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Infrared, X-rays Diffraction and Thermal Studies of Chromium soaps

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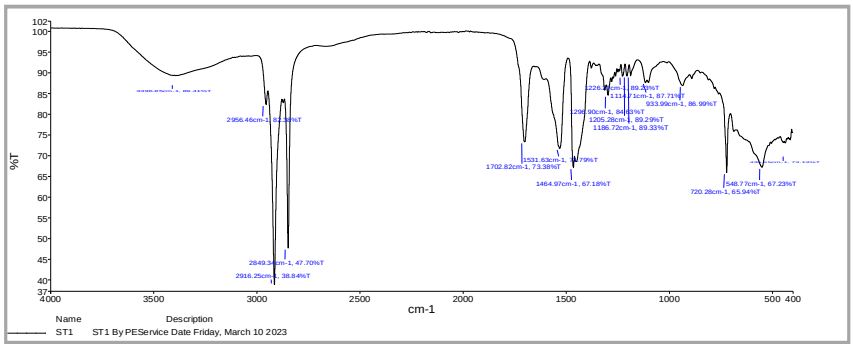
ABSTRACT

The physicochemical characteristics of chromium soaps (myristate, palmitate, and stearate) were evaluated in the solid state using the infrared spectroscopic technique, thermal and X-ray diffraction Analysis to find out the structure of Chromium soaps.IR results reveal that the fatty acid exists with a dimeric structure through intermolecular hydrogen bonding between two molecules of fatty acids whereas the metal-to-oxygen bonds in chromium soaps(myristate, palmitate, and stearate) were ionic in nature. The X-ray diffraction measurements confirm that these soaps possess a double-layer structure with long posing. The results of the thermogravimetric analysis reveal that the decomposition process of these soaps is of zero order and the energy of activation for the decomposition process lies in the range of 18.75, and 32.33 KJmol$^{-1.}$

Graphical Abstract:



Infrared Absorption spectrum of Chromium Stearate.

Keywords: IR Spectra, X-ray diffraction, Chromium (myristate, palmitate, and stearate).

INTRODUCTION

Metallic soaps are materials of considerable commercial importance. Significant application areas [**1-5**] for metal soaps include lubricating greases which are intended to improve flow, coating smoothness, finish, printability, antidusting effects, driers in paints, dry cleaning industries, cosmetic gels, heat stabilizers for plastics and in the development of polyvinylchloride as an important commercial polymer. Other uses of metal soaps are as fungicides and pesticides [**6**], optical polymer

fibers [**7, 8**], coating pigment in the paper industry [**9**], and in the preparation of nanofilms [**10-13**]. However, the physicochemical characteristics and structure of metal soaps depend to an extent on the method and conditions of their preparation and so studies on the nature and structure of these soaps are of great importance for their uses in industries and for explaining their characteristics under different conditions.

The present investigation studies the physicochemical characteristics and structure of chromium soaps (myristate, palmitate, and stearate) in the solid state using Infrared, X-ray diffraction, and thermal measurements.

MATERIALS AND METHODS

Chromium soaps (myristate, palmitate, and stearate) were prepared by direct metathesis of the corresponding potassium soap with a slight excess of Chrome alum solution under vigorous stirring. All the chemicals were used of AR grade. The precipitated soaps were washed and filtered with distilled water and methanol and dried. The soaps were recrystallized with benzene and methanol and the purity of the soaps was identified by IR spectra.

The thermal analysis was carried out with an instrument Perkin Eimer model TGA 4000 at a constant heat from 30 °C to 300°C at 30.00°C/min in a nitrogen atmosphere and maintaining similar conditions throughout the investigations. The XRD patterns were recorded with Bruker D8 Advance powder X-ray diffraction (XRD) system. After that, the diffractogram was analyzed with the software Origin 6.1. The intensities of the diffracted X-rays as a function of diffraction angle, 2Θ for chromium soaps were measured and the inter planar spacing's, d have been calculated from the positions of intense peaks using Bragg's relationship, $n\lambda = 2d\sin\Theta$, where λ is the wavelength of radiation. Infrared absorption spectra of chromium soaps acid were recorded with Fourier transform infrared spectrometer, Tensor 27, Bruker in the region 4000-400 cm-1 using the potassium bromide disc method.

