Innovation The Research Concept

X- Ray Diffraction Studies on Scandium Soaps in Solid State

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Abstract

The x-rays diffraction analysis were used to determine the structure of scandium soaps (myristate and palmitate) in solid state. The x-ray diffraction measurements confirm that scandium 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. 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 magnese, cobalt and copper soaps in non aqueous medium Rawat (13). In the present work attempts have been made to determine the structure of scandium soaps (myristate and palmitate) in solid state through X-Rays diffraction analysis.

Aim of the Study

The results of the survey of literature reveals that the physico-chemical properties of scandium soaps have not been systematically investigated while they have many uses in industries and academic field. The aim of this research work is to study the structure of scandium soaps in solid state through x- rays diffraction analysis.

Experimental

The scandium soaps (myristate and palmitate) were synthesized by direct metathesis of corresponding potassium soaps with the required amount of aqueous solution of scandium nitrate at $50\text{-}55^0$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 $50\text{-}60^0$C and the finally drying of the soaps were carried out under reduced pressure.

The X-ray diffraction patterns of scandium soaps (myristate and palmitate) 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, $2\Theta = 3^0$ to 65^0 (where Θ is 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 1.543Å.

Review of Literature

Present research work reviews the literature relevant with the aim of study. The spectroscopic studies of metallic soaps have been studied by several workers. Some of them are listed below.

Anushri Gupta (2012) was studied the infrared absorption spectra, x-ray diffraction studies and thermal behavior of cerium and thorium laurate.

M.K.Rawat (2007) was studied the ultrasonic velocity and allied properties of magnese, cobalt and copper soaps in non aqueous medium

M.S.Khirwar (2016) was studied the thermogravimetric analysis of yttrium soaps in solid state.

Result and Discussion

The x-ray diffraction patterns of scandium soaps (myristate and palmitate) have been investigated with a view to characterize in solid state. The intensities of diffracted x-ray as a function of diffraction angle, 2θ for scandium soaps are recorded over the range of 3-70^0. 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 scandium soaps. The appearance of diffraction for scandium myristate upto the 15th order and scandium Palmitate upto the 12th order confirms good crystallinity for scandium soaps.

The long spacings average planer distance for scandium myristate and scandium palmitate are 36.04, 33.74 $\mathring{A}$, respectively. The difference in long spacings of scandium soaps (myristate and palmitate: 2.30$\mathring{A}$) 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 (myristate 47.0 $\mathring{A}$ and palmitate: 47.0 $\mathring{A}$) from the Paulings values of atomic radii and bond angles. It is therefore, concluded that the molecular axes of scandium soap molecules are somewhat inclined to the basal planes. The metal ions, Sc^{3+} 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 scandium 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 scandium 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.

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

Table1
X-rays Diffraction Analysis of Scandium myristate

Sr.No.	2θ	θ	$\sin\theta$	D	d($\mathring{A}$)	n
1.	2.935	1.467	0.0256	17.936	35.872	2
2.	4.39	2.184	0.0381	11.386	34.158	3
3.	7.526	3.763	0.0656	8.963	35.852	4
4.	9.413	4.706	0.0820	7.013	35.065	5
5.	9.193	4.596	0.0801	6.205	37.230	6
6.	18.235	9.117	0.1584	3.863	38.630	10
7.	16.251	8.125	0.1413	2.365	35.475	15

Average value of d = 36.04 Å

Table2
X-rays Diffraction Analysis of Scandium palmitate

Sr.No.	2θ	θ	$\sin\theta$	D	d($\mathring{A}$)	n
1.	3.109	1.554	0.0271	18.356	36.712	2
2.	4.213	2.106	0.0367	11.916	35.748	3
3.	6.329	3.164	0.0551	8.392	33.568	4
4.	5.928	2.964	0.0517	6.918	34.590	5
5.	0.863	0.431	0.0075	4.996	29.976	6
6.	0.532	0.266	0.0046	3.985	31.188	8
7.	0.210	0.105	0.0018	2.869	34.428	12

Average value of d = 33.74 Å

Conclusion

It is concluded that the structure of scandium soaps in solid state studied by the X-Rays Diffraction results confirm that these metal soaps possess double layer structure with long spacing.

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