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E: ISSN NO.: 2455-0817 *Remarking An Analisation*

X- Ray Diffraction Studies on Chromium Soaps in Solid State

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Abstract

The x-rays diffraction analysis were used to determine the structure of Chromium soaps (palmitate and stearate) in solid state. The x-ray diffraction measurements confirm that Chromium soaps possess double layer structure with long spacings.

Keywords: Metal Soaps, X-Ray Diffraction.

Introduction

The transition metal soaps are being widely used in industry, technology and allied sciences. The uses of metal soaps largely depend on their physical state, stability, chemical reactivity and solubility in polar and non polar solvents. These metal soaps has been a subject intense investigation in the recent past on account of its role in such diversified field as medicine, cosmetic emulsifier, lubricant, germicides and anti oxidant.

Review of Literature

The methods of preparation of potassium soaps and metal soaps were described by several workers (1-6). The infrared absorption spectra, x-ray diffraction studies and thermal behavior of cerium and thorium laurate were studied by Gupta et al.(7). The thermal decomposition kinetics of nickel and manganese soaps were studied by Mehrotra et al.(8).The thermogravimetric analysis of yttrium soaps in solid state was studied by Khirwar(9).The physicochemical studies on erbium soaps of saturated higher fatty acids in solid state studied by Rajesh et al.(10). The viscometric and spectral studies of copper soap in benzene and methanol mixture were studied by Rawat(11). The studies on miceller properties of Scandium and yttrium metal soaps was studied by Khirwar (12). The studies of ultrasonic velocity and allied properties of manganese, cobalt and copper soaps in non aqueous medium Rawat (13).

Aim of the Study

In the present work attempts have been made to determine the structure of Chromium soaps (palmitate and stearate) in solid state through X-Rays diffraction analysis.

Experimental

The Chromium soaps (palmitate and stearate) were synthesized by direct metathesis of corresponding potassium soaps with the required amount of aqueous solution of Chromium nitrate at 50-55^{0}C under vigorous stirring. The precipitated soaps were washed several times with distilled water and then acetone to remove the fatty acid and metal nitrate. The soaps were purified by recrystallization with the benzene and DMSO mixture, dried in an air oven at 50-60^{0}C and the finally drying of the soaps were carried out under reduced pressure.

The X-ray diffraction patterns of Chromium soaps (palmitate and stearate) were obtained with a Rich-Seifert 2002D isodebyeflex diffractometer using cu-k_α radiations filtered by a nickel foil over the range of diffraction angle, 2Θ = 3^0 to 65^0 (where Θis Bragg's angle).The XRD curves were recorded under the applied voltage of 35KV using scanning speed of 1^0 per minute and chart speed of 1 cm per minute. The wave length of radiations was taken as 1.643Å.

Result and Discussion

The x-ray diffraction patterns of Chromium soaps (palmitate and stearate) have been investigated with a view to characterize in solid state. The intensities of diffracted x-ray as a function of diffraction angle, 2θfor Chromium soaps are recorded over the range of 3-80^0. The interplaner spacings, d, have been calculated from the position of the intense peaks using Braggs relation-ship.

$$n\lambda = 2\mathrm{d}\sin\theta$$

Where λ is the wave length of radiation.

The calculated spacings and relative intensities with respect to the most intense peaks are recorded (Table:1-2). A large number of peaks arising from the diffraction of x-ray by planes of metal ion (known as basal planes) are observed in the diffraction patterns of Chromium soaps.The appearance of diffraction for Chromium palmitate upto the 14^{th} order and Chromium stearate upto the 11^{th} order confirms good crystallinity for Chromium soaps.

The long spacings average planer distance for Chromium palmitate and Chromium stearate are 35.24, 32.14*Å*, respectively. The difference in long spacings of Chromium soaps (palmitate and stearate: 3.10*Å*) correspond almost to the length of methylene (CH_2) group in the fatty acid radical constituent of the soap molecules. It is therefore suggested that the Zig-Zag chains of the fatty acid radical constituent of the soap molecules extend straight forward on the both sides of each basal plane. The values of long spacings for metal soaps are very smaller then calculated dimensions of anions (palmitate 37.0*Å* and stearate: 38.0*Å*) from the Paulings values of atomic radii and bond angles. It is therefore, concluded that the molecular axes of Chromium soap molecules are somewhat inclined to the basal planes. The metal ions of Chromium fit into spaces between oxygen atoms of the ionized carboxyl group without a large strain of the bond.

A number of diffraction peaks in the intermediate range are also observed in the diffraction patterns of Chromium soaps and are attributed to the diffraction of x-ray by plans of atoms of much smaller separation than the basal planes. The calculated spacings i,e the lateral distances between one soap molecule and the next in a layer. It is observed that the long spacing peaks are fairly intense while the short spacing peaks are relatively weak.

The values of the long spacings for Chromium soaps are agreement with the double layer structure of the soaps proposed by Vold and Hattiangdi[14]. On the basis of long and short spacings, it is suggested that the metal ions are arranged in parallel planes equally spaced in the soap crystal with fully extended Zig-Zig chains of fatty acid radicals on both sided of each basal plane.

Conclusion

The results suggest that Chromium soaps posses double layer structure with molecular axes somewhat inclined to the basal planes.

Table-1: X-rays Diffraction Analysis of Chromium palmitate

Sr.No.	2θ	θ	sin θ	D	d(*Å*)	n
1.	3.256	2.967	0.0356	15.636	37.872	3
2.	6.345	3.184	0.0568	10.386	36.158	4
3.	8.326	4.763	0.0756	8.963	34.852	5
4.	9.419	6.606	0.0820	6.013	35.065	6
5.	12.123	7.596	0.0901	4.207	37.230	7
6.	17.265	8.117	0.1284	3.868	38.630	10
7.	16.351	9.135	0.1513	1.335	35.475	13

Average value of d = 35.24*Å*

Table2: X-rays Diffraction Analysis of Chromium stearate

Sr.No.	2θ	θ	sin θ	D	d(*Å*)	N
1.	4.109	2.554	0.0171	15.356	38.712	2
2.	5.213	3.106	0.0367	13.916	35.748	3
3.	7.329	3.164	0.0451	8.392	33.568	4
4.	8.928	2.964	0.0317	6.918	34.590	5
5.	9.863	0.431	0.0075	5.996	29.976	7
6.	0.532	0.266	0.0046	3.985	30.188	9
7.	0.210	0.105	0.0018	2.869	34.428	13

Average value of d = 32.14*Å*

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