RESULTS AND DISCUSSION

The infrared spectral bands (Figures 1-3) and their tentative assignments for chromiummyristate, chromium palmitate, and chromium stearate are assigned and compared with potassium myristate, potassium palmitate, and potassium stearate as well as with corresponding fatty acid (myristic acid, palmitic acid, and stearic acid) in tables 1. The absorption maxima near 2640 - 2650 (O-H stretching vibrations, 1700-1650 (C=O stretching vibrations), 940-930, 690-689 and 550 cm^{-1} in the spectra of fatty acids indicate the presence of a localized carboxyl group in the form of dimeric structure and the existence of intermolecular hydrogen bonding between two molecules of acid.

The absorption bands observed near 2640-2650 and 940-930 cm^{-1} corresponding to the –OH group in the spectra of fatty acids have completely disappeared in the spectra of chromium soaps. The absorption band observed at 1700-1650 cm^{-1} corresponding to the carbonyl group of the fatty acid is also observed in the spectra of chromium soaps with weak intensity which may be due to the incomplete resonance of the carbonyl group in the chromium soaps. The singlet band observed near 690-720 cm^{-1} is the characteristic of trivalent metal soaps.

The appearance of two absorption bands observed near 1450-1460cm^{-1} and 1540-1530 cm^{-1} in the spectra of chromium soaps correspond to symmetric and asymmetric vibrations of carboxylate ion. The band observed near 437-439-435 cm^{-1} in the spectra of chromium soaps correspond to Cr-O bonds. The chromium soaps do not exhibit any absorption band near 3600-3000 cm^{-1} which confirms the absence of water molecules in these soap molecules.

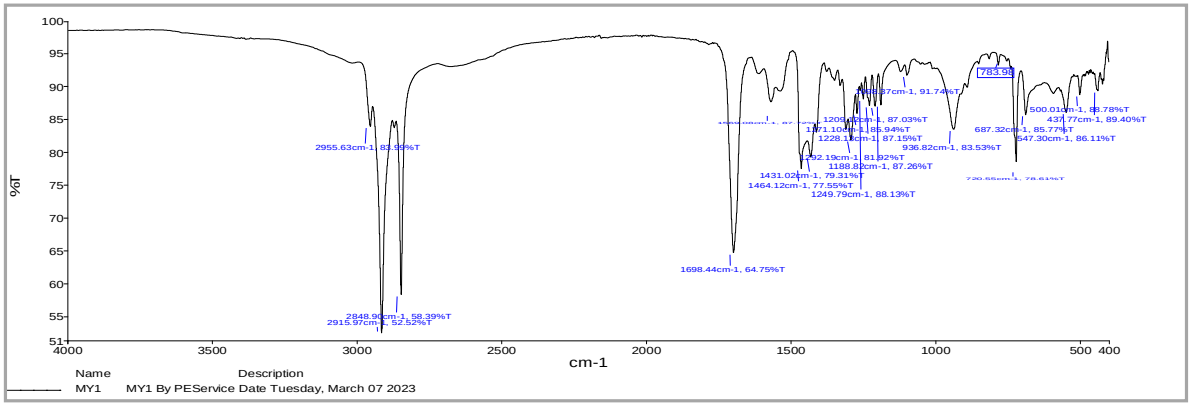


Figure 1. Infrared Absorption spectrum of Chromium Myristate.

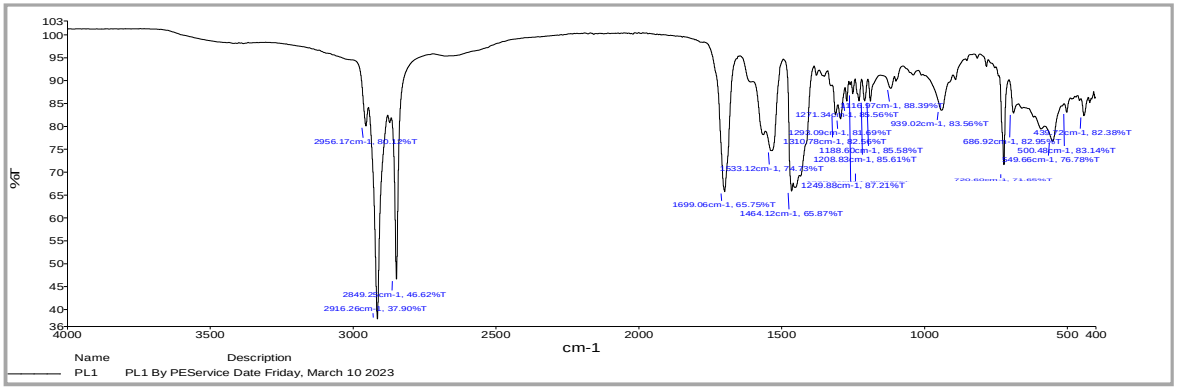


Figure 2. Infrared Absorption spectrum of Chromium Palmitate.

