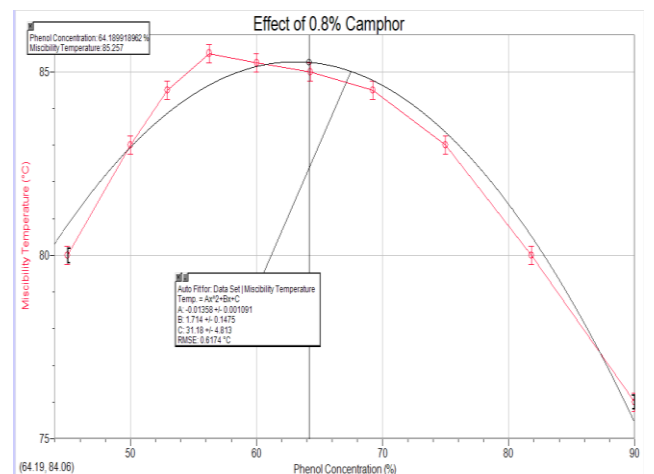


Fig. 21. Phenol Concentration vs. Miscibility Temperature with added 0.8% Camphor

From Fig. 21, we get that CST of phenol-water system with added 0.8% Camphor is 85.3°C.

VIII. ANALYSIS OF PROCESSED DATA

Throughout the analysis, uncertainty in temperature was 0.50°C. By analyzing the graphs and tables, it was found that when the concentration of phenol is decreased from 75% to 30%, the miscibility decreased and consequently, the miscibility temperature increased from 21°C to 67°C upto 50% concentration of phenol. After this point, as the concentration decreased, the miscibility temperature also decreased to 52°C (at 30% concentration of phenol), thus producing a nearly parabolic graph. The peak of which gave the CST of the system. The CST obtained for this pure phenol-water system was 67°C.

On adding impurities solutions of concentrations from 0.2% to 0.8% in the phenol water system with decreasing concentration of phenol (from 90% to 45%) the CST determined were:

S.No.	Concentration of added impurity (°C)→	0.2%	0.4%	0.6%	0.8%
	Name of added impurity ↓				
1.	Sodium Chloride (NaCl)	67.3	69.0	70.9	73.5
2.	Potassium Chloride (KCl)	65.8	70.6	68.7	72.6
3.	NaCl + KCl	67.2	70.3	71.3	72.9
4.	Naphthalene	77.8	79.0	80.2	82.4
5.	Camphor	82.2	83.3	85.1	85.3

Interpreting the data used for Fig. 2 to 5, when the concentration of NaCl solution added in 90% phenol-water solution changes from 0.2% to 0.8% the miscibility temperature also increases from 61°C to 70°C. The CST calculated for these concentrations of NaCl using the Fig. 2 to 5 show that as the concentration increases CST also increases. This is because the effect of an impurity on the CST of the system depends on whether the impurity is partially or completely soluble in one or both the components of the system and in this case, as NaCl is completely soluble in water whereas insoluble in phenol solution, it increased the UCST of the system, hence decreasing the miscibility.

Similarly, interpreting the data used for Fig. 6 to 9 from for KCl solution, the miscibility temperature also increases from 62.5°C to 67.0°C. The CST calculated for these concentrations of KCl using the Fig. 6 to 9, show that as the concentration increases CST also increases. Since KCl is also completely soluble in water whereas insoluble in phenol solution, it increased the UCST of the system, hence decreasing the miscibility.

The effect of organic substances added in the phenol water system, is also similar to that of ionic impurities, i.e. the miscibility temperature increases and the miscibility of the system decreases. Interpreting the data used for the Fig. 14 to 17, we see that when the concentration of naphthalene solution added in 90% phenol-water solution changes from 0.2% to 0.8% the miscibility temperature also increases from 71.0°C to 75.0°C. The CST calculated for these concentrations of naphthalene using the Fig. 14 to 17, show that as the concentration increases CST also increases.

Naphthalene increased the UCST of the system, hence decreasing the miscibility. This is because naphthalene is soluble only in phenol.

The data of Fig. 18 to 21 show that the same trend is followed by Camphor too, i.e. the miscibility temperature increases from 71.0°C to 76.0°C with the increase in concentration. This is because camphor is insoluble in water and soluble in phenol. The CST calculated for these concentrations of Camphor using the Graphs 18 to 21, show that Camphor increased the UCST of the system, hence decreasing the miscibility.

