

## Research Article



## Effect of the Critical Solution Temperature of a Partial Miscible Phenol-Water Solution with Addition of Potassium Chloride

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### ABSTRACT

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 the phase gets affected by the addition of impurities. To observe 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 phase was analyzed. It was found that addition of ionic compound KCl, in phenol- water system show lesser increase in CST as they decrease the miscibility to a lesser extent.

**Keywords:** Critical Solution Temperature, KCl, Phenol-Water System.

### INTRODUCTION

A homogenous mixture of a solute and a solvent is called as a true solution. The liquid-liquid are of three types such as completely miscible liquid pairs - E.g. Water-Alcohol, Water-Sulphuric acid. Partially miscible liquid pairs - E.g. Ether-Water, Phenol-Water and liquid pairs that are practically immiscible - E.g. Mercury-Water, Nitrobenzene-water<sup>1</sup>.

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 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<sup>2</sup> 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). 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. For a ternary system, there are three components co-existing in a system. 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. 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<sup>3, 4</sup>. 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.

### MATERIALS AND METHODS

#### Materials

Potassium chloride was obtained from Qualigens Fine Chemicals, India. Phenol was purchased from standard chemicals, Hyderabad, India and distilled water was collected from distillation unit from college.

#### Methods<sup>5</sup>

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

1. Phenol concentration scales of 8%, 15%, 40%, 55% and 80% were set in the experiment.
2. Five 20 mL of the mixture of phenol and water with the concentration scales were prepared in the test tubes.



3. The volume of phenol and water required were calculated by using the formula:-

Let X be the concentration scale of phenol:

Volume of phenol =  $20 \text{ mL} \times X/100$

Volume of water =  $20 \text{ mL} \times (100-X)/100$

4. The measured amount of water and phenol were transferred into a test tube.

5. The phenol transfer process must be done in the fume cupboard as the phenol is very toxic.

6. A thermometer was placed into the test tube and sealed by using parafilm followed by aluminium foil and it was to make sure that the thermometer did not touch the bottom surface of the test tube.

7. During the process of heating, the test tube was shaken gently in the water bath to increase the rate of speed of Average miscibility of temperature of solution (T) =

$\frac{\text{temperature at which turbidity disappears (Clear solution)} + \text{temperature at which turbidity reappears (turbid solution)}}{2}$

12. The graph of temperature against percentage of phenol was plotted to produce a phase diagram and the critical solution temperature was determined.

### Step II: Finding the Effect of KCl on the CST of the Phenol-Water system-

#### Preparation of KCl solution

1. One gram of potassium chloride (KCl) salt was weighed by an electronic weighing machine.

2. This was dissolved in 100 ml distilled water in a beaker to get 1% w/v KCl solution.

#### Calculation

Average temperature when clear ( $T_1$ ) =  $\frac{\text{temperature from set I} + \text{temperature from set II}}{2}$

Average temperature when turbid ( $T_2$ ) =  $\frac{\text{temperature from set I} + \text{temperature from set II}}{2}$

### RESULTS AND DISCUSSION

From the observed tables it was found that when the concentration of phenol was increased from 8 % to 40% the miscibility increased and consequently the miscibility temperature increased from 51 °C to 71°C. But further increased concentration of phenol 55% to 80% the miscibility decreased and consequently the miscibility

the dispersion of two liquids until a clear mixture was obtained.

8. The temperature at which the turbid liquid becomes clear ( $T_1$ ) was observed and recorded.

9. The test tube was removed from the hot water and it was cooled by applying ice bath until the liquid became turbid and the temperature at this point was immediately recorded ( $T_2$ ).

10. Similarly 15%, 40%, 55% and 80% of phenol concentration scales in the experiment were prepared.

11. The average miscibility temperature for each test tube at which two phases were no longer seen or at which two phases existed were determined.

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3. Similarly 0.2 %, 0.4 %, 0.6 %, and 0.8 % w/v of KCl solutions were prepared by adding 0.2, 0.4, 0.6 and 0.8 grams of KCl to 100 ml of distilled water respectively in 4 different 100 ml beakers, with the help of measuring cylinders.

4. From the prepared solution 5 ml of various w/v % KCl solutions were taken and added in the boiling tube containing different % phenol-water composition solution respectively.

