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Evaluation of heavy metal content of salts between Eskıkışla and Ocakbaşı (Kırıkkale) villages

Bilgehan Yabgu Horasan *1, **Alican Ozturk** 2

1Selcuk University, Sarayonu Vocational High School, Environmental Protection Technologies, Konya, Turkey
2Konya Technical University, Faculty of Engineering and Natural Sciences, Geological Engineering Department, Konya, Turkey

Keywords
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ABSTRACT

Salt, which is the main nutrient, is a very important raw material in the life of living organisms and industry. Salt production and consumption have improved and increased due to technological developments and industrialization. Salt has been used in the world and in our country since very ancient times. It is used in many fields, especially in the chemical industry, metallurgy, textile, medicine, and agriculture. In addition to sea, lake and spring salts, Turkey has very rich rock salt reserves. The Neogene aged Çankırı-Çorum Basin is one of the important basins where evaporitic formations are observed in the Central Anatolia region. Chemical analyses were carried out to determine the amount of heavy metal in salt samples taken from Oligocene aged units observed in the area between Ocabaşı and Eskikışla springs (Delice) in the Çankırı-Çorum basin. SEM-EDX, XRF and ICP-OES devices were used to determine the geochemical properties and quantities of heavy elements. According to the results of XRF, Na % (33.70-38.30), Cl %(52.10 - 57.70) SO_3 %(0.75-1.93), Al_2O_3 %(0.90-1.93), CaO %(0.58-2.69), MgO %(0.40-1.41) Fe_2O_3 % (0.18-0.64), SiO_2 %(1.75-4.95), SrO, % (<0.010), BaO % (<0.010), Cr_2O_3 %(<0.010), K_2O %(0.008-0.25), MnO %(<0.010) results are found. In the determination of heavy metals made with ICP-OES, it was determined that the heavy metals (Hg, As, Pb, Cd) published in the Turkish food codex salt communiqué were below the limit values.

1. INTRODUCTION

Salt, which is known to have been consumed throughout human history, is known to be a source of income and trade in Chinese civilization, meat and fish salting in Egyptian civilization, a means of paying taxes and salaries to soldiers in Romans, an order of wealth in Renaissance Europe and a sacred food in Central Asian Turkish culture (Caglar 2015). Evaporites are rocks that chemically precipitate as a result of the evaporation of saturated and salty water on the surface or near the surface with the effect of sun rays. Because they provide important information about paleoclimate and environmental conditions and have an economic value industrially, they are important deposits (Altay 2010). They have a wide range of uses, especially in the salt chemical industry, metallurgy, textile, medicine, and agriculture (Donat 2016). Salt, which is known to have been used for different purposes by civilizations in human history, continues to be used by humanity today without losing its importance. Salt is obtained from the ocean (sea waters), lake salts and rock salts (U.S. 2018). Turkey is a country that is self-sufficient in salt production and consumption in 2015, it has received $ 11 million in revenue by exporting salt to 64 countries (Donat 2016). China ranks first in salt production and export in the world, followed by the USA, India, Canada, and Australia (U.S. 2019). The world's largest producer of salt, China, USA, and India, meet a significant portion of salt demand. Among the salt-producing countries on the European continent, Germany leads, followed by Turkey, France, Spain, and Poland. Especially during the harsh winter months, the amount of production is increasing, and most of the salt needed by the European continent is met by these countries. Rock salt (halite), an odorless, water-soluble, easily friable material, consists of Na^+ and Cl^- ions and crystallizes in the cubic system. Although it is colorless in its pure form, it can be in different colors (green, gray, yellow) depending on the environment. The deposits of minerals are different in waters of terrestrial origin and waters of marine origin. The change of molar ratios in seawater creates important changes in the type of minerals and order of the deposition of potassium minerals. Evaporite minerals formed by evaporation of terrestrial waters are more diverse than evaporite minerals formed in waters of

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* **Corresponding Author**

*(bilgehanyabgu@gmail.com) ORCID ID 0000 – 0003 – 1899– 0041
(alicanozturktr@gmail.com) ORCID ID 0000 – 0003 – 2748 – 6322

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marine origin. Borates with economic value, natural soda, and sodium sulfate minerals are formed in a terrestrial environment (Eugster and Hardie 1978; Altay 2010) Operable salt sources are divided into solid and liquid. The areas where liquid production is carried out are oceans, seas, lakes, and water sources containing salt. The areas where solid production is carried out are buried salt deposits. In this context, rock salt deposits observed in the area between Ocakbaşı and Eskikisla plateaus (I - 31 - B3 map) located in the borders of Delice District of Kırıkkale province were investigated and their geochemical properties were examined (Figure 1). In addition, the heavy metal content in the composition of rock salt in the region was evaluated according to the Turkish food index.