The miscibility temperature in the case of the mixture of NaCl and KCl also increased and hence reduced the miscibility of the system. Though the rise in temperature with concentration was not significant, it mostly fluctuated between the temperature values of 63.5°C to 65°C. Interpreting the data of Fig. 10 to 13, we see that when the concentration of NaCl and KCl increased, the miscibility temperature also increased. I expected these value readings to be distinct since NaCl and KCl are two ionic compounds that have a high lattice enthalpy. However, it can be noticed that the readings are close to those observed using NaCl and KCl used as separate impurities. Hence, it can be inferred that there wasn't much effect of combining these two compounds but it must be noticed that we can do further research in this using a mixture with different proportions of NaCl and KCl.

IX. CONCLUSION AND EVALUATION

The comparative graph below (Fig. 22) shows that the UCST of Phenol-Water system increases with increase in concentrations of impurities irrespective of their nature, but for same concentration of impurities the increase is more for organic impurities than ionic. At all concentrations, CST of phenol-water system containing Camphor as impurity are maximum and it increases with increase in concentration.

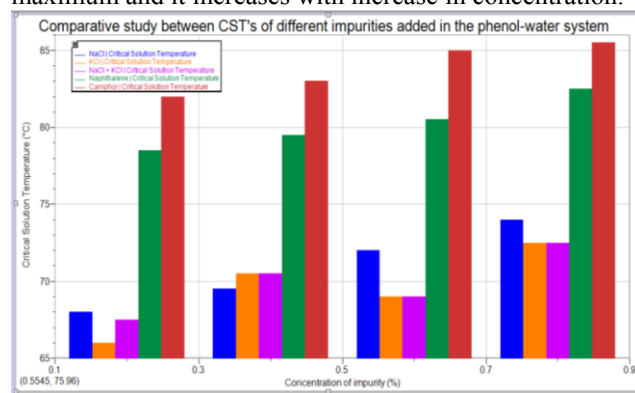


Fig. 22. Comparative Study between CSTs

CST of pure phenol-water was 67°C and with Camphor it varied from 82.2°C to 85.3°C at 0.2% and 0.8% concentrations. Thus, the increase in CST was about 15.2°C to 18.3°C. Similarly, for Naphthalene, CST increases by 10.8°C 15.4°C. But, for similar concentration of NaCl, the increase in CST was between 0.3°C to 6.5°C. And, for KCl, initially CST falls by 1.2°C at 0.2% concentration and then increased to 5.6°C at 0.8%. The fluctuation in this case may

be due to some experimental errors. CST of mixture of KCl and NaCl also showed an increase from 0.2°C to 5.9°C.

Thus, it can be concluded that the ionic compounds, which get hydrated with water, show lesser increase in CST as they decrease the miscibility to a lesser extent. The CST of KCl was lesser than that of NaCl since NaCl is more soluble. On the other hand, organic compounds, which dissolve in phenol (do not get hydrated with water), decrease the miscibility to a larger extent. Camphor decreases more since it is more soluble in phenol.

The standard deviation of the readings was noted over the three trials to measure the accuracy of the experiment performed. The standard deviation in all the data is between 0 and 2. Despite of trying to minimize all the errors, there might have been a few uncontrolled and unintended errors such as parallax errors. Uncertainties in the calculations, although calculated, came out to be too small to affect the results. Hence, the uncertainties in the concentration were neglected. For this experiment, all the solutions were made first and were then heated one by one, thus the other solutions were left in the open, and they might have undergone some changes due to atmospheric interactions. Also, due to the cold weather in the region where the laboratory is situated, temperature of disappearance and reappearance of turbidity might have been affected. Thus, in the ideal scenario, the solutions should be prepared one by one. The intensity of the flame of the Bunsen burner might have fluctuated during the heating of the solutions which might have caused an increase in the temperature measured by the thermometer. Considering the small inaccuracies, it can be argued that the experimental data came out to be of a good quality.

