

# INFRARED ABSORPTION SPECTRA OF NICKEL BUTYRATE SOAP

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## ABSTRACT

The applications of metallic soaps are mostly depend on the conditions and the methods of the preparations of the metallic soaps, thus there is a great significance of the nature and structure of the metallic soaps for their uses in various industries under different conditions. The absorption bands have been observed near 2660-2650 cm<sup>-1</sup>, 1700-1670 cm<sup>-1</sup>, 1470-1460 cm<sup>-1</sup>, 960-930 cm<sup>-1</sup>, 690 and 550cm<sup>-1</sup> in the spectra of fatty acids have indicated the presence of localized -COOH group(20) in the form of dimeric structure and the existence of the intermolecular hydrogen bonding between two molecules of acids. The absorption bands observed near 2660-2650 cm<sup>-1</sup>, 1700-1670 cm<sup>-1</sup> and 960-930 cm<sup>-1</sup> corresponding to the -COOH group in the spectra of fatty acid having completely disappeared in the spectra of corresponding sodium and nickel soaps. The infrared spectra of nickel butyrate soap does not show any absorption maxima in the regions of 3500-3300 per cm which indicate that the absence of any coordinated water molecules in the soap molecules. Thus the results confirm that the fatty acid exist with dimeric structure through hydrogen bonding where as the metal to oxygen bonds in the metal soaps possess ionic character.

**Key Words:** metallic soap, Dimeric, Absorption, intermolecular hydrogen bonding, nickel soap

## INTRODUCTION

The metallic soaps are being used increasingly in various industries such as textiles, paints lubricants, greases, cements, cosmetics, emulsifiers, water-proofing agents, medicines, pharmaceuticals, germicides, printing inks, varnishes, catalysts, softeners, hardeners and stabilizers etc. Therefore, the applications of metallic soaps are mostly depend on the conditions and the methods of the preparations of the metallic soaps, thus there is a great significance of the nature and structure of the metallic soaps for their uses in various industries under different conditions.

On account of their unique characteristics, metallic soaps have vast importance in technological and academic fields. However, the applications of the metallic soaps are based on the empirical knowledge and the selection of for a specific purpose is mainly governed by economic factors. Several studies have been done on the alkali,

alkaline and transition metal soaps but less work has been done on the Nickel (Ni) butyrate soaps. Various methods and techniques have been used by several workers for studying the preparation, properties and uses of metal soaps (1-5).

The methods of preparation of rare earth metal soaps were reported by several workers (6-10). Ralston (11) prepared the lithium soaps of lauric, myristic, palmitic, and stearic acids by adding the calculated amount of lithium acetate to ethanol solution of the respective fatty acids and determined the solubility of those soaps in a number of solvents. The soaps of second series of transition metals were prepared by several workers (12) by treating the halides (generally chlorides) with carboxylic acids. Several workers reported the methods of preparation of transition metal soaps by double decomposition, metathesis and fusion or by direct reaction of metal oxide with an organic acid (13-15).

## II. METHODOLOGY

In this study, first the fatty acids were purified by distilling under reduced pressure. The purity of acids was checked by determining their boiling points, melting points. The m.p. of purified acids were; 162<sup>o</sup> C of butyric acid, 185<sup>o</sup> C of caproic acid, 238<sup>o</sup> C of caprylic acid. Then used solvent 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 points of purified benzene was 80.1<sup>o</sup>C. Methanol was purified by keeping over KOH for twenty four hours and then distilling. The distillate was refluxed with 1% of calcium metal for about eight hours and redistilled. The boiling point of purified methanol was 65.0<sup>o</sup> C.