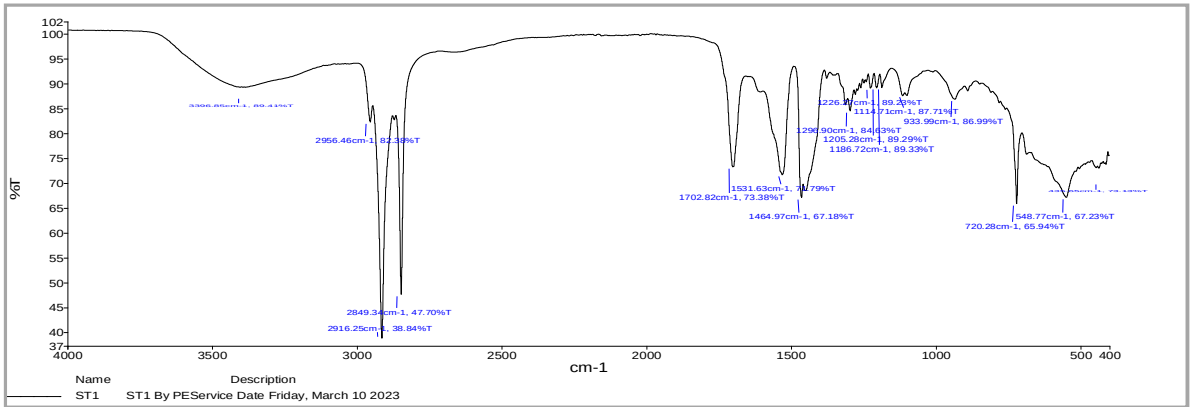


Figure 3. Infrared Absorption spectrum of Chromium Stearate.

X-ray Diffraction Analysis: The x-ray diffraction studies of chromium myristate, chromium palmitate, and chromium stearate have been done to characterize the structure in the solid state [**14, 15**]. The calculated spacing together with the relative intensities concerning the most intense peaks are recorded in tables 2-4. The intensity and sharpness (half-width; i.e. angular width of the peak at half its maximum intensity) of the peaks are the measures of the degree of crystallinity of the metal soaps. The appearance of diffraction up to 45, 31, and 50th order for myristate, palmitate, and stearate respectively confirm good crystallinity of these metal soaps. The average planar distances, i.e. long spacing's for chromium myristate, chromium palmitate, and chromium stearate were found to be d =41.7247 (Å), d = 46.8786 (Å) and d =51.7978 (Å) respectively.

Table 1. Infrared Absorption frequencies (cm^{-1}) together with their assignments

S.No.	Absorption	Myristic Acid	Chromium Myristate	Palmitic Acid	Chromium Palmitate	Stearic acid	Chromium Stearate
1	CH_3, C-H asymmetrical stretching	2960VW	2955VW	2960VW	2956VW	2960W	2956VW
2	CH_2, C–H asymmetrical stretching	2920VS	2915W	2920S	2916S	2920VS	2916VS
3	CH_2, C–H symmetrical stretching	2855VS	2848VS	2860S	2849S	2850S	2849S
4	OH, stretching	2640W	-	2650VS	-	2650S	-
5	C=O stretching	1700VS	-	1650VS	1699VS	1700VS	1702VS
6	COO^-, C–O asymmetrical stretching	-	1464M	-	1464S	-	1531S
7	CH_2 deformation		-	1470M	1430W	-	1464W
8	COO^-, C–O symmetrical stretching	-	1430VW	-	1550-1530S	-	1464W
9	CH_2, (adjacent to COOH group) deformation	1375VW	-	-	-	1412VS	-
10	CH_3, symmetrical deformation	1340W	1292W	1350M	1310W	1375W	1296W
11	Progressive bonds (CH_2 twisting and wagging)	1350-1190W	1350-1110S	1370-1090W	1293-1116 W	1350-1202M	1226-1116M
12	CH_3, rocking	1120W	1110S	1220W	1208W	1110M	1114W
13	OH, out of plane deformation	940M	-	930VS	-	930M	-
14	CH_2, rocking	725W	720VS	720S	690VS	730M	720MS
15	COOH. Bending mode	690M	-	690M	-	689S	-
16	COOH wagging Mode	550M	-	550M	-	550M	-
17	Cr—O bond	-	437VS	-	439W	-	435M

