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Investigation of Critical Solution Temperature (CST) of Phenol-Water system with addition of some foreign compounds

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Abstract:** Generally, solubility of partially miscible liquids is increases with increasing temperature and the temperature at which they are completely miscible is termed as the Critical Solution Temperature (CST) or Consolute Temperature. Above This temperature the phases of a system are completely miscible is known as Upper critical solution temperature(UCST) or Upper Consulate Temperature (UCT) and it gets affected by the addition of impurities. Phenol-Water system exhibits upper critical solution temperature and it is useful for testing the purity of the mixture. Addition of foreign compound causes an increase in the critical solution 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 mixed. These two layers become more soluble with the increase in temperature and at a certain temperature they are completely miscible. This temperature is known as the CST of phenol-water system. It was observed that the UCST of phenol-water system increases with increase in concentrations of impurities irrespective of their nature. 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 were prepared and their effect on the phase was analyzed. It was found that addition of ionic compounds like KI, K_2CrO_4 in phenol- water system show lesser increase in CST as they decrease the miscibility to a lesser extent. On the other hand, addition of organic compounds like Cyclohexane, Acetone in phenol- water system show larger increase in CST as they decrease the miscibility to a larger extent.

***Keywords:** Critical Solution Temperature, Phenol-Water System, Potassium Iodide(KI), Potassium chromate(K_2CrO_4), Acetone, Cyclohexane.*

I. INTRODUCTION

A homogenous mixture of a solute and a solvent is known as a true solution. The liquid-liquid type solutions are of three types i.e. 1. completely miscible liquid pairs - E.g. Water-Acetic acid, Water-Ethylalcohol. 2. completely immiscible liquid pairs - E.g. Oil-Water, Nitrobenzene-water. 3. Partially miscible liquid pairs - E.g. Phenol-Water, Aniline-cyclohexane and Methanol-carbon disulphide[1]. Particularly its partial miscibility with water. partially miscible liquids become more soluble with the increase in temperature and at a certain temperature they are completely miscible[2]. This temperature is known as the critical solution temperature (CST). The temperature above which the phases of a system are completely miscible is known as 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) and it gets affected by the addition of impurities and it gets affected by the addition of impurities[3, 4].

The properties of Phenol have been studied for past decades due to their immense use in production of polycarbonates, as antiseptic, in paint strippers, chemically resistant coatings, in the aviation industry,in the manufacture of cosmetics like sunscreens, hair color, epoxies, Bakelite, nylon, detergents, herbicides such as phenoxy herbicides, and numerous pharmaceutical drugs for the formation of microcapsules and in polymer coating methods[5-7]. 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.

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These two liquids become more soluble with the increase in temperature and at a constant temperature these are completely miscible is called miscibility temperature [8-11]. 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[12-17]. It was found that addition of ionic compounds(KI, K_2CrO_4) in phenol- water system show lesser increase in CST as they decrease the miscibility to a lesser extent. On the other hand, addition of organic compounds (Cyclohexane, Acetone) in phenol- water system show larger increase in CST as they decrease the miscibility to a larger extent[18,19].

II. MATERIALS AND METHODS

A. Materials

All chemicals used in this experiments are of AR grade fine chemicals and distilled water was collected from distillation unit from our organisation.

B. Methods

1) *Procedure for Calculating the CST of a Phenol- Water System (Without Adding Foreign Compound):* Two cleaned burettes were taken, one is filled with phenol and the other one is with distilled water. Prepared four sets of mixtures of phenol and water as shown in table-1 and taken into four different test tubes designated A,B,C and D respectively. A hard glass boiling tube was thoroughly cleaned and dried and taken mixture A into it . Fixed a wire stirrer and a thermometer in the boiling tube through the cork and placed the boiling tube in the water bath. Experimental setup was shown in the Figure. The phenol-water mixture in the tube formed into two layers. The mixture was thoroughly mixed with the wire stirrer, a turbidity(cloudiness) produced in the tube. The water bath was heated and the mixture was stirred at regular intervals of time. The temperature was noted when the turbidity disappeared completely(shown in Figure) and the burner was turned off. The set up was allowed to cool down and noted the temperature when the turbidity reappeared (shown in Fig.3). The average of the two temperatures will be the miscibility temperature of the mixture. Similarly, In the same manner the miscibility temperature for each mixture was recorded in Table 2. Now the four mixtures A,B,C an D were diluted with 10 ml of distilled water each to give four more mixtures E,F,G and H having 40,36,32 and 28 % phenol respectively. The miscibility temperature for these mixture was calculated as mentioned above and recorded in Table 2.