Sodium soaps (butyrate) was prepared by refluxing equivalent amounts of corresponding fatty acid aqueous solution of sodium hydroxide for 6-8 hours on a water bath. The soaps were purified by recrystallization with methanol and dried under reduced pressure. The purity of soaps was checked by the determination of their melting points. Nickel butyrate soap was prepared by direct metathesis of the corresponding sodium soap with slight excess of the solution of nickel nitrate under vigorous stirring. The precipitated soaps were washed with water, methanol and acetone to remove the excess of metal salts, sodium soap and unreacted fatty acid. The soap thus obtained was dried in an air oven and the final drying of the soaps were carried out under reduced pressure. The soaps were purified by recrystallization with benzene-methanol mixture. The purity of the soap was confirmed by determination of their melting point. The elemental analysis for C, H, and metal are further confirmed by the molecular formula of soaps. The melting point of Nickel butyrate soap is 112<sup>o</sup>C.

The calculated amount of soap was weighed in a standard flask and the solution was made by adding the required amount of solvent. In this way different amount of Nickel soap was prepared. Care was taken to avoid trace of water, which were found on hydrolysis of soap. Further, IR Absorption Spectra of Nickel butyrate soap and of corresponding fatty acids were obtained with a Perkin-Elmer 577 Model Grating Spectrometer in the region of 4000-400 cm<sup>-1</sup> using KBr disc method.

### III. RESULTS AND DISCUSSIONS

The infrared absorption maxima of nickel butyrate soap have been assigned and recorded in figure-1 and compared with those of corresponding fatty acid and sodium soaps. The vibrational frequencies characteristics of the aliphatic portion of the fatty acid do not vary even when acid is converted into sodium or nickel soap. The absorption bands of –C-H stretching vibrations(16-17) viz. the symmetrical vibration of –CH<sub>2</sub>, the unsymmetrical stretching vibrations of –CH<sub>2</sub>, the asymmetrical vibrations of –CH<sub>3</sub> and the deformation of –CH<sub>3</sub> are observed at 2865-2850 cm<sup>-1</sup>, 2930-2900 cm<sup>-1</sup>, 2955-2940 cm<sup>-1</sup> and 1370-1350 cm<sup>-1</sup> respectively in the spectra of metal soap as well as in corresponding fatty acids. The progressive bands (18-19) of weak intensity are observed near 1340-1040 per cm in the spectra of nickel soap corresponding to the wagging and twisting vibrations of chains of successive methylene groups in their molecules.

The absorption bands have been observed near 2660-2650 cm<sup>-1</sup>, 1700-1670 cm<sup>-1</sup>, 1470-1460 cm<sup>-1</sup>, 960-930 cm<sup>-1</sup>, 690 and 550cm<sup>-1</sup> in the spectra of fatty acids have indicated the presence of localizes –COOH group(20) in the form of dimeric structure and the existence of the intermolecular hydrogen bonding between two molecules of acids. The absorption bands observed near 2660-2650 cm<sup>-1</sup>, 1700-1670 cm<sup>-1</sup> and 960-930 cm<sup>-1</sup> corresponding to the –COOH group in the spectra of fatty acid having completely disappeared in the spectra of corresponding sodium and nickel soaps. The absorption maxima near 690 cm<sup>-1</sup> and 50 cm<sup>-1</sup> in the spectra of fatty acids were assigned to bending and wagging mode of vibration of carboxyl group of acid molecules and were not observed in the spectra of metal soaps. The IR spectra of nickel and sodium soaps show marked differences with the spectra of corresponding fatty acids in some spectral regions. The IR spectra of nickel butyrate soap does not show any absorption maxima in the regions of 3500-3300 cm<sup>-1</sup> which indicate the absence of any coordinated water molecules in the soap molecules. The infrared absorption spectral frequencies (cm<sup>-1</sup>) of Nickel butyrate soap with their assignment were given below in Table-1.

