

# Structural Characterization of Some Table Salt Samples by XRD, ICP, FTIR and XRF Techniques

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In this study, we have investigated structure and impurities of six different salts which have a great importance in our everyday life, namely source salt, Himalayan salt, rock salt, sea salt, lake salt and reduced sodium salt (mainsalt) by means of different analytical methods, namely X-ray powder diffraction, scanning electron microscope–energy dispersive X-ray spectroscopy, inductive coupling plasma optic emission spectroscopy, X-ray fluorescence spectrometer and the Fourier transform infrared spectroscopy. With the light of obtained results, suitability and performance of the different analytical methods were discussed in some details.

PACS: 61.05.cp

## 1. Introduction

Salt is one of the oldest minerals discovered and used by human. It has been used to sweeten and hide of nutrition from old. Depending on the formation, salts may be named as lake, sea, rock, source, Himalayan salt. Lake salt is obtained from old sea areas or from salty water passing over rock salts and forming lakes in appropriate areas. Sea salt is obtained from sea. In this study, sea salt was obtained by evaporation of sea water collected from Aegean region. Rock salt is found as solid under soil. This salt is formed by evaporation of inland sea water in hot climate condition with time. Source salt is obtained from river, well and sources [1].

In a study, mineralogical investigation of samples taken to determine of heavy metal quantities were conducted using X-ray powder diffractometer (XRD) system and geochemical analyses of samples were determined using atomic absorption spectrometer with graphite oven. Using the methods mentioned above, the concentrations of Cu, Al, Pb, As, Hg, Fe, Cr, Se, Cd, Co, Mn, Ni were compared. X-ray diffraction analysis showed that the samples mainly include halite (NaCl), whereas, according to X-ray powder diffraction analysis results, the samples also include CaCl<sub>2</sub> at much less amount [2]. Lake salts may also include some heavy metals, such as Fe 1.27 – 1.55 mg/l, Pb 0.33 – 0.67 mg/l, Mn 0.15 – 0.17 mg/l, Cu 0.11 – 0.20 mg/l, Zn 0.02 – 0.08 mg/l [3].

There are various methods used for structural analyses. Hannaker et al. [4] compared inducting coupling plasma–optic emission spectroscopy and X-ray fluorescence (ICP–OES and XRF) methods in the analyses of some geologic samples. In wide concentration interview, they used a lot of elements. In wide concentration examination, they used many elements and stated that analysis results were similar to the results obtained by ICP–OES and XRF. Somogyi et al. [5] performed Mn, Zn, Fe and Sr analyses in sedimentary rock samples in marsh using XRF and ICP. XRF and ICP gave similar results

in Mn, Zn and Fe analyses, but ICP gave lower standard deviations in Sr analyses. Marina and Lopez [6] compared with XRF and ICP methods of phosphorus amounts in ceramic raw material. They reported that there are obtained quotable results by both methods and sensitivity of XRF is high due to the fact that XRF account for smaller error in sample preparation. In this study, by using XRD, SEM/EDX, ICP, XRF and the Fourier transform infrared (FTIR) structure of this salt samples were illuminated.

## 2. Method

In this study, the characterization of the salts is carried out with a number of experimental approaches in order to investigate all the relevant features. XRD patterns of the salts were obtained by a Rigaku model powder X-ray diffractometer with Cu K alpha radiation. Also, by using ZEIS EVO 50 scanning electron microscope, elemental analyses of these salts were performed. By using Perkin Elmer Optima 5300 DV optic emission spectrometer (ICP), chemical elements in salts were determined. The salts were subjected to wavelength distribution X-ray fluorescence spectrometer (WD/XRF) to analyze the chemical composition or elements present in the sample. The salts were investigated for their vibration spectra with infrared spectroscopy using Perkin Elmer 1800 model instrument from 450 cm<sup>-1</sup> to 4400 cm<sup>-1</sup> by using KBr tablet.

## 3. Results and discussion

In order to understare the nature of the salts, XRD, SEM (EDX), ICP, XRF and FTIR studies were performed. The presences of above minerals were further tested by XRD studies (Fig. 1). XRD is used to determine the mineralogical composition of the raw material components as well as qualitative and quantitative phase analysis of multiphase mixtures. XRD analyses were used

to determine the structure of salts. Halite potassian was observed in all salt samples (Table I).

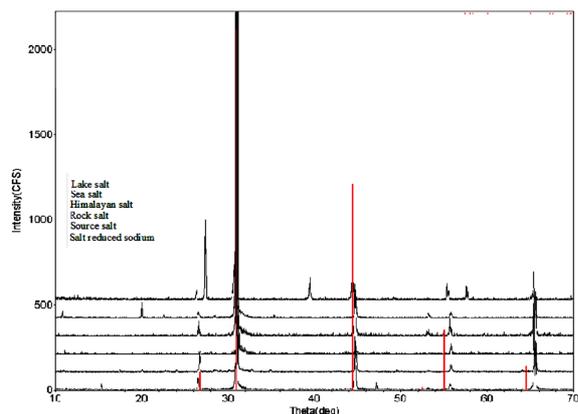


Fig. 1. XRD patterns of table salts.