5. The miscibility temperatures for the mixture prepared at different concentrations of phenol were found by repeating steps.

temperature decreased. Hence plotting graph temperature vs composition of phenol nearly parabolic graph is obtained. The highest miscibility temperature is known as CST. The CST obtained from pure phenol-water system was 67.5°C. All the observed values were reported in table 1 to 5.

**Table 1:** Phenol-Water System

Test Tube	Phenol		Water		Temperature when clear, ( $T_1$ ), °C			Temperature when turbid, ( $T_2$ ) °C			Average miscibility temperature ( $T$ ), °C	CST
	%	ml	%	ml	I	II	Average	I	II	Average		
A	8	1.6	92	18.4	54	50	52	49	47	48	50	67.5
B	15	3.0	85	17.0	56	54	55	55	51	53	54	
C	40	8.0	60	12.0	73	67	70	68	62	65	67.5	
D	55	11.0	45	9.0	70	66	68	62	58	60	64	
E	80	16.0	20	4.0	58	54	56	50	46	48	52	



**Table 2:** Effect of 0.2% KCl on the miscibility of Phenol-Water System

Test Tube	Phenol		Water		Temperature when clear, (T <sub>1</sub> ), °C			Temperature when turbid, (T <sub>2</sub> ), °C			Average miscibility temperature, °C	CST
	%	ml	%	ml	I	II	Average	I	II	Average		
A	8	1.6	92	18.4	54	52	53	51	47	49	51	68
B	15	3.0	85	17.0	58	54	56	54	50	52	54	
C	40	8.0	60	12.0	75	67	71	68	62	65	68	
D	55	11.0	45	9.0	69	63	66	64	56	60	63	
E	80	16.0	20	4.0	59	54	56	52	48	50	53	

**Table 3:** Effect of 0.4 % KCl on the miscibility of Phenol-Water System

Test Tube	Phenol		Water		Temperature when clear, (T <sub>1</sub> ), °C			Temperature when turbid, (T <sub>2</sub> ), °C			Average miscibility temperature, °C	CST
	%	ml	%	ml	I	II	Average	I	II	Average		
A	8	1.6	92	18.4	57	53	55	51	47	49	52	69
B	15	3.0	85	17.0	61	55	58	56	52	54	56	
C	40	8.0	60	12.0	76	66	71	72	62	67	69	
D	55	11.0	45	9.0	71	65	68	67	57	62	65	
E	80	16.0	20	4.0	60	52	56	54	50	52	54	

**Table 4:** Effect of 0.6% KCl on the miscibility of Phenol-Water System

Test Tube	Phenol		Water		Temperature when clear, (T <sub>1</sub> ), °C			Temperature when turbid, (T <sub>2</sub> ), °C			Average miscibility temperature, °C	CST
	%	ml	%	ml	I	II	Average	I	II	Average		
A	8	1.6	92	18.4	57	51	54	52	48	50	52	70
B	15	3.0	85	17.0	60	54	57	55	51	53	55	
C	40	8.0	60	12.0	77	69	73	73	61	67	70	
D	55	11.0	45	9.0	70	64	67	65	57	61	64	
E	80	16.0	20	4.0	62	52	57	55	51	53	55	

**Table 5:** Effect of 0.8 % KCl on the miscibility of Phenol-Water System

Test Tube	Phenol		Water		Temperature when clear, (T <sub>1</sub> ), °C			Temperature when turbid, (T <sub>2</sub> ), °C			Average miscibility temperature °C	CST
	%	ml	%	ml	I	II	Average	I	II	Average		
A	8	1.6	92	18.4	58	54	56	54	50	52	54	71
B	15	3.0	85	17.0	62	56	59	58	52	55	57	
C	40	8.0	60	12.0	76	70	73	72	66	69	71	
D	55	11.0	45	9.0	73	63	68	65	57	62	65	
E	80	16.0	20	4.0	61	51	56	57	51	54	55	

### Comparison

From the data comparison it was found that CST was affected by the concentration of potassium chloride. All the observed values were depicted in table 6.

**Table 6:** Comparison of CST with different concentration of KCl addition

Sl. No.	Concentration of KCl added (% w/v)	CST, °C
1	0	67.5
2	0.2	68
3	0.4	69
4	0.6	70
5	0.8	71

### CONCLUSION

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 mixed. Due to addition of potassium chloride there was increase in the critical solution temperature. It was observed that the UCST of phenol-water system increases with increase in concentrations of impurities irrespective of their nature.

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