**Table-1. Infrared absorption spectral frequencies with their assignment (cm<sup>-1</sup>) of Nickel butyrate soap.**

S.No.	Absorption	Butyric Acid	Sodium Butyrate	Nickel Butyrate
1.	CH <sub>3</sub> , C-H asymmetrical stretching	2940	2950	2964
2.	CH <sub>2</sub> , C-H asymmetrical stretching	2900	2910	2931
3.	CH <sub>2</sub> , C-H symmetrical stretching	2850	2860	2877
4.	OH, stretching	2650		
5.	C=O stretching	1670		

6.	COO <sup>-</sup> , C-O asymmetrical stretching		1550	169
7.	COO <sup>-</sup> , C-O symmetrical stretching		1430	1418
8.	CH <sub>2</sub> , (adjacent to COOH group)	1410	1420	
9.	C-O stretching, OH in plane deform	1460		
10	Progressive bands (CH <sub>2</sub> twisting and wagging)	1280-1040	1320-1060	1311-1218
11.	CH <sub>3</sub> Rocking	1100	1070	1102
12.	OH out of plane deformation	930		
13.	CH <sub>2</sub> , Rocking	720	730	769
14.	COOH, Bending mode	690		
15.	COOH, Wagging mode	550		
16.	Ni-O bond			455

#### IV. CONCLUSIONS

The infrared spectra of sodium, and Nickel soaps show marked differences with the spectra of corresponding fatty acids in some spectral regions. It is therefore, concluded that the resonance character of the ionized carboxyl group is retained in these metal soaps and the metal to oxygen bond is ionic in nature. The infrared spectra of nickel butyrate soap does not show any absorption maxima in the regions of 3500-3300 per cm which indicate that the absence of any coordinated water molecules in the soap molecules. Thus the results confirm that the fatty acid exist with dimeric structure through hydrogen bonding where as the metal to oxygen bonds in the metal soaps possess ionic character.

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## REFERENCES

1. J. H. Skellon and J. W. Spence, *J. Appl. Chem.*, 3, 10, 1953.
2. R. C. Mehrotra, *Z. Wiss, Friedrich-Schiller, Univ. Jena, Math. Naturwiss, Rath.*, 14, 171, 1965.
3. L.D.Skrylev, V.F. Sazonova, M.E. Kornelli and N. A. Sumitina, *Khim Khim. Tekhnol*, 21, 149, 1978.
4. A.M.Bhandri, S.Dubey and R.N.Kapoor, *J. Am. Oil Chem. Soc.*, 4, 47, 1970
5. A.K. Solanki and A.M. Bhandari, *Tenside Detergents*, 18, 34, 1981.
6. K.N. Mehrotra and S.K. Upadhyay, *J. Chem. Engg. Data*, 33, 468, 1988.
7. K.N. Mehrotra and S.K. Upadhyay, *Pol.J. Chem.*, 65, 1035, 1991.
8. K.N. Mehrotra, R. Shukla and M. Chauhan, *J. Bull. Electrochem.*, 4(5), 513, 1988.
9. K.N. Mehrotra, M. Chauhan and R. K. Shukala, *Phys. Chem. Liq.*, 21, 239, 1990.
10. G. Courtious, *Compt. Rend.*, 158, 1511, 1914.
11. R. N. Kpoor and R. C. Mehrotra, *Chem. And Ind.*, 68, 1958.
12. R.N. Kapoor and R. C. Mehrotra, *J. Chem. Soc.*, 422, 1959.
13. O. Ohari, Koichi and Sakurna, *Yasumika, Jpn. Kokai, Tokoyo kho J P* , 08, 73, 1966.
14. De Navare, Maison Selommon, Michel, *L. 3 (BK2)*, 553, 2002.
15. Imori, Toru, Yashida Mizuho, *J P PCT Int.* 28, 1999.
16. K. Nakanishi, *Infrared absorption spectroscopy*, Holden Day, San Francisco, P. 14, 1977.
17. R.M. Silverstein, G.C. Bassler and T. C. Morrill, *Spectrometric identification of organic compounds*, John Willey & sons, 14, 107, 1981.
18. R. N. Jones, A. F. McKay and R. G. Sinclair, *J. Am. Chem. Soc.* 74, 2575, 1952.
19. R. A. Mulliken, R.J. Meyer, S.M. Ronouci, H. A. Schnette and V.W. Meloch, *Anal. Chem.*, 29, 39, 1953.
20. K. Nakanishi, *Infrared absorption spectroscopy*, Holden Day, San Francisco, P. 39, 1977.