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Spectroscopic Studies on Carboxylates of Vanadium in Solid State

Abstract

The Spectroscopic studies were used to determine the structure of carboxylates of vanadium (myristate and palmitate) in solid state. The x-ray diffraction measurements confirm that carboxylates of vanadium possess double layer structure with long spacings.

Keywords: Metal Soaps, Carboxylates, X-Ray Diffraction.

M.S.Khirwar
Assistant Professor,
Deptt. of Chemistry,
R.B.S. College,
Agra, India

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 (1-8). 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 IR spectra, X-ray and thermal analysis of lanthanum soaps were studied by Shukla et. Al (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 carboxylates of vanadium (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 spectroscopic studies on carboxylates of vanadium have not been systematically investigated while they have many uses in academic and industrial field. The aim of this research work is to the spectroscopic studies on carboxylates of vanadium in solid state.

Experimental

The carboxylates of vanadium (myristate and palmitate) were synthesized by direct metathesis of corresponding potassium soaps with the required amount of aqueous solution of vanadium 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 carboxylates of vanadium (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 on carboxylates of transition metals have been studied by several workers. Some of them are listed below.

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

Rajesh Dwivedi (2014) was studied the Physicochemical Studies on Erbium Soaps of Saturated Higher Fatty Acids in Solid State.

R.K.Shukla (2003) was studied the IR spectra, X-ray and thermal analysis of lanthanum soaps.

Result and Discussion

The x-ray diffraction patterns of carboxylates of vanadium (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 carboxylates of vanadium 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 carboxylates of vanadium. The appearance of diffraction for vanadium myristate upto the 15th order and vanadium Palmitate upto the 12th order confirms good crystallinity for carboxylates of vanadium.

The long spacings average planer distance for vanadium myristate and vanadium palmitate are 36.04, 33.74 Å, respectively. The difference in long spacings of carboxylates of vanadium (myristate and palmitate: 2.30Å) 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 Å and palmitate: 47.0 Å) 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 vanadium metal ions 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 carboxylates of vanadium 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 carboxylates of vanadium 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.

Table1. X-rays Diffraction Analysis of Vanadium myristate

Sr.No.	2θ	θ	sin θ	D	d(Å)	n
1.	2.905	2.417	0.0356	16.936	34.872	3
2.	4.192	3.164	0.0681	14.586	34.178	4
3.	7.326	4.763	0.0756	8.913	35.852	5
4.	9.483	5.704	0.0920	7.013	35.005	6
5.	9.195	7.596	0.0801	8.205	36.230	7
6.	18.205	8.113	0.1484	3.863	38.600	10
7.	16.253	9.105	0.1513	2.365	35.475	14

Average value of d = 35.04 Å

Table2. X-rays Diffraction Analysis of Vanadium palmitate

Sr.No.	2θ	θ	sin θ	D	d(Å)	n
1.	3.106	1.554	0.0271	16.556	37.712	3
2.	4.213	2.106	0.0307	10.906	36.743	4
3.	6.300	3.164	0.0559	7.392	34.528	5
4.	7.928	2.964	0.0417	6.908	342.500	6
5.	0.853	0.631	0.0075	4.996	28.676	8
6.	0.532	0.466	0.0056	3.935	30.184	9
7.	0.310	0.100	0.0038	2.569	32.425	11

Average value of d = 30.29 Å

Conclusion

It is therefore, concluded that the results suggest that carboxylates of vanadium posses double layer structure with molecular axes somewhat inclined to the basal planes.

End Notes

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