2. GEOLOGICAL SETTINGS

The Neogene aged Çankırı-Çorum Basin, located within the Anatolide Tectonic Union, is surrounded by the ophiolitic melange in the north and west, and the Kırşehir Massif in the south (Varol et al. 2004). Old and young tectonic scars are seen in the region (Figure 1). Traces of old and young tectonics are seen in the area. For this reason, the stratigraphy of the underlying rocks is complex. Basin by many researchers (Dellaloğlu 1973; Birgili et al.1975; Seymen 1981; Ünalan and Harput 1983; Kara and Dönmez 1990; Tüysüz and Dellaloğlu 1992; Erdoğan et al. 1996; Seyitoğlu et al. 1997; Varol et al. 2004) have been studied for various purposes. The generally accepted opinion of Tüysüz and Dellaloğlu (1992) regarding the formation of the Çankırı-Çorum Basin is as follows: Çankırı-Çorum Basin, a basin formed after the collision between the two continents on the Izmir-Ankara-Erzincan suture as a result of the collision between Sakarya and Kırşehir in the time interval between late Paleocene(?) early Eocene. The basic units of the basin are Kırşehir Continent, Sakarya Continent, and İzmir-Ankara-Yozgat suture zone. On this foundation, there are units of the Tertiary-aged Çankırı-Çorum Basin. The bottom sedimentary units of the Çankırı-Çorum Basin are Maastrichtian and Paleocene turbidites (Karadenizli 2011; Norman 1972; Kazancı and Varol 1990). The Paleogene part of the filling in the basin consists of marine clastic and the Neogene part consists of terrestrial, clastic and evaporitic rocks. The facies of this filling, which are different and thick, belong to Eocene and Oligocene (Birgili et al. 1975) The Oligocene aged Shin formation, in which salt deposits are found includes massive-stratified conglomerates, parallel and cross-stratified sandstones, stratified siltstones and massive mudstones (Karadenizli 2011).