The average planar distances, i.e. long spacing, for chromium myristate, chromium palmitate, and chromium stearate were found to be 41.7247 (Å), 46.8786 (Å), and 51.7978 (Å) respectively. The difference in the observed values of long spacings for chromium myristate, palmitate, and stearate is 10.0731, which corresponds to the double length of methylene groups ($-CH_2$) in the fatty acid radical constituent 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 both sides of each basal plane. The values of long spacing for chromium soaps Myristate 41.7247 (Å), Palmitate 46.8786 (Å), and Stearate 51.7978(Å) are smaller than the calculated dimensions of anions (myristate 42 (Å), palmitate

Table 2. X-ray diffraction analysis of Chromium Myristate

S.No.	2Θ	Θ	SinΘ	D	d(Å)	N
1	15.45	7.725	0.134419	20.8118	41.6235	2
2	18.55	9.275	0.16117	10.4909	41.9637	4
3	21.05	10.525	0.18266	8.4253	42.1265	5
4	20.2	10.1	0.175367	6.9397	41.6382	6
5	22.7	11.35	0.196802	5.8379	40.8657	7
6	23.25	11.625	0.201505	4.7362	42.6258	9
7	26.25	13.125	0.227076	4.2561	42.5610	10
8	27.9	13.95	0.241075	3.4130	40.9560	12
9	29.3	14.65	0.252914	3.1987	41.5833	13
10	30.15	15.075	0.260083	2.9771	41.6795	14
11	31.25	15.625	0.26934	2.7568	41.3520	15

Average value of Myristate = d(Å) = 41.7247

47 (Å), and stearate 52 (Å) from Pauling's value of atomic radii and bond angle, which suggests that the molecular axes of soap molecules are somewhat inclined to the basal plane. The metal ions, Cr^{3+} fit into spaces between oxygen atoms of the ionized carboxyl group without large strain of the bond. Some diffraction peaks in the intermediate range of diffraction angles 6°- 70°, are also observed in diffraction patterns of chromium soaps and these are attributed to the diffraction patterns of chromium soaps and these are attributed to the diffraction of X-rays by planes of atoms of much smaller separation than the basal planes. The calculated spacing from these peaks corresponds to the shorter side spacing's, i.e. the lateral distance between one soap molecule and the next in a layer. It is based on short and long spacing's. As a consequence of this, X-ray spectroscopy is a very helpful technique that can be used to characterize a wide variety of materials [**16**].

Table 3. X-ray diffraction analysis of Chromium Palmitate

S. No.	2Θ	Θ	SinΘ	D	d(Å)	N
1	18.3	9.15	0.15902	46.9342	46.9342	1
2	19.98	9.99	0.173476	15.6052	46.8186	3
3	20.03	10.015	0.173906	11.8192	47.2768	4
4	20.18	10.09	0.172195	9.2431	46.2155	5
5	20.67	10.335	0.179317	6.6672	46.6704	7
6	21.1	10.55	0.183094	5.2484	47.2356	9
7	21.95	10.975	0.190381	4.6832	46.832	10
8	22.2	11.1	0.192522	4.3245	47.5698	11
9	23.5	11.75	0.203642	3.9514	47.4169	12
10	24.02	12.01	0.208082	3.2893	46.0502	14
11	24.8	12.4	0.214357	2.9155	46.6485	16

Average value of Palmitate = d(Å) =46.878

Table 4. X-ray diffraction analysis of Chromium Stearate

S.No.	2Θ	Θ	SinΘ	D	d(Å)	N
1	22.82	11.41	0.197824	25.8417	51.6834	2
2	23.2	11.6	0.201078	17.0881	51.2642	3
3	29.12	14.56	0.251394	12.9061	51.6244	4
4	29.95	14.975	0.258398	10.4728	52.3642	5
5	30.75	15.375	0.265135	8.6993	52.1958	6
6	30.82	15.41	0.265724	6.5248	52.1984	8
7	31.75	15.875	0.27354	5.6678	51.0102	9
8	32.95	16.475	0.283597	4.4142	52.9704	12
9	34.55	17.275	0.296908	3.7042	51.8588	14
10	34.25	17.125	0.294457	3.4642	51.9630	15
11	35	17.5	0.300706	3.1652	50.6432	16

Average value of Stearate = d(Å) = 51.7978

Thermal Analysis (TGA): The thermal decomposition of chromium soaps (myristate, palmitate, and stearate) was analyzed by thermogravimetric analysis [**15, 17**]. The results of the thermogravimetric analysis are given in figure. 4, 5, and 6. It is found that the weight of the final residue isin agreement with the theoretically calculated weights of chromium trioxide from the molecular formulas of the soaps. The thermal decomposition of chromium soaps may be expressed as.