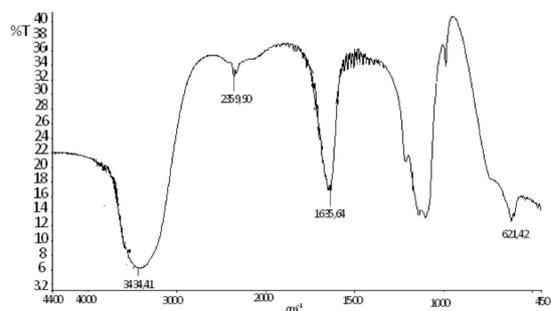


Fig. 2. FTIR spectrum of lake salt.

Secondly, structure of these salts was investigated by SEM/EDX. This is given in Table II. ICP results are shown in Table III. As known from literature, in structure of the salts, Na and Cl are found. In our study, Na has been found for all salts.

XRF results of samples are shown in Table IV.

FTIR studies of the salts help in the identification of minerals present in the salts. The coupled vibrations are appreciable due to availability of various constituents. In

Fig. 2, FTIR spectrum of lake salt is shown.

In all samples, there are 3410, 3452, 3420, 3446, 3434, 3468 and 1635 peaks. These peaks come from water in the structure of the sample. We estimated that 2360 value comes from carbon dioxide in air and it has been shown in all samples (Table V).

X-ray diffraction studies show the presence of halite potassian. XRF and ICP analyses clearly indicate that the higher rate of sodium (for ICP) and chlorine (for XRF) than other elements is predicted. Results have complemented features to each other.

XRD results.

TABLE I

Salts	Matter determined	PDF number
source	halite potassian, syn	00-026-0918
Himalayan	halite potasian, syn	01-075-0305
minsalt	halite potassian, iron potassium fluoride	01-075-0304, 01-076-2399
rock	halite potasian, syn	01-075-0305
sea	halite potasian, syn	01-075-0305
lake	halite syn	01-089-3615

SEM/EDX analysis results.

TABLE II

Elem.	C norm. [wt.%]					
	lake	sea	Himal.	rock	source	minsalt
Cl	48.25	38.84	45.60	20.10	53.09	48.09
Mg	18.24	43.34		16.09		5.13
S	17.50	4.12		6.55	0.02	
K	6.04	3.42		2.61		44.56
Ca	4.37		0.29	3.38	0.58	0.83
Na	4.08	4.55	53.64	23.71	46.31	1.40
Al	0.71	5.73	0.47	9.13		
Si	0.49					
Ni	0.31					
Cu				18.43		

ICP measurement results [mg/kg].

TABLE III

Salts	Al	Ca	Cu	Fe	K	Mg	Na	P
	396.153	317.933	327.393	238.204	766.490	285.213	589.592	214.914
source	< 5	2123.5	< 5	17.78	265.15	144.85	374150	2.275
Himalaya	< 5	178.1	< 5	5.59	552.3	< 5	378700	< 5
minsalt	< 5	92.36	< 5	5.818	244800	1434	99455	< 5
rock	< 5	1044.75	< 5	11.595	283.95	89.56	368900	2.197
sea	5.629	7944	< 5	13.465	9278.5	10395	325900	3.421
lake	3.213	1827.5	< 5	8.469	4048	3379	339000	< 5

XRF results [%].

TABLE IV

Salts	Cl	Na <sub>2</sub> O	CaO	SO <sub>3</sub>	K <sub>2</sub> O	MgO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	Br	SrO
source	46.07	32.69	1.24	1.39	0.079	0.416	0.0937	0.042	0.0114	–	0.0088
Himalayan	40.76	35.52	0.050	0.161	0.053	0.649	0.0594	0.025	0.0176	–	–
minsalt	30.19	7.59	0.053	0.023	26.84	0.873	0.0218	0.007	–	0.0158	–
rock	42.24	29.29	1.253	1.112	0.411	0.364	0.1951	0.115	0.0314	0.0088	0.0038
sea	3.096	1.79	0.218	0.547	0.170	0.626	0.0093	0.005	0.0022	0.0031	0.0019
lake	37.92	20.43	0.769	2.323	1.034	5.129	0.1474	0.084	0.0180	0.0156	0.0078

FTIR spectrum bands [cm<sup>-1</sup>].

TABLE V

Sea salt	Source salt	Rock salt	Lake salt	Himalayan salt	Minsalt
3851.97					
3410.03	3420.68	3446.96	3434.61	3468.68	3452
2360.16	2361.66	2360.30	2359.90	2360.26	2360.52
1635.73	1635.91	1635.96	1635.64	1635.96	1636.08
1520.90					
					1488.55
1436.78					1423.62
1124.58	1118.00		1102.65		1119.68
605.86	603.62	668.10	604.17	668.11	594.91

### Acknowledgments

Authors thank the HÜBAK for financial support, Harran University Central Lab. (HÜMEL) for XRD and ICP measurement, Harran University, Faculty of Arts and Science, Department of Chemistry for FTIR measurement, Erciyes University (TEKMER) for XRF measurement.

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