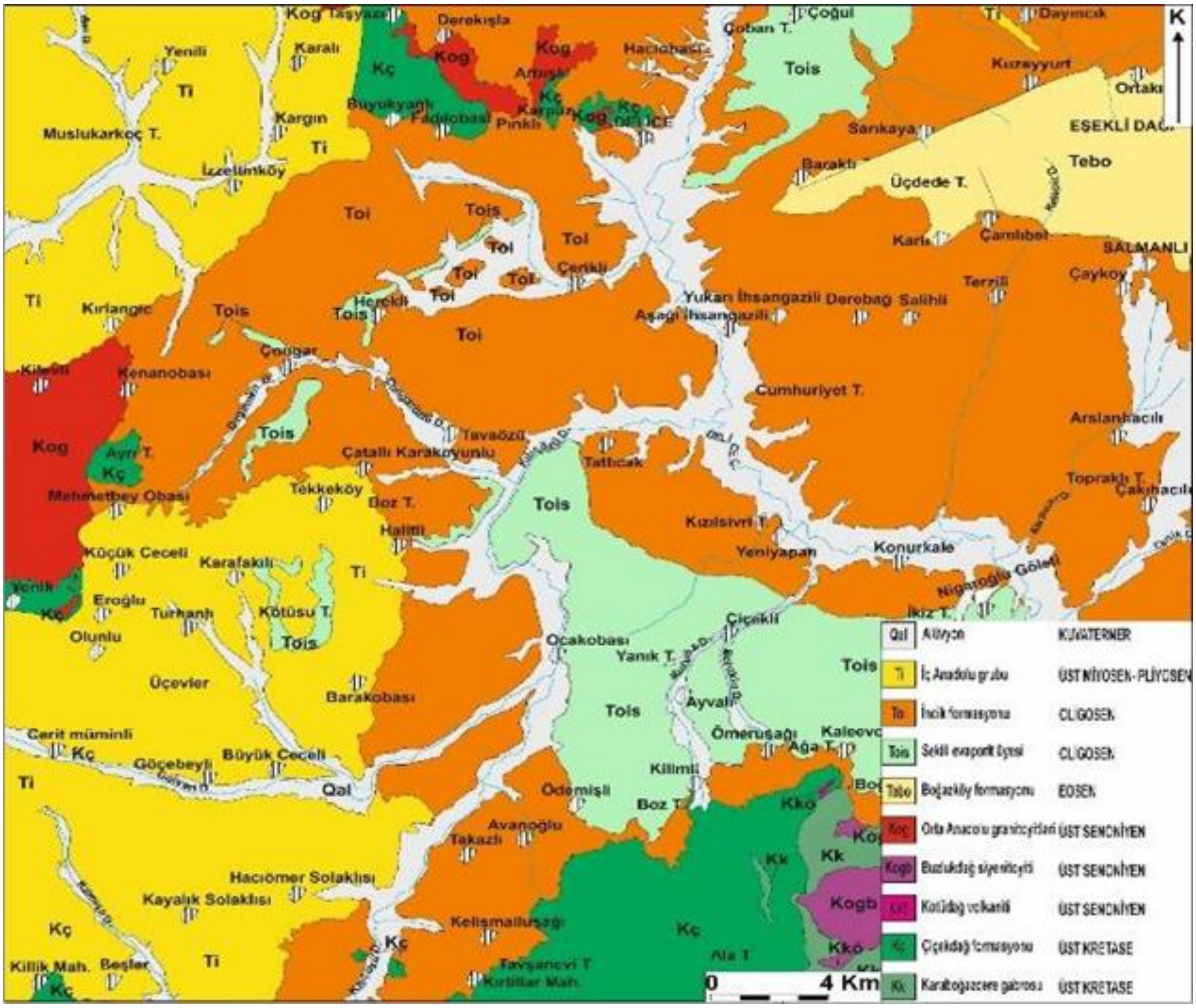


Figure 1. Geological map of the study area and its environs (taken from Dönmez et al. 2005).

2.1. İncik Formation (Toi)

The unit outcrops in large areas in the field and consists of evaporite, red, brown and gray colored, parallel cross-layered, low-cornered round grained, medium-well, loosely affixed terrestrial conglomerate, sandstone and mudstone intercalations. The formation is composed of clastic rocks (Birgili et al. 1975) is called as İncik formation. The lower parts of the İncik formation are intercalated with thin-medium-thick parallel-bedded sandstones, interbedded with medium-well bonded sandstones, gypsum and anhydrite mudstones, and the middle and upper levels are intercalated with mudstones and increasing upwards, cross-layered conglomerate and sandstone (Özgüner and Kızıldağ 2015). Investigation of oil and salt relationship in the study area and its vicinity (Tepecik 2007), investigation of glauberite-halite coexistence in the region (Sönmez 2014) and Halite (NaCl), Gypsum ($CaSO_4$) studies have been carried out for different purposes for salt deposits such as Anhydride ($CaSO_4.2H_2O$), Globerite ($CaNa_2$ $(SO_4)_2$), Tenardite (Na_2SO_4) detection of minerals (Horasan and Ozturk 2019).

2.2. Sekili evaporit member (Tois)

The red, brown, gray, light green, white and variegated colored, medium-thick evaporite, mudstone and sandstone intercalation of the İncik formation is named Sekili evaporite member by Kara (1991). The unit outcrops in the middle and northwest of the Kırşehir-İ31 and İ32 section. The shaped evaporite member is generally composed of anhydrite and gypsum and contains sandstone, and marl intermediate bands and mudstone upward. Gypsum and anhydrides are white and yellowish-white in color, medium-thick bedded, generally pure and crystalline and occasionally lumpy.

It is observed that gypsum and anhydrides are thinner in the lateral direction and mudstones thicken. Sandstone and mudstones are generally thin to medium in thickness and have red-brown, light green and variegated appearance in green colors (Dönmez et al. 2005).

