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Investigation of colorimetric and differential thermal analysis of rubidium soaps

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ABSTRACT

Colorimetric analysis of Rubidium soaps solution of alkanols (laurate, stearate) in presence of thioflavin T indicates about the ease of detection of metal ion. Beer –Lamberts' law is quite successful for the relationship between the optical density and soap concentration over wide range. The critical micelles concentration (c.m.c) of rubidium soap solution of alkanols in presence of Thioflavin T is in quite agreement with the values obtained from the study of other physical properties .Rubidium soaps (laurate, stearate) exhibit exotherm and endotherm through differential thermal analysis. The Exotherm and endotherm indicate the crystallization, melting and decomposition states of these soaps ie there exist three temperatures, temperature of crystallization, melting and decomposition.

Keywords: Colorimetric, Decomposition, Endotherm, Crystallization, Indicate, Temperature.

1. INTRODUCTION

Metallic soaps structure and physico-chemical character due to amphiphilic nature are imperative for investigation and these proved useful in industry, agriculture and medicine as catalyst, inhibitor, fungicide, pesticide, preservative of wood, metallurgical process, greases, intended to improve flow, coating smoothness and thus possess indelible position for human culture.

Mesophase and glass formation in binary systems of caesium and alkali-earth metal butyrates was investigated[1]. Phase diagram of mesogenic binary system of cobalt (II) and univalent metal octanoates was studied[2]. The phase behavior of lithium stearate in cetane and in decalin was investigated[3]. Differential thermal analysis of metal soaps was carried out[4].

Mesophase and glass formation in binary systems of thallium butyrate with magnesium, calcium or zinc butyrate was investigated[5]. Analytical methods like gravimetric, polarography, volumetric and spectrographic are proposed for studying metal in traces. Apart from these colorimetric analyses are more reliable to detect metal ion in low concentration. Research workers have tried to establish that metal ion content can be detected successfully in organic solvents even in traces with colorimetric investigation[6-10].

2. EXPERIMENT

2.1. Material and Method

The chemicals of BDH/Analar were used in the experiment. The soaps were prepared, purified, dried and characterized as in article[11].

2.2.Differential thermal analysis

Differential thermal analysis of rubidium soaps Laurate, Stearate was carried out on a Stantanthermobalancce using alumina as a reference material. The heating rate was 7.5°C /min. The reproducibility of the measurements was checked by repeating the measurements.

2.3. Colorimetry

The absorption measurements in the region of 300-550 mμ were carried out with Hilgers' Unispec H700 photoelectric spectrophotometer. A Bausch and Lomb 'Spectronic 20' colorimeter with a red filter and I P -40 photo tube was used for absorption measurements in the longer wavelength region

(600-900 mμ). The reproducibility of the measurements was checked by repeating the measurements.

The absorption spectra of Thioflavin T with and without rubidium soaps in non-aqueous solution were taken in order to select the wave length to perform the experiment. After determining the maxima, absorption value for soap solutions of different concentration containing the same amount of Thioflavin T at the wavelength, where maxima existed were determined

3. RESULTS AND DISCUSSION

3.1. Differential Thermal Analysis

The results of differential thermal analysis of rubidium soaps laurate and stearate are shown in Fig.1 and the corresponding endotherm and exotherm observed are shown table 1

The exotherm at 20^0C in the differential thermal analysis curve of laurate indicates the crystallization of the soap. The endotherm at 210^0 C corresponds to the melting of the soap. The linear portion of the curve between 210^0C to 280^0C shows that the temperature of the sample and furnace remain the same and the soap decomposes insignificantly during this range of temperature. The endotherm at 355^0 C along with the shoulder endotherm at 370^0C corresponds to the boiling of the soap. The exotherm at 380^0 C indicates the start of decomposition of rubidium laurate . The shoulder endotherm at 425, 450 and 480^0C indicates the rapid decomposition of the soap.

The thermogravimetric analysis of rubidium laurate reveals that the loss in the weight of the soap is very small up to a temperature of 260^0C and this is in complete agreement with the linear portion of the cure of differential thermal analysis of the soap Fig1. The TGA studies show that the loss in weight of the soap is insignificant up to 260^0C increases slowly between 260^0C and 380^0C, then increases suddenly between 380^0C to 480^0 C and finally shows no change with further increase in temperature above 580^0C. The rapid loss in weight of the soap between 320^0C to 480^0 C is evidently caused by the decomposition of the soap and is in agreement with the exotherm at 380^0C and with the endotherm at 425^0C, 450^0 C and 480^0C observed in the curve of differential thermal analysis of rubidium laurate. It is also observed that a white crystalline substance is condensed on the cold part of tube containing the soap surrounded by an inert atmosphere and it is identified as laurate.

3.2. Stearate

The exotherm at 30^0C in the differential thermal analysis curve of rubidium stearate indicates the crystallization of soap. The endotherm at 205^0 C corresponds to the melting of the soap. The linear portion of the curve between 205 to 290^0C shows that the temperature of the sample and the furnace remains the same and the loss in the weight of the soaps is insignificant during this range of temperature. The endotherm at 305^0C corresponds to the boiling of soap and an endotherm at 420^0C along with shoulder endotherms at 455^0C and 480^0C corresponds to the decomposition of the soap. The decomposition of the soap is also revealed by the exotherm at 440^0C.