X. FURTHER SCOPE

This experiment can further be extended to find the miscibility of other partially miscible systems such as water-ether using ionic and organic substances. As said heretofore, mixtures of impurities with different compositions can be prepared to find their effect on the phenol-water CST. Different kinds of ionic salts of cations and anions having different size and charge can be used to determine the comparative effect on the CST. This can help in various pharmaceutical salting out processes. In place of UCST, impurities which affect the LCST (like succinic acid), can also be added and the results can be compared.

REFERENCES

- [1] Atkins, P. W., and Julio De Paula. "Equilibrium: Simple Mixtures." *Atkins' Physical Chemistry*. 8th ed. Oxford: Oxford UP, 2006. 180-82. Print.
- [2] Clugston, M. J., and Rosalind Flemming. "Chemical Equilibrium." *Advanced Chemistry*. Oxford: Oxford UP, 2000. 182-84. Print.
- [3] Vermerris, Wilfred, and Ralph L. Nicholson. "Chapter 7: Phenolic Compounds And Their Effects on Human Health." *Phenolic Compound Biochemistry*. Dordrecht, the Netherlands: Springer, 2006. 235-37. Print.
- [4] Britannica Eyclopedia. "Bakelite." *Encyclopedia Britannica Online*. Encyclopedia Britannica, Web. 15 Oct. 2015.
- [5] DeVoe, Howard. "Phase Diagrams: Ternary Systems." *UC Davis ChemiWiki*. UC Davis ChemiWiki, Web. 16 Oct. 2015.
- [6] Dhirendra, Singh. "Water-Phenol Miscibility Diagram." *Academia.org*. Academia.org, Web. 18 Oct. 2015.
- [7] Drugs.com. "Phenol Spray." *Drugs.com: Phenol Spray*. Drugs.com, 1 Jan. 2000. Web. 14 Oct. 2015.
- [8] Greener-Industry. "Phenol." *Greener-industry.org.uk*. Www.greener-industry.org.uk, Web. 15 Oct. 2015.
- [9] Jadhav, Rajendra S., Vishal Mane, Avinash V. Bagle, Dilip G. Hundiwale, Pramod P. Mahulikar, and Gulzar Waghoo. "Synthesis of Multicore Phenol Formaldehyde Microcapsules and Their Application in Polyurethane Paint Formulation for Self-healing Anticorrosive Coating." *International Journal of Industrial Chemistry Int J Ind Chem 4.1* (2013): 31. Springer. Web. 16 Oct. 2015.
- [10] Nelson, Stephen A., Prof. "Ternary Phase Diagrams." *Tulane University*. Prof. Stephen A. Nelson, 13 Jan. 2011. Web. 16 Oct. 2015.
- [11] PubChem. "Camphor." *PubChem*. PubChem, 25 Mar. 2005. Web. 18 Oct. 2015.
- [12] PubChem. "Naphthalene." *PubChem*. PubChem, 25 Mar. 2005. Web. 18 Oct. 2015.
- [13] Turtinen, Lloyd W., and Brian D. Juran. "Protein Salting-Out Method Applied to Genomic DNA Isolation from Fish Whole Blood." *Protein Salting-Out Method Applied to Genomic DNA Isolation from Fish Whole Blood* 24.2 (1998): 238-39. *BioTechniques*. Web. 16 Oct. 2015.
- [14] UA, Waterloo. "Solutions." *Science.uwaterloo.ca*. University of Waterloo, Web. 14 Oct. 2015.
- [15] Xamplified. "True Solution, Suspension and Colloidal Solution." *Colloidal Solution*. Xamplified, Web. 14 Oct. 2015.

AUTHOR'S PROFILE



Anmol Mathur is a Chemical Engineering student at the School of Chemical and Biomolecular Engineering of the Georgia Institute of Technology, Atlanta, GA, United States of America. He has the honors of Dean's List and is also a member of the American Institute of Chemical Engineers (AIChE). He also works as a Research Scientist in Naq Global Companies. He did his International Baccalaureate Diploma Programme from Jayshree Periwal International School, Jaipur, India. Over the years, he has researched in subjects ranging from industrial and organic chemistry to mathematical economics. With several patents to his name, he now specializes in technologies for the fertilizer industry and has worked in collaboration with research groups across the world to produce novel eco-friendly solutions for fertilizer quality improvement. LinkedIn: <https://www.linkedin.com/in/anmol-mathur-65545398>