3. MATERIALS AND METHODS

In this study, salt formations observed in the area between Ocakbaşı and Eskikışla springs in Delice district of Kırıkkale province were investigated. The geology of the study area was examined and mapped by using the previous studies. A total of 14 samples were collected during the field studies. Of these samples, 8 Salt (Halite), 2 Gypsum and Anhydrite samples X-RF, 2 Salt (Halit) samples, SEM - EDX and 2 Salt (Halite) ICP-OES and studies were performed. The analyses were carried out in METU MERLAB, Selçuk University İLTEK and Konya Food Agriculture University laboratories.

Textural examinations of SEM-EDX, were performed with Zeis Evo / Is20 brand scanning electron microscope, which is located in Selçuk University Advanced Technology Research and Application Center (İLTEK). The samples taken from the study area were analyzed with 123 eV energy by Bruker brand EDX detector.

X-Ray Fluorescence (XRF) spectrometer is one of the important devices used to determine the elemental and chemical composition. In this study, standard methods were analyzed with Rigaku ZSX Primus II device within METU merlab.

With the ICP-OES, heavy metals contained in salt were analyzed with Perkin Elmer 7000 DV Model device.

3.1. Evaluation of X-RF Results

Chemical analyses were performed on the samples collected from the Sekili member of İncik formation in the study area. Chemical analyses were carried out using X-RF to calculate Na, Cl, SO_3, Al_2O_3, CaO, MgO, Fe_2O_3, SiO_2, SrO, BaO, Cr_2O_3, K_2O, MnO. Na^+ and Cl^- ratios were found to be very high. K_2O value of the main oxides was found to be very low. The lowest Na (33.70%) and Cl (52.10%) values were determined in the Nkt-3 sample and the highest Na (38.30%) and Cl (57.70%) values were determined in Nkt-5 sample. As a result of the alteration occurring in the ophiolitic rocks at the base of the basin, the Mg formed is transported to the sedimentation environment with the effect of groundwater and surface waters, and the magnesite can precipitate at the increasing water level in the lake. In the Sabkha environment, the Halite mineral can be crystallized both within the matrix and individually. Halite can also be deposited by evaporation in an aqueous environment in the lake water (Sönmez 2014). In the field of investigation; Halite (NaCl), Gypsum ($CaSO_4$), Anhydride ($CaSO_4.2H_2O$), Globerite ($CaNa_2$ $(SO_4)_2$), Tenardite (Na_2SO_4) minerals were determined (Horasan and Ozturk 2019).

In the Matrix, clay minerals, Quartz, Serpentine group minerals and minerals of detritic origin can be observed. The lake environment has been fed by both groundwater and above ground waters, due to seasonal changes. Ion enrichment in the lake (Na^+, Cl^-) sedimentation took place as a result of evaporation in the lake during the arid period of the lake and the claystone was deposited in the open conditions in short and long periods in the lake. Successive formation of Halite, anhydrite, and plaster occurred due to the chemistry and temperature of the medium due to seasonal changes (Sönmez 2014). Samples of gypsum ($CaSO_4.2H_2O$) and anhydrite ($CaSO_4$) found in clay bands with glassy brightness and fibrous surfaces were taken at the examination area and chemical analyses were done with XRF (Table 2, 3.)

The dominant gypsum observed in the evaporite basins can be grouped under two headings as primary and secondary. The concept of primary gypsum; sedimentologically formed in the lake or mud plaster undergoes no diagenetic changes that have been used for the preservation of the primary physical and textural properties (Helvacı 2002). Secondary gypsum was generally formed by the intake of anhydrides. There are three formation mechanisms of gypsum formation by anhydride hydration.

These three formation mechanisms;

- Hydration of anhydride into gypsum by taking water into the crystal cage
- Hydration via Basanite series (Step method)

-Formation of plaster by redissolution of anhydrite

The formation of plaster is possible by direct ingress of water directly into the anhydride crystal lattice. The plaster formed in this way is alabastrine and is called secondary gypsum (Helvacı 2002).

Anhydride ($CaSO_4$) is generally characterized by its modular and electrolytic structures in the sabka environment. Since anhydrite minerals are not stable in surface conditions, they are converted to gypsum by the effect of meteoric waters (Helvacı 2002).

