Structural Study On Manganese And Potassium Myristate And Stearate By X-Ray Diffraction (XRD)

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***Abstract*—X-ray Diffraction (XRD) used to determine the layered structure and crystallinity of fatty acid, manganese and potassium myristate and stearate soap. The important frequencies of absorption Maxima in X-ray Diffraction (XRD) of fatty acids and manganese and potassium myristate and stearate soap weere recorded and compare the result of corresponding myristic and steric acid and potassium soaps. X-ray diffraction (XRD) of metallic soaps of potassium and manganese revealed highly crystalline layered structures with ionic metal-to-oxygen bonds. Analysis shows a "double layer" structure, where metal ions lie between parallel planes of fatty acid chains. Long spacing peaks indicate the spacing between these planes, which are observed in metallic soaps.**

***Index Terms*—Manganese, Myristate, Stearate, X-ray Diffraction, Metallic soaps**

I. INTRODUCTION

The study of metallic soaps is becoming increasingly important in technology and academic fields, and the application of these metal soaps depends largely on their physical state, stability, and chemical reactivity, nature of the bonding and structure of the soaps. Several workers studied on nature and structures of these soaps are of great importance for their use in industries and for explaining their character under different conditions. The current work deals with the X-ray diffraction (XRD) studies of manganese and potassium soaps in the solid state, initiated with a view to obtain structural information and the nature of the metallic soaps. The high metal content in metallic soaps makes them unique in many diversified fields.

XRD is an important technique used to study the crystal structures and molecular arrangements of materials. It works on the principle that when X-rays fall on a crystalline substance, they are scattered by the atoms in different directions, producing a diffraction pattern. This pattern provides information about the spacing between atoms and the structure of the material. In the case of manganese and potassium soaps, X-ray diffraction helps in understanding their structural properties and degree of crystallinity. Metal soaps such as manganese soap and potassium soap are metallic salts of fatty acids. These compounds are widely used as lubricants, stabilizers, catalysts, and in various industrial applications. By analyzing the X-ray diffraction patterns of manganese and potassium soaps, can determine the arrangement of molecules, interlayer spacing, and structural differences between them. This study helps in understanding their physical properties and improving their industrial application.

II. AIM

X-ray diffraction (XRD) analysis of metallic soaps reveals characteristic solid-state structure typically exhibiting a layered "head-to-head" or "head to tail" arrangement with specific interplanar spacings dependent on the metal cation and fatty acid chan length. The studies show that XRD patterns are unique to each soap.

III. EXPERIMENTAL

Purification of Chemicals:

Fatty Acids:

Myristic and stearic acids were purified by distilling under reduced pressure and recrystallizing with alcohol. The purity of the acids was checked by determining their melting points. The melting point of the purified acids are as follows:

Myristic acid M.P.: 54.0℃

Stearic acid M.P.: 71.5℃

Solvents:

Benzene was purified by keeping over sodium for a couple of days and then distilling. The distillate was refluxed over sodium metal and then redistilled. The boiling point of purified benzene was 80.1℃.

Methanol was purified by keeping it over potassium hydroxide for twenty-four hours and then distilling. The distillate was refluxed with 1% of calcium metal for approximately eight hours and redistilled. The boiling point of the purified methanol was 65.0℃.

Preparation of Soaps:

Potassium myristate and stearate were prepared by refluxing equivalent amounts of the corresponding fatty acids and aqueous solution of potassium hydroxide for 6 – 8 hours in a water bath. The soaps were purified by recrystallization with methanol and dried under reduced pressure.

Manganese soaps were prepared by direct metathesis of the corresponding potassium soap (myristate and stearate) with a slight excess of the solution of manganese chloride, under stirring. The precipitated soaps were washed several times with distilled water and acetone. The soaps were purified by recrystallization with benzene – methanol mixture. The purity of the soaps was confirmed by determining their melting points. The melting points of the purified soaps are as follows:

Manganese myristate: 127℃

Manganese stearate :110℃

Preparation of Soap Solutions:

The calculated amount of soap was weighed in a standard flask, and the solution was prepared by adding the required amount of solvent mixture of benzene and methanol. In this way, numbers of solutions of different concentrations of manganese myristate, manganese stearate were prepared.

IV. MEASUREMENTS:

X-ray Diffraction Analysis:

The X-ray diffraction patterns for manganese metal soaps of myristate and stearate were obtained with a RIGAKU (Geigerflex – RB – RU – 200) X-ray diffractometer using Cu – Kα radiations filtered by a nickel foil over the range of diffraction angle 2θ = 3O to 80O (where θ is Bragg's angle). The XRD curves were recorded under the applied voltage of 35kv using a scanning speed of 1O per minute and chart speed of 1 cm per minute. The wavelength of the radiations was taken as 1.54 A0.