$$(RCOO)_3Cr \longrightarrow Cr^{3+} + 3RCOO^-$$

$$2(RCOO)_3Cr \longrightarrow 3RCOR + Cr_2O_3 + 3CO_2$$

Where R = $C_{17}H_{35}$, $C_{15}H_{31}$ and $C_{13}H_{27}$

The results of the thermal decomposition of chromiumsoaps were explained in the light of some well-knownequations, the Freeman-Carroll's **[18]** and Coats Redfern's [**19**] equations expressed as follows

$$\Delta[\log(dw/dt)] = \frac{-E}{\Delta(\log Wr)} \quad \frac{\Delta(1/T)}{2.303\,R} \quad \left(\frac{+n}{\Delta(\log Wr)}\right)$$

Where, E = Energy of activation, n = order of reaction, T = Temperature on an absolute scale, Wr = difference between the total loss in weight and loss in weight at time t i.e. $W_O - W_v$, dw/dt = rate of weight loss obtained from the loss in weight of soaps and the loss at the predetermined time.

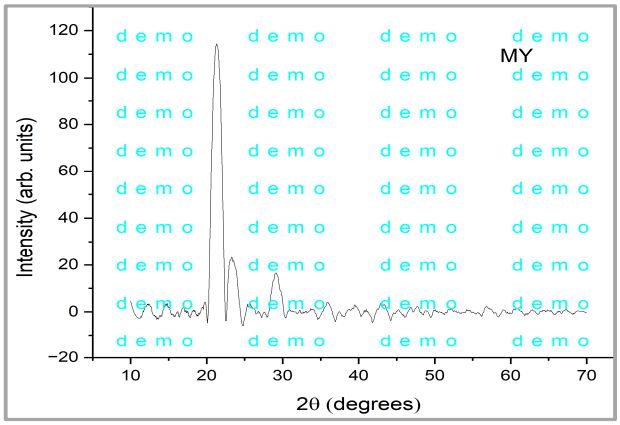


Figure 4. X-ray diffraction of Chromium Myristate.

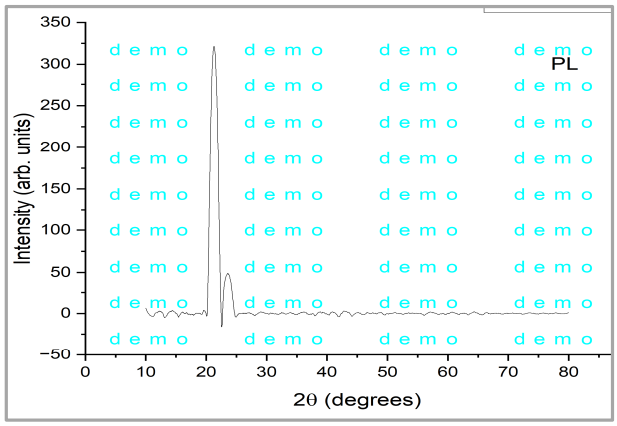


Figure 5. X-ray diffraction of Chromium Palmitate.

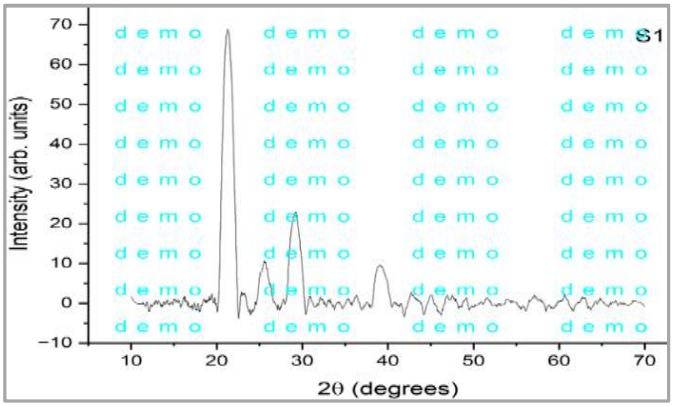


Figure 6. X-ray diffraction of Chromium Stearate.

As a result, from table 5, 7, and 9, Freeman Carroll's equation indicated that the thermal decomposition of these soaps shows kinetically of Zero order and the value of activation from the decomposition of Chromium soaps obtained from the range of 23.46 - 32.33 K.cal mole^{-1}.

$$\left(\frac{\log\ 1-(1-\alpha)^{1-n}}{T^2(1-n)}\right) = \frac{\log AR}{a\,E}\left(\frac{1-\ 2RT}{E}\right) - \frac{E}{2.303RT}$$

Where, a =Fraction of the soap decomposed, T = Temperature on absolute scales, R = Gas constant, A = Frequency factor, a = Rate of heating in °C per minute, E = Energy of activation, and n = Order of the reaction.

