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Research Article

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A study on Krafft temperature of cetylpyridinium bromide in aqueous and mixed solvent systems

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ABSTRACT

Krafft Temperature determination for cationic surfactant Cetylpyridinium bromide(CPBr)had done in aqueous and 5%, 10%, 15% & 20% Ethylene Glycol-water systems. To study the krafft temperature, conductance measurements were used and the concentration of Cetylpyridinium bromide taken is above its CMC value. The temperature at which the values of conductance have an abrupt change is considered as the Krafft Temperature .There are two changes in the slope of the line is observed in the plots of specific conductance versus temperature in all the cases studied. The first slope change is highly abrupt compared to the second one. This interesting observation indicates the formation of premicellar aggregates of Cetylpyridinium bromide before attaining the Critical Micelle Concentration in aqueous and in mixed solvent systems of ethylene glycol and water which support the previous finding of premicellar aggregates formation during CMC determination. The author has previously reported the micellization of Cetylpyridinium bromide in aqueous and in mixed solvent systems of ethylene glycol and water. Again, it is observed that the krafft temperature of Cetylpyridinium bromide in aqueous medium is less than that of it in the mixed solvent systems of ethylene glycol and water.

Key words: krafft temperature, Cetyl Pyridinium Bromide (CPBr), Ethylene glycol, mixed solvent systems.

INTRODUCTION

Surfactant systems have been widely investigated and have variety of applications in various fields[1-8]. Various researchers have studied the krafft temperature and properties of cationic surfactants in aqueous medium[9-15]. The role of solvent in the formation of micelle in surfactant solution is important in the point of view of fundamental study and applied fields . The knowledge of the physical properties and thermodynamics of micellization of surfactant in solvent mixtures may be useful to select appropriate surfactant system of specific utility[16-18]. The minimum concentration of the surfactant at which the micellization starts is known as the critical micelle concentration (CMC) and Krafft temperature is the minimum temperature at which micelle formation occurs in surfactant solution. Below the Krafft temperature, the surfactant molecules remain as monomers and at the Krafft temperature, micelles formation starts and above the Krafft temperature, the surfactant remains in micelle form as Krafft temperature is a point of phase change. Cetylpyridinium bromide (C_{21}-H_{38}-N.Br) is a cationic surfactant having various applications as use in mouthwashes, as a disinfectant, as a germicide, deodorant, laboratory reagent, surfactant, and topical antiseptic and is also frequently used in analytical and physical chemistry [19]. In the present study the krafft temperature of Cetylpyridinium bromide in aqueous and in mixed solvent systems of water and 5%,10%,15% &20% ethylene glycol has been determined using conductance measurements.

EXPERIMENTAL SECTION

The cationic surfactant Cetylpyridinium bromide was obtained from SD fine chem. Limited. Ethylene glycol was purchased from Merck specialties private limited and used without purification. All other chemicals used were of Guaranteed Reagent grade. In order to determine the Krafft temperature of Cetylpyridinium bromide(CPBr), 100ml of aqueous and mixed solvent solutions of CPBr of concentration more than its CMC was prepared and kept in refrigerator for 24hrs. The temperature of the precipitated system was slowly increased with constant stirring and conductance was measured by conductivity meter . Plotting the graph between conductance and temperature showed an abrupt change in slope [20-21].Before starting the experiment, the conductivity cell was calibrated using KCl solution . The errors in the conductance measurements were within $\pm$ 5 %. The conductance was measured after thorough mixing and after attaining temperature equilibrium .

RESULTS AND DISCUSSION

Krafft temperature or krafft point is the temperature at which the solubility of surfactant starts to increase dramatically. At this temperature, the solubility of the surfactant becomes more to form micelles. Below this temperature, no micelles will form, no matter how much surfactant monomers are added. The krafft temperature depends on the head group and counter ion of the surfactant. The krafft temperature of CPBr in aqueous and in mixed solvent systems of water and ethylene glycol (EG) has been determined and the results are shown in **Table 1**, **figure 1, 2**, **3, 4 & 5 and Table-2**