The results of thermogravimetric analysis show that the loss in weight of the soap is very small up to a temperature of 290^0C and is in complete agreement with the linear portion of the curve of differential thermal analysis of rubidium stearate. The loss in weight of the soap is insignificant up to a temperature of 260^0C, increases between 260 and 380^0C, then increases rapidly between 440^0C and 500^0C and then increases again slowly with further increase in temperature. The rapid loss in weight of the soap between 440^0C and 500^0C is evidently caused by the decomposition of the soap and is in agreement with the exotherm at440^0C and endotherm at 480^0C observed in the curve of differential thermal analysis of the soap. The results show that the decomposition of the soap also takes place along with the boiling of the soap. It is observed that the white crystalline substance is condensed at the cold part of the tube containing the sample and it is identified as stearone.

3.3. Colorimetry

The absorption maxima for Thioflavin T indifferent solvents are observed at the following wave length

Solvents	Wave length
Methanol	412
Butanol	414
Pentanol	415

The absorption maxima are well defined in all solvents and are within the range 412-415 mμ. figure 2. The plots of optical density against the concentration of rubidium soaps (laurate, stearate) in alkanols (methanol, butanol, pentanol) are characterized by an intersection of two straight lines at definite soap concentration (c.m.c) , indicating the formation of the micelles fig

3 and 4. It is observed that the molecules of alcohol and dye penetrate into the palisade layer of the soap micelle with their polar group contiguous to the polar group of soap molecules and the hydrocarbon portion parallel to the hydrocarbon portion of the soap molecules and a larger micelle is produced.

Table – 1: Endotherm and Exotherm of Rubidium soaps

	Name of the soaps	
	Laurate	**Stearate**
Endotherms	35	40
	210 m	205m
	355 b	305 b
	370 s	
	425	420
	450 s	455 s
	480 s	489 s
Exotherm	20 c	30 c
	365	440 d
	380 d	510
	555	565
	580	

Key to abbreviation:b: boiling, m: melting d: decomposition s: shoulder c: crystallization

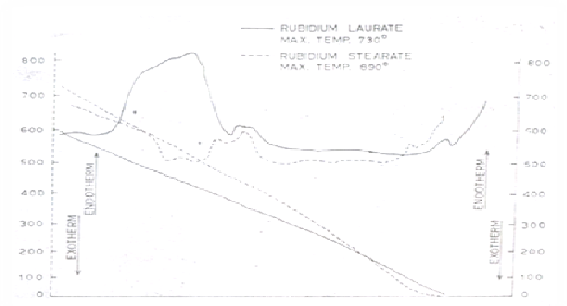

Figure – 1: Differential thermal analysis of Rubidium soap.

The values of the c.m.c for these soaps are in agreement with the value obtained from the study of other physical properties. It is observed that the values of the c.m.c are independent of the solvent but decrease with the increase in chain length of the anion in these soaps. The plots of optical density against the concentration of soap (g. moleL^{-1})are linear below and above the c.m.c for all these soaps in the alkanol solvent .The existence of linear relationship between the optical density and the soap concentration over wide range of soap concentration proves the validity of Beer- Lamberts' law in this concentration range. It is therefore concluded that the colorimetric method can be successfully used for the estimation of metal ion (rubidium) content atλmax in dilute solutions of rubidium soaps containing suitable amount of dye and for the determination of the c.m.c of these soaps.

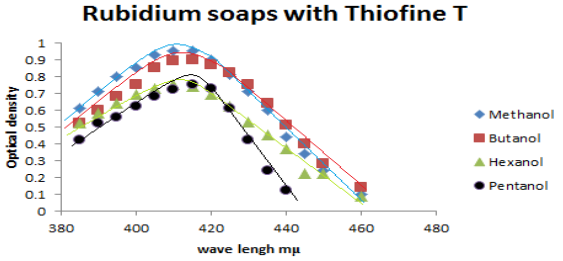

Figure – 2: Optical density vs wave length for thioflavin T.

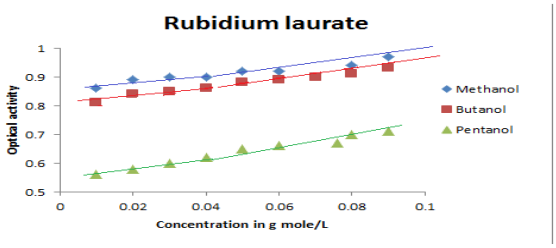

Figure – 3: Optical density vs concentration of soap in g Mol L^{-1}.

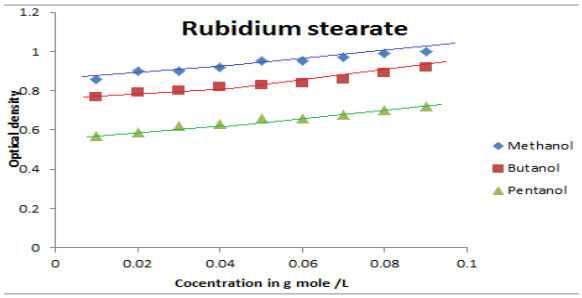

Figure – 4: Optical density vs concentration of soap in g Mol L^{-1}.

4. CONCLUSION

Differential thermal analysis reveals about the existence of three temperatures, temperature of crystallization, melting of soaps and their decomposition provide the information about their structures. The exotherm and endotherm of rubidium soaps provide information about their inhibit character for dehydrochlorination reaction of PVC at high temperature. The colorimetric analysis is established to detect metal ion rubidium in rubidium soaps solution of alkanols even present in traces. The linear relation between optical density and soaps concentration below and above c.m.c proves the validity of Beer and Lamberts' law.

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