Table 1. Composition of mixtures

Mixture	A	B	C	D
80% Phenol Soln.(in ml)	9	8	7	6
Distilled Water(in ml)	1	2	3	4
% of Phenol	72	64	56	48

Table 2. Miscibility temperatures of phenol-water system(without adding Foreign compound)

S.No.	Mixture	% composition of phenol	Miscibility temperature (in ^{0}C)		
			Turbidity disappears	Turbidity re-appears	Average
1	A	72	55.0	50.0	52.5
2	B	64	61.0	56.0	58.5
3	C	56	67.0	61.0	64.0
4	D	48	69.5	64.5	67.0
5	E	40	66.5	60.5	63.5
6	F	36	63.0	56.0	59.5
7	G	32	59.5	52.5	56.0
8	H	28	57.5	49.5	53.5

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2) *Finding the Effect of Foreign Compound on the CST of the Phenol-Water System:* One gram of a foreign compound was weighed and dissolved in 100 ml distilled water in a beaker to get 1% (w/v) solution of respective compound. By taking this 1% (w/v) solution and phenol eight different mixtures of different compositions were prepared as mentioned in 2.2.1. The miscibility temperature of the respective mixtures were determined and tabulated in the following.

Table 3. Miscibility temperature of phenol-water system with 1%(w/v) Potassium Iodide

S.No.	Mixture	% composition of phenol	Miscibility temperature (in ^{0}C)		
			Turbidity disappears	Turbidity re-appears	Average
1	A	72	56.5	51.5	54.0
2	B	64	62.5	57.5	60.0
3	C	56	68.5	62.5	65.5
4	D	48	71.0	67.0	69.0
5	E	40	67.5	61.5	64.5
6	F	36	63.5	57.5	60.5
7	G	32	61.0	54.0	57.5
8	H	28	58.5	51.5	55.0

Table 4. Miscibility temperature of phenol-water system with 1%(w/v) Potassium chromate

S.No.	Mixture	% composition of phenol	Miscibility temperature (in ^{0}C)		
			Turbidity disappears	Turbidity re-appears	Average
1	A	72	57.5	53.5	55.5
2	B	64	64.0	59.0	61.5
3	C	56	70.0	64.0	67.0
4	D	48	73.5	67.5	70.5
5	E	40	67.5	63.5	65.5
6	F	36	65.5	59.5	62.5
7	G	32	62.5	55.5	59.0
8	H	28	60.5	52.5	56.5

Table 5. Miscibility temperature of phenol-water system with 1% (w/v) Acetone

S.No.	Mixture	% composition of phenol	Miscibility temperature (in ^{0}C)		
			Turbidity disappears	Turbidity re-appears	Average
1	A	72	66.5	62.5	64.5
2	B	64	72.0	67.0	69.5
3	C	56	79.0	73.0	76.0
4	D	48	82.5	76.5	79.5
5	E	40	78.5	72.5	75.5
6	F	36	75.0	68.0	71.5
7	G	32	73.5	64.5	68.0
8	H	28	69.5	61.5	65.5

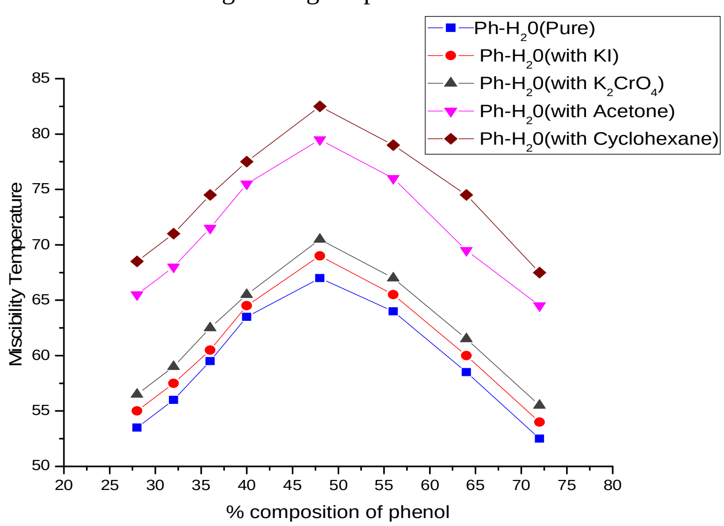
Table 6. Miscibility temperature of phenol-water system with 1% (w/v) Cyclohexane

S.No.	Mixture	% composition of phenol	Miscibility temperature (in ^{0}C)		
			Turbidity disappears	Turbidity re-appears	Average
1	A	72	69.5	65.5	67.5
2	B	64	76.5	72.5	74.5
3	C	56	82.5	75.5	79.0
4	D	48	84.5	80.5	82.5
5	E	40	80.5	74.5	77.5
6	F	36	78.0	71.0	74.5
7	G	32	74.5	67.5	71.0
8	H	28	71.5	65.5	68.5

III. 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. The presence of a foreign compound dissolved in one or both of the phases changes the CST values as well as the liquid phase composition at CST. Substances soluble in only one of the liquids raise the upper CST . It was observed that the UCST of phenol-water system increases with increase in concentrations of impurities irrespective of their nature. CST of pure phenol-water was 67ºC and with adding Cyclohexane it varied Thus, the increase in CST was about 15.5ºC. Similarly, with Acetone the increase in CST was about 12.5ºC. But, for similar concentration of KI, the increase in CST was about 2 ºC and for K_2CrO_4 the increase in CST was about 3.5 ºC. 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. On the other hand organic compounds which dissolve in phenol decrease the miscibility to a larger extent.