Table- 1

	Specific Conductance (mS/cm)				
Temperature (°C)	In Aq. medium	In 5% EG	In 10% EG	In 15% EG	In 20% EG
-3	-	-	.43	-	-
-2	-	.77	.62	.47	-
-1	-	.80	.67	.50	-
0	-	.82	.68	.53	.42
1	.55	.85	.71	.55	.43
2	.58	.88	.74	.57	.45
3	.86	.91	.77	.59	.47
4	.90	.93	.79	.61	.49
5	.94	.95	.82	.63	.50
6	.96	.98	.85	.65	.51
7	1.00	1.01	.87	.67	.52
8	1.02	1.04	.89	.68	.53
9	1.05	1.07	.90	.70	.54
10	1.08	1.10	.92	.71	.55
11	1.11	1.12	.94	.72	.56
12	1.14	1.15	.95	.73	.57
13	1.17	1.18	.97	.75	.59
14	1.20	1.22	1.02	.81	.64
15	1.23	1.29	1.09	.86	.68
16	1.26	1.36	1.15	.91	.71
17	1.29	1.41	1.20	.94	.74
18	1.32	1.51	1.25	.98	.77
19	1.35	1.55	1.30	1.02	.81
20	1.38	1.59	1.34	1.05	.85
21	1.41	1.63	1.38	1.09	.87
22	1.44	1.66	1.42	1.12	.90
23	1.46	1.70	1.46	1.15	.92
24	1.49	1.74	1.48	1.18	.93
25	1.52	1.78	1.52	1.31	.97
26	1.54	1.82	1.54	1.32	.99
27	-	1.86	-	1.45	1.00
28	-	-	-	1.50	1.02