Coats and Redfern's equation table 6, 8 and 10) also provides a method for the evaluation of energy of activation and thermal decomposition of metal soaps Coats and Redfern's equation for the thermal decomposition of a compound can be expressed as

The plots of Chromium (myristate, palmitate, and stearate) show the value of energy activation obtained from the slope of the curve lies in the range 18.75-27.62 k.cal mol^{-1} and the thermogravimetric data shown in the table 6, 8, and 10).

Table 5. Freeman-Corroll's treatment of thermogravimetricdata of Chromium Myristate

S.No.	(1/T) *10^5	-Δ(log Wr)	-Δ{log(Dw/Dt)}	-[Δ(1/T)/ (logWr)*10^4	Δ{log(Dw/Dt)}/ Δ(log Wr)
1	0.007463	2.3786	5.3187	31.3	2.236063
2	0.006849	2.3786	7.0419	28.7	2.960523
3	0.006369	2.3787	7.3027	26.7	3.070038
4	0.005952	2.3892	6.4282	24.9	2.690524
5	0.005682	2.379	7.4436	23.8	3.128878
6	0.005319	2.3791	7.5114	22.3	3.157244
7	0.005128	2.3806	7.2684	21.5	3.05318
8	0.004854	2.3839	7.056	20.3	2.959856
9	0.004739	2.4013	6.5182	19.6	2.714446
10	0.004673	2.4238	6.2379	19.2	2.573603
11	0.004525	2.4872	6.0285	18.1	2.42381
12	0.004367	3.1345	5.4887	13.9	1.751061
13	0.004274	3.1353	5.5181	13.6	1.759991
14	0.004049	4.0645	5.4968	9.9	1.352393
15	0.003861	4.1443	5.5102	9.3	1.329585
16	0.003759	4.4129	5.5355	8.4	1.254391
17	0.003704	5.3882	5.5779	6.8	1.035207
18	0.003559	6.1586	5.5951	5.7	0.908502
19	0.003509	4.8809	5.5611	7.1	1.13936
20	0.003448	3.4516	5.6284	9.9	1.630664
21	0.003425	0	5.6647		0

Table 6. Coats-Redfern's Treatment of Thermogravimetric data of Chromium Myristate

S.No.	Temperature (T) Å	1/T * 10^5	A	α/T^2 * 10^5	-[log(α/T^2]
1	134	746	0.001728	0.00957	7.01868
2	146	684	0.001749	0.00821	7.08566
3	157	636	0.001849	0.007505	7.1246
4	168	595	0.023542	0.00832	7.0795
5	176	568	0.002657	0.00855	7.0677
6	188	531	0.002812	0.00795	7.0996
7	195	512	0.005918	0.01554	6.8084
8	206	485	0.012632	0.02969	6.5273
9	211	473	0.047698	0.1069	5.9709
10	214	467	0.090959	0.1985	5.7022
11	221	452	0.201573	0.4126	5.3845
12	229	436	0.746445	1.4233	4.8466
13	234	427	0.746794	1.3636	4.8652
14	247	409	0.886285	1.4525	4.8378
15	259	386	0.902743	1.3456	4.8711
16	266	375	0.904068	1.2776	4.8935
17	270	370	0.904809	1.2411	4.9062
18	281	355	0.904917	1.1460	4.9408
19	285	350	0.90206	1.1105	4.9544
20	290	344	0.897268	1.0668	4.9719
21	292	342	0.904902	1.0685	4.9712

Table 7. Freeman-Corroll's treatment of thermogravimetric data of Chromium Palmitate