V. RESULT AND DISCUSSION

The values of long spacings for manganese soaps are in agreement with the double layer structure of the soaps proposed by Vold and Hattiangdi in which the metal ions arranged in parallel planes equally spaced in the soap crystal with fully extended zig-zag chain of fatty acid radicals on both sides of each basal plane are shown in Fig 1.

Generally, the metal soaps do not form large crystal sufficient for a detailed single crystal examination and so the X-ray diffraction patterns of manganese soaps of different fatty acids have been analysed to characterize the structure of these soaps in solid state.

Key findings of X-ray diffraction (XRD) analysis of metallic soaps:

The intensities of diffracted X-rays as a function of diffraction angle, 2θ for manganese soaps of stearic and myristic acids are recorded with the help of X-ray spectro photometer and recorded curves as shown in

Fig 2. The interplanar spacings, d, have been calculated from the position of intense peaks using Bragg's relationship.

$n\lambda = 2d \sin\theta$

Where, λ is the wavelength of radiation. The calculated spacings together with the relative intensities with respect to most intense peak is recorded in tables 1 and 2 for manganese soaps. In X-ray diffraction patterns of manganese soaps (myristate and stearate), numerous peaks are observed over the range of the diffraction angle 5–80^O. These peaks are attributed to the diffraction of X-rays by planes of metal ion known as basal planes. The appearance of diffractions upto 26th and 38th order for myristate and stearate of manganese confirms good crystallinity for these soaps. The average planar distance i.e., long spacings of manganese myristate, manganese stearate are 38.99 A^O and 47.05 A^O respectively.

The difference in the observed values of long spacings of manganese laurate (33.8 A^O) and myristate (39.00 A^O) is 5.2 A^O, manganese myristate (39.00 A^O) and palmitate (43.5 A^O) is 4.5 A^O and manganese palmitate (43.5 A^O) and stearate (47.6 A^O) is 4.1 A^O corresponds to double the length of methylene ($-CH_2$) in the fatty acid radical constituent of the soap molecules. It is, therefore, suggested that zig-zag chains of fatty acid radical constituent of soap molecules extend straight forward on both sides of each basal plane. The values of long spacings for manganese soaps are somewhat smaller than the calculated dimensions of anions (laurate: 37.00, myristate: 42.00, palmitate: 47.00 and stearate: 52.00 A^O) from Paulings values of atomic radii and bond angles which suggests that the molecular axis of these soap molecules are somewhat inclined to the basal planes. The metal ions Mn^{++} fit into spaces between oxygen atoms of the ionized carboxyl group without a large strain of the bond.

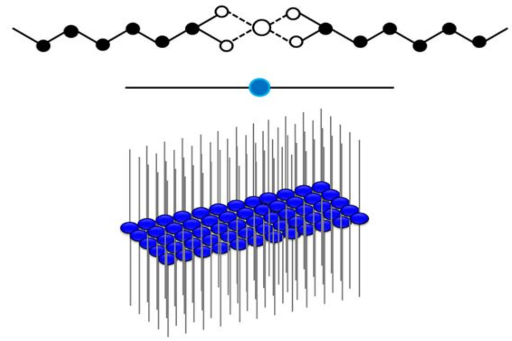


Fig 1: Double- layer structure of metal soap crystal

A large number of peaks in the intermediate range are also observed in the diffraction patterns of manganese soaps and are attributed to the diffraction of the X-rays by planes of atom of much smaller separation than the basal planes. The calculated spacings from their peaks correspond to the shorter side spacings i.e. the lateral distance 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 result suggests that manganese and iron soaps of different acids possess single layer structure with molecular axes somewhat inclined to the basal plane.

Table 1 X-ray diffraction analysis of manganese myristate

S. No.	2θ	θ	sinθ	2sinθ	λ/2sinθ	d	η
1.	5.1004	2.5502	0.0400	0.0800	19.2500	38.5000	2
2.	6.9124	3.4562	0.0603	0.1206	12.7695	38.3084	3
3.	9.2453	4.6226	0.0806	0.1612	9.5533	38.2133	4
4.	11.5340	5.7670	0.1005	0.2010	7.6617	38.3085	5
5.	13.8123	6.9061	0.1202	0.2404	6.4060	38.4359	6
6.	15.9212	7.9606	0.1385	0.2770	5.5596	38.9169	7
7.	18.1104	9.0552	0.1573	0.3146	4.8951	39.1608	8
8.	20.2124	10.1062	0.1755	0.3510	4.3875	39.4875	9
9.	22.6524	11.3262	0.1964	0.3928	3.9206	39.2057	10