Table 1. Results of chemical analysis with XRF on samples taken from the field (Tois)

	Na	Cl	SO_3	Al_2O_3	CaO	MgO	Fe_2O_3	SiO_2	SrO	BaO	Cr_2O_3	K_2O	MnO
Unit	%	%	%	%	%	%	%	%	%	%	%	%	%
1	36.95	57.15	0.45	0.99	0.78	0.73	0.28	2.63	<0.010	<0.010	<0.010	0.12	<0.010
2	37.55	57.40	0.33	0.90	0.68	0.71	0.29	2.10	<0.010	<0.010	<0.010	0.098	<0.010
3	33.70	52.10	2.17	1.93	2.69	1.41	0.64	4.95	<0.010	<0.010	<0.010	0.20	0.010
4	36.75	56.05	0.63	1.05	0.90	0.88	0.40	3.20	<0.010	<0.010	<0.010	0.10	<0.010
5	38.30	57.70	0.18	0.75	0.58	0.40	0.18	1.75	<0.010	<0.010	<0.010	0.060	<0.010
6	34.90	53.61	2.18	1.40	2.35	1.12	0.45	3.68	<0.010	<0.010	<0.010	0.25	0.015
7	37.10	56.02	0.38	0.95	0.90	0.67	0.38	2.60	<0.010	<0.010	<0.010	0.10	<0.010
8	37.10	55.80	0.58	1.13	0.87	0.70	0.40	3.20	<0.010	<0.010	<0.010	0.12	<0.010

Table 2. XRF element results (Tois)

Component	Gypsum (%)	Anhydride (%)
O	49.3	48.7
Ca	27.8	28.6
S	20.1	20.6
C	1.52	1.27
Si	0.66	0.40
Al	0.32	0.14
Fe	0.16	0.091
Mg	0.088	0.078
K	0.048	0.041
Sr	0.035	0.020

Table 3. XRF Anaoxide analysis results (Tois)

Component	Gypsum (%)	Anhydride (%)
SO_3	51.4	52.5
CaO	40.4	41.3
CO_2	5.64	4.72
SiO_2	1.45	0.887
Al_2O_3	0.629	0.270
Fe_2O_3	0.240	0.116
MgO	0.149	0.113
K_2O	0.0604	0.0695
SrO	0.0439	0.0259

3.2. Evaluation of ICP-OES Results

The chemical composition of the brine is linked to the geology of the drainage basin surrounding the lake. Ions concentrated in the lake by evaporation are transported in solutions by streams and groundwater as a result of the decomposition of the surrounding rocks (Hüseyinca 2015). Jones and Deocampo (2003) explained the degradation reactions of certain rocks that are effective in the source areas and their relationships with the main anions and cations. For example, limestones Ca and HCO_3, dolomites Mg, magmatic and metamorphic rocks, depending on the composition of silica-rich Ca-Na-HCO_3, pyrite shales from sulfur-rich waters, hydrothermal waters are rich in SO_4 and basic and ultrabasic rocks are rich in Mg-HCO_3. Apart from these physical factors, another factor affecting the chemistry of lake water is the chemical precipitation and dissolution reactions, because the minerals that reach saturation and precipitate in saltwater decrease the concentration of ions that make up them and cause the saltwater to be enriched by other ions. Conversely, water increases in the lake can dissolve the minerals that have settled and change the chemistry of the saltwater (Smoot and Lowenstein 1991).