S.No.	(1/T) * 10^5	-Δ(log Wr)	-Δ{log(Dw/Dt)}	-[Δ(1/T)/ (logWr)*10^4	Δ{log(Dw/Dt)} / Δ(log Wr)
1	0.007576	2.6833	5.7447	28.2	2.140909
2	0.00641	2.6834	7.8538	23.8	2.926809
3	0.005682	2.6832	8.2924	21.1	3.090489
4	0.005291	2.6837	7.8761	19.7	2.934792
5	0.005263	2.6864	7.3151	19.5	2.723012
6	0.004854	2.6901	7.0947	18	2.637337
7	0.004739	2.6974	6.883	17.5	2.551716
8	0.004444	2.7031	6.7822	16.4	2.509045
9	0.004367	2.7227	6.5594	16	2.409153
10	0.004237	2.7617	6.3226	15.3	2.289387
11	0.003922	2.9102	5.9869	13.4	2.057213
12	0.003861	3.281	5.7723	11.7	1.759311
13	0.003831	3.5032	5.7395	10.9	1.638359
14	0.003704	4.1594	5.7399	8.8	1.379983
15	0.003676	5.2268	5.7892	7	1.107599
16	0.003597	4.5337	5.8176	7.9	1.28319
17	0.003571	5.1214	5.8449	6.9	1.14127
18	0.003559	4.5382	5.8761	7.8	1.294809
19	0.003509	4.4223	5.9059	7.9	1.335482
20	0.003497	4.7493	5.9277	7.3	1.248121
21	0.003472	0	5.9459		0

Table 8. Coats-Redfern's Treatment of Thermogravimetric data of Chromium Palmitate

S.No.	Temperature (T) Å	1/T * 10^5	α	α/T^2 * 10^5	-[log(α/T^2
1	132	757	0.000673	0.003862	7.4131
2	156	641	0.000889	0.003652	7.4373
3	176	568	0.000448	0.001446	7.8397
4	189	529	0.001395	0.003905	7.4083
5	190	526	0.007193	0.09925	6.7005
6	206	485	0.014965	0.03525	6.4528
7	211	473	0.030256	0.06794	6.1678
8	225	444	0.042085	0.03525	6.0802
9	229	436	0.081131	0.06794	5.8368
10	236	427	0.154125	0.08312	5.6282
11	255	392	0.378924	0.1456	5.2481
12	259	386	0.695516	0.2369	4.9909
13	261	383	0.789552	0.5648	4.9653
14	270	370	0.899073	1.0209	4.9153
15	272	367	0.927636	1.0829	4.9207
16	278	359	0.917175	1.2151	4.9314
17	280	357	0.926906	1.2002	4.9304
18	281	355	0.917309	1.1691	4.9472
19	285	350	0.913341	1.1738	4.9521
20	286	349	0.922309	1.1293	4.9325
21	288	347	0.930296	1.1165	4.9502

It is suggested that the decomposition reaction of Chromium soaps (myristate, palmitate, and stearate) is kinetically of zero order and the energy of activation for the process lies in the range of 18.75 – 32.33 k.cal per mole table 11.

Table 9. Freeman-Corroll's treatment of thermogravimetric data of Chromium Stearate

S.No.	(1/T) * 10^5	-Δ(log Wr)	-Δ{log(Dw/Dt)}	-[Δ(1/T)/ (logWr)*10^4	Δ{log(Dw/Dt)}/ Δ(log Wr)
1	724	2.5895	5.6486	27.9	2.181348
2	641	2.5895	6.1403	24.7	2.37123
3	606	2.5914	6.1734	23.3	2.382264
4	561	2.5918	6.2298	21.6	2.403658
5	529	2.5935	6.3087	20.3	2.432504
6	505	2.5955	6.0821	19.4	2.343325
7	456	2.627	5.8502	17.3	2.226951
8	444	2.6821	5.8031	16.5	2.16364
9	420	2.8475	5.7306	14.7	2.012502
10	411	2.9941	5.636	13.7	1.882369
11	401	3.5646	5.6317	11.2	1.579897
12	390	3.1689	5.6446	12.3	1.781249
13	359	3.0412	5.6851	11.8	1.869361
14	348	4.8624	5.7824	7.1	1.189207
15	343	4.8624	5.7654	7	1.185711
16	340	5.2084	5.8504	6.5	1.123262

Table 10. Coats-Redfern's Treatment of Thermogravimetric data of Chromium Stearate

S.No.	Temperature (T) Å	1/T * 10^5	A	α/T^2 * 10^5	-[log(α/T^2
1	138	724	0.004798	0.02515	6.5994
2	156	641	0.004861	0.01997	6.6995
3	165	606	0.008974	0.03294	6.4821
4	178	561	0.009889	0.03118	6.5060
5	189	529	0.013345	0.03737	6.4274
6	198	505	0.017745	0.04527	6.3441
7	219	456	0.082869	0.1728	5.7625
8	225	444	0.185733	0.3668	5.4355
9	238	420	0.426989	0.7536	5.1228
10	243	411	0.576056	0.9755	5.0107
11	249	401	0.847509	1.3369	4.8642
12	256	390	0.885864	1.3516	4.8691
13	278	359	0.914013	1.1826	4.9271
14	287	348	0.942294	1.150	4.9391
15	291	343	0.947636	1.1190	4.95
16	294	340	0.947322	1.0959	4.96