The addition of a foreign substance to a binary liquid system produces a ternary 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' and is used to select the best solvent for the manufacturing of drugs in pharmaceutical industries.



Graph-1: comparision graph of experimental results

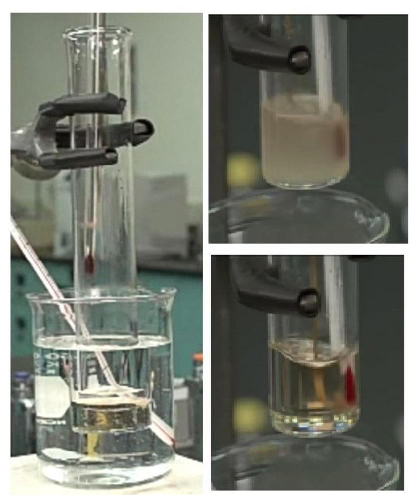


Figure-1: Experimental setup of CST

IV. FURTHER OUTLOOK

This experiment can be further extended to find the miscibility Temperatures of other partially miscible systems such as Aniline-cyclohexane and Methanol-carbon disulphide using ionic and organic substances. This can be helpful for salting out processes in drug manufacturing.

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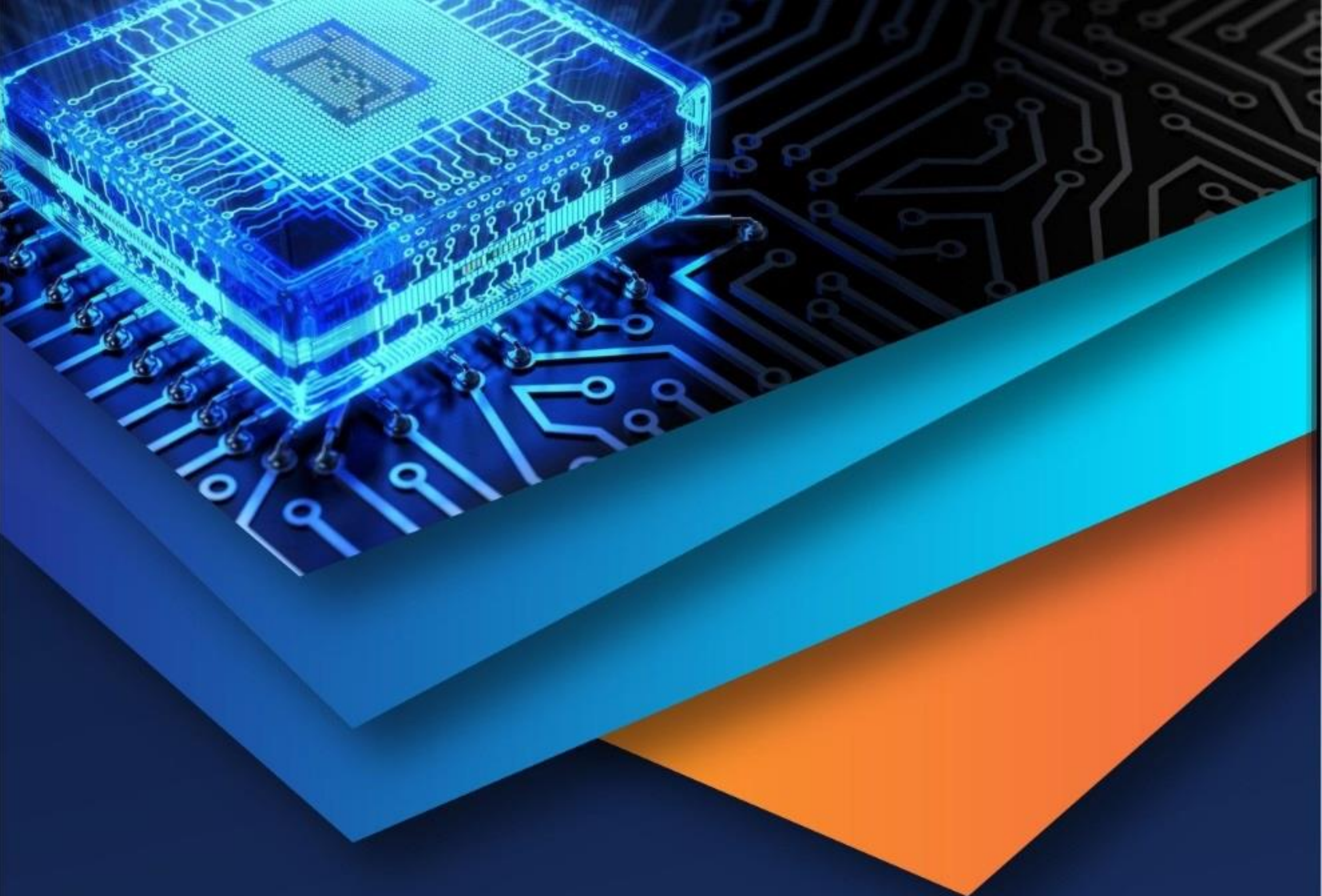

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