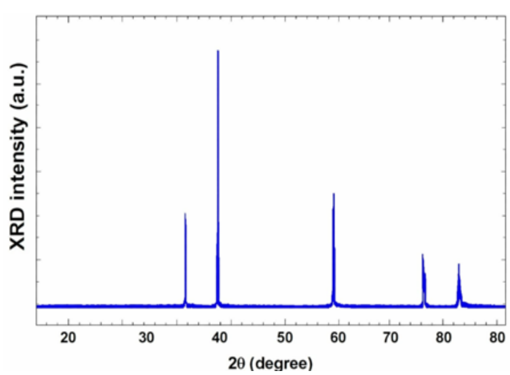
10.	24.9423	12.4711	0.2159	0.4318	3.5664	39.2311	11
11.	26.8924	13.4462	0.2325	0.4650	3.3118	39.7419	12
12.	30.2104	15.1052	0.2606	0.5212	2.9547	38.4113	13
13.	36.4425	18.2212	0.3127	0.6254	2.4624	39.3987	16
14.	40.8994	20.4497	0.3494	0.6988	2.2038	39.6680	18
15.	46.5426	23.2713	0.3951	0.7902	1.9489	38.9775	20
16.	50.9224	25.4612	0.4299	0.8598	1.7911	39.4045	22
17.	60.8934	30.4467	0.5067	1.0134	1.5196	39.5105	26

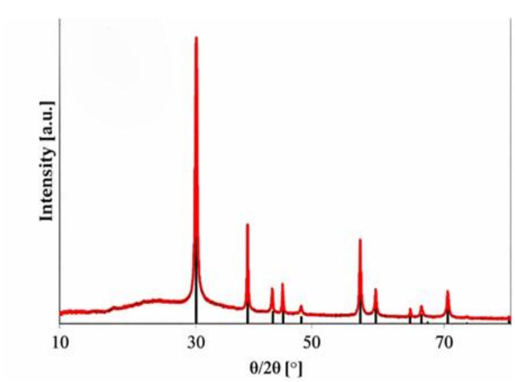
Average value of d = 38.99 A^O

Table 2 X-ray diffraction analysis of manganese stearate

S. No.	2θ	θ	sinθ	2sinθ	λ/2sinθ	d	η
1.	3.7050	1.8525	0.0323	0.0646	23.8287	47.6574	2
2.	5.5600	2.7800	0.0485	0.0970	15.8821	47.6463	3
3.	7.4050	3.7025	0.0646	0.1292	11.9281	47.7144	4
4.	9.2525	4.6262	0.0806	0.1612	9.5505	47.7525	-5
5.	11.1600	5.5800	0.0972	0.1944	7.9220	47.5320	6
6.	12.9725	6.4862	0.1129	0.2258	6.8189	47.7323	-7
7.	20.8725	10.4362	0.1811	0.3622	4.2525	46.7775	11
8.	21.7500	10.8750	0.1886	0.3772	4.0829	48.9948	12
9.	22.8675	11.4337	0.1982	0.3964	3.8858	46.6300	12
10.	37.3700	18.6850	0.3204	0.6408	2.4049	48.0880	20
11.	46.2550	23.1275	0.3927	0.7854	1.9612	47.0688	24
12.	76.9475	38.4737	0.6221	1.2442	1.2381	47.0478	38

Average value of d =47.55 A^O





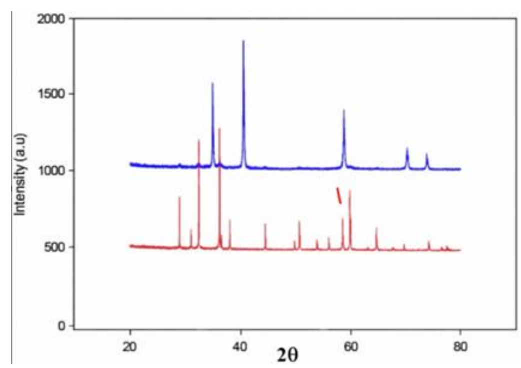


Fig 2: x-ray diffraction of manganese myristate and stearate

VI. CONCLUSION:

X-ray powder diffraction provides a critical "fingerprint" of metallic soaps. It confirms their crystalline, layered, lamellar structure and is essential for identifying specific soap phases in industrial products and degradative metallic soaps (e.g., in oil paintings). The most intense peaks are at low angles, providing precise information about the long-chain spacing.

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