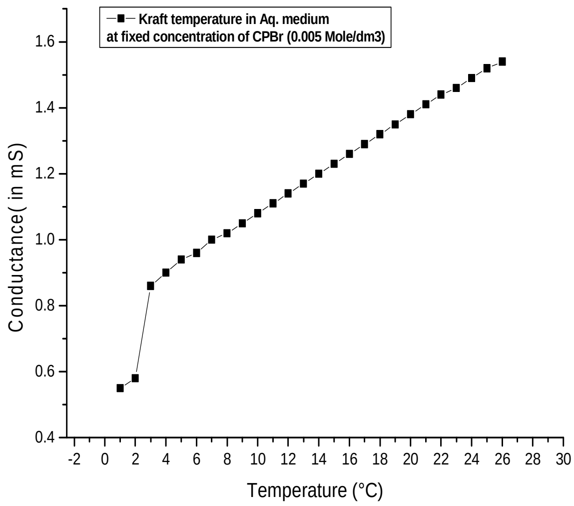

Figure 1:Krafft temperature in aqueous medium

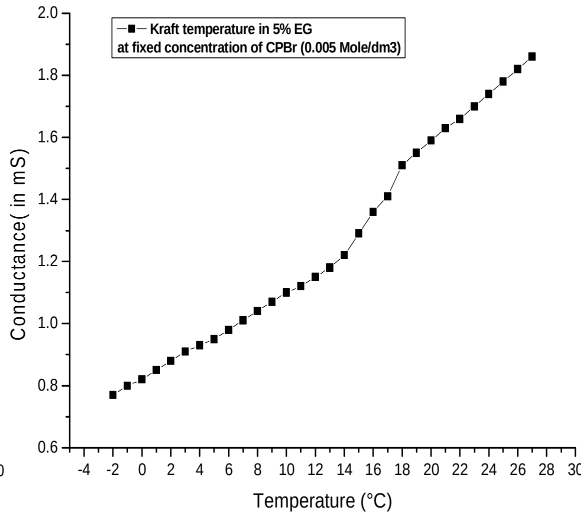

Figure 2:Krafft temperature in 5% EG

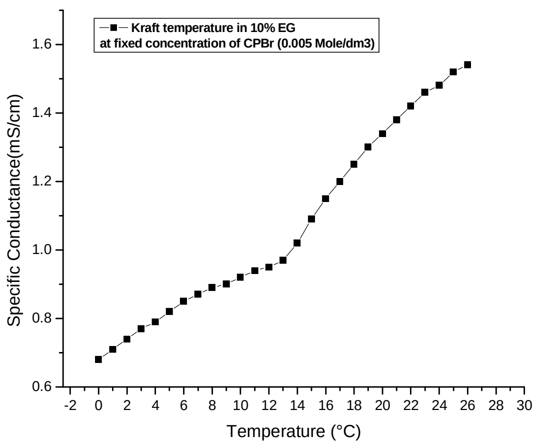

Figure 3:Krafft temperature in 10% EG

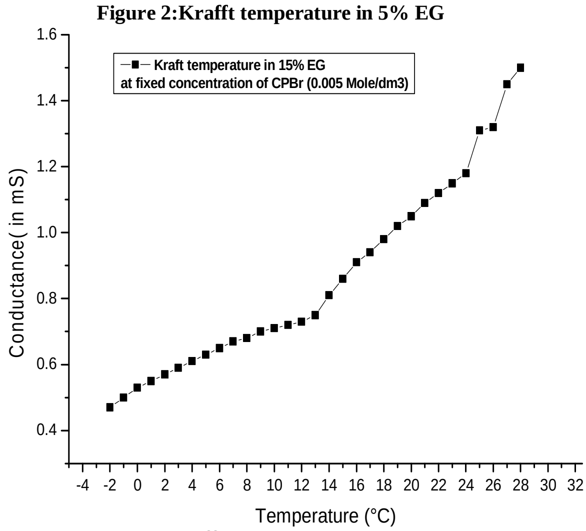

Figure 4:Krafft temperature in 15% EG

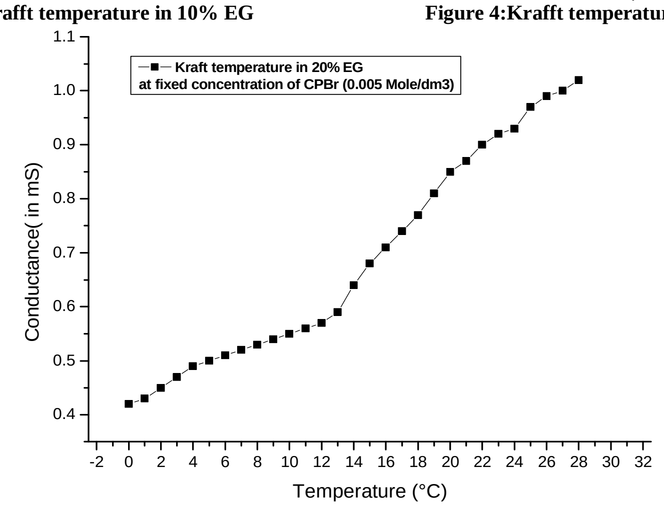

Figure 5:Krafft temperature in 20% EG

Table-2

Solvent system	First Krafft Temp. (KT_1, For pre-micellar Aggregates)℃	Second Krafft Temp. (KT_2)℃
Aqueous	2	7
5%EG	13	19
10% EG	13	23
15% EG	13	23
20%EG	13	23

The close observation of the plots of conductance versus temperature reveals that in each case of the systems studied,there are two abrupt changes of slopes of the line. In the case of aqueous solution of CPBr,the first breakpoint is observed at atemperature of 2℃ and the other one at around 7℃ . In the case of 5% ethylene glycol(EG) system, the first abrupt change in slope is observed at atemperature of 13℃ and the second one at 19℃ . In the case of 10% EG, the first and second breakpoints are at 13℃ and 23℃ . Similarly for 15% EG and 20%EG it is at 13℃ and 23℃ . The change of slope of the first breakpoints of all the cases studiedare highly abrupt compared to the second slope change.

CONCLUSION

Krafft Temperature determination for Cetylpyridinium bromide had done in aqueous and 5%,10%,15%,&20% ethylene glycol-water systems. From the results and plots of specific conductance versus temperature, it is observed that, in all the cases there are two slope changes of the line. The first slope change is highly abrupt compared to the seconed one in all cases studied.This interesting observation indicates the formation of premicellar aggregates of Cetylpyridinium bromide before attaining the Critical Micelle Concentration.In aqueous system the pre-micellar aggregate formation starts at a temperature of 2℃ and complete micelle formation occurs at 7℃.So in the present study the krafft temperature ofCetylpyridinium bromide in aqueous systemis found to be 7℃. One more interesting observation is,on changing the medium from aqueous to mixed solvent system of EG and water, the temperature at which premicellar aggregate formation starts increases from 2℃ to13℃ and the krafft temperature increases from 7 to 19 in the case of 5% and to 23℃ in the remaining cases studied. In the previous study of determination of CMC of Cetylpyridiniumbromide , the author has discussed about the formation of premicellar aggregates of Cetylpyridinium bromide in aqueous medium and in 5%, 10%, 15%,& 20% ethylene glycol-water systems. But in pure ethylene glycol medium premicellar aggregates formation was not observed. The solvent properties play a major role in CMC of a surfactant. Among various factors affecting CMC, the hydrophobic effect of hydrocarbon chain, the steric repulsions of the head group and the electrostatic interactions of polar head groups (for ionic surfactants) play great roles in micellization.The Krafft Temperature depends upon nature of solvent, size of head group ,andcounterion[22]..

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