Table 11. Energy of activation (kCal mol^{-1}) for the decomposition of metal soaps by using various equations

S.No.	Name of the metal soap	Freeman and Carroll's equation	Coats and Redfern's equation
1.	Chromium Myristate	23.46	18.75
2.	Chromium Palmitate	27.13	21.48
3.	Chromium Stearate	32.33	27.62

Figures 7-9 depict thermograms illustrating the loss in weight (w) of Chromium soaps (myristate, palmitate, and stearate) plotted against time (t). The values of (dw/dt) are determined from the curves by drawing tangents at suitable intervals. By plotting Δ [log(dw/dt)]/ΔlogWr against Δ(1/T)/Δ(log Wr), a linear relationship emerges. The slope of this relationship facilitates the calculation of the activation energy for the decomposition process, while the intercept provides the value of 'n'. The reaction order was determined to be zero, and the activation energy values for the decomposition were found to range between 18.75 and 32.33 kJ mol^{-1}.

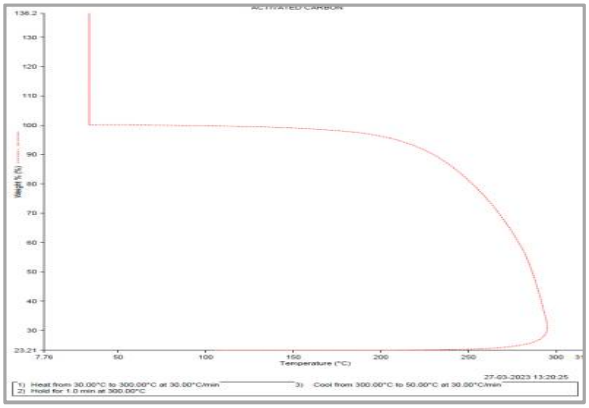


Figure 7. Thermogram of Chromium Myristate.

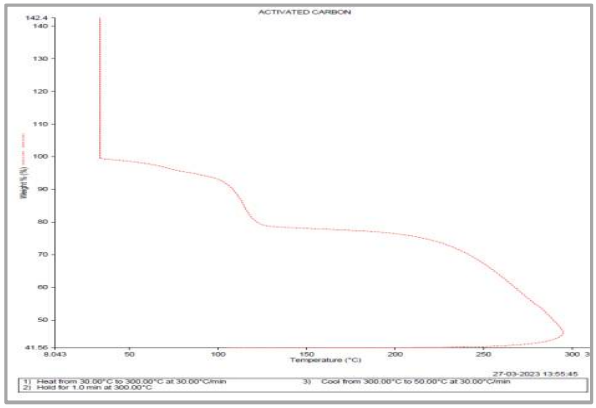


Figure 8. Thermogram of Chromium Stearate

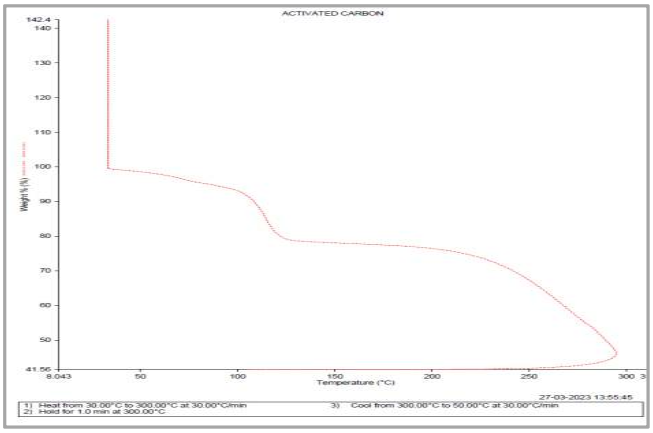


Figure 9. Thermogram of Chromium Palmitate

APPLICATION

Chromium soaps have valuable applications in the academic and technological fields and another important role in many diverse fields like as thickness, dryers, catalysts, paints, lubricants, plastics wetting agents, ink etc.

CONCLUSION

The IR results showed that fatty acid exists in a dimeric structure as a result of hydrogen bonding between the carboxyl groups of two fatty acid molecules, whereas chromium soaps possess an ionic character. The X-ray analysis showed that chromium soaps have a double layer structure with molecular axes slightly inclined to the basal plane. The thermal decomposition of these soaps was found to be zero order and the energy of activation for the decomposition process was in the range of 23.46-18.75, 27.13-21.48, and 32.33-27.62 KJmol^{-1}.

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