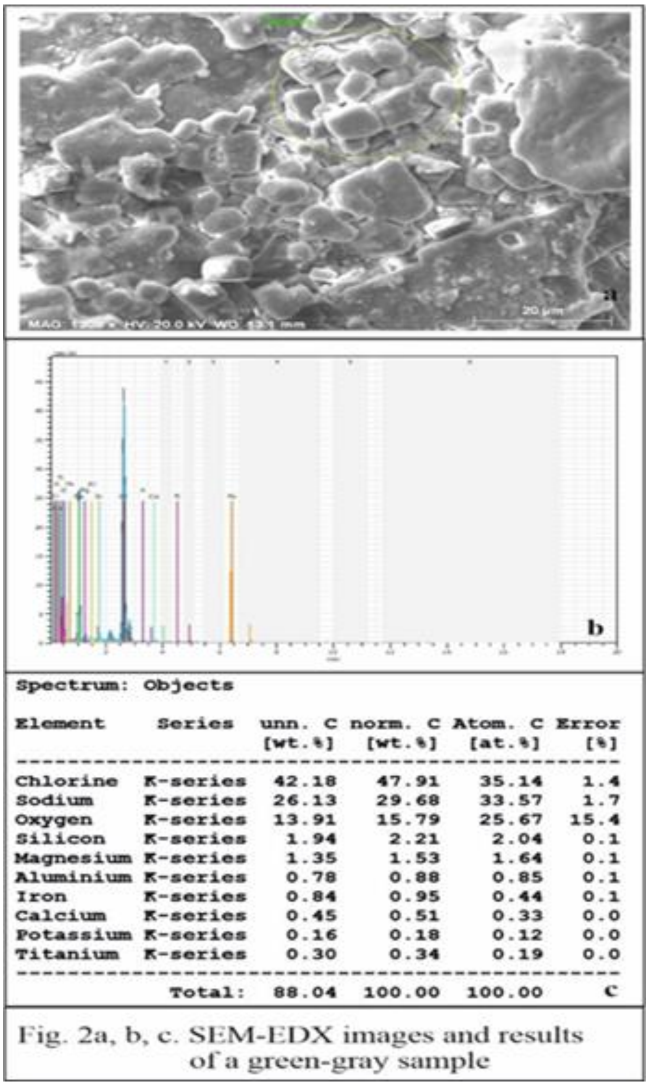
The number of heavy metals such as Pb, Cd, Hg, As, Fe, Cr, Al and Zn were determined elementally on 2 samples selected from the collected samples. Hg and Cd could not be detected. According to the Turkish food security communiqué, As, Hg, Pb, and Cd values are well below the limit values (Table 4).

Table 4. ICP - OES Results (Tois)

NICP-1			NICP-2			
H.M	Unit	Result	H.M	Unit	Result	TFC limit Value
Pb	mg/kg	0,018	Pb	mg/kg	0,016	2 mg/kg
Cd	mg/kg	-	Cd	mg/kg	-	0.5 mg/kg
Hg	mg/kg	-	Hg	mg/kg	-	0.1 mg/kg
As	mg/kg	0,067	As	mg/kg	0,058	0.5 mg/kg
Fe	mg/kg	40,4	Fe	mg/kg	38,9	
Cr	mg/kg	0,14	Cr	mg/kg	0,12	
Al	mg/kg	2.04	Al	mg/kg	2,8	
Zn	mg/kg	18,3	Zn	mg/kg	17,6	

3.3. Evaluation of SEM - EDX Results

On the greenish, gray-colored Halite (salt) samples taken from the study area, the presence of Cl, Na, O, Si, Mg, Al, Fe, Ca, K and Ti were determined by scanning electron microscopy (Figure 2a, b, c). Another sample from the study area, transparent, halide (salt) sample was found only Na and Cl (Figure 3). The passage of gray-green clays and salt crystals within the body of salt were examined and the type and percentage of the elements found in the cellar were determined (Fig. 2a, b, c). Na (sodium) and Cl (Chlorine) were detected by SEM-EDX analysis (Fig. 3a, b, c) of the clear halite (salt) sample without any pollution.



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Spectrum: Objects

Element    Series   unn. C norm. C Atom. C Error
                    [wt.%]  [wt.%]  [at.%]   [%]
------------------------------------------------
Chlorine  K-series  42.18   47.91   35.14    1.4
Sodium    K-series  26.13   29.68   33.57    1.7
Oxygen    K-series  13.91   15.79   25.67   15.4
Silicon   K-series   1.94    2.21    2.04    0.1
Magnesium K-series   1.35    1.53    1.64    0.1
Aluminium K-series   0.78    0.88    0.85    0.1
Iron      K-series   0.84    0.95    0.44    0.1
Calcium   K-series   0.45    0.51    0.33    0.0
Potassium K-series   0.16    0.18    0.12    0.0
Titanium  K-series   0.30    0.34    0.19    0.0
------------------------------------------------
           Total:   88.04  100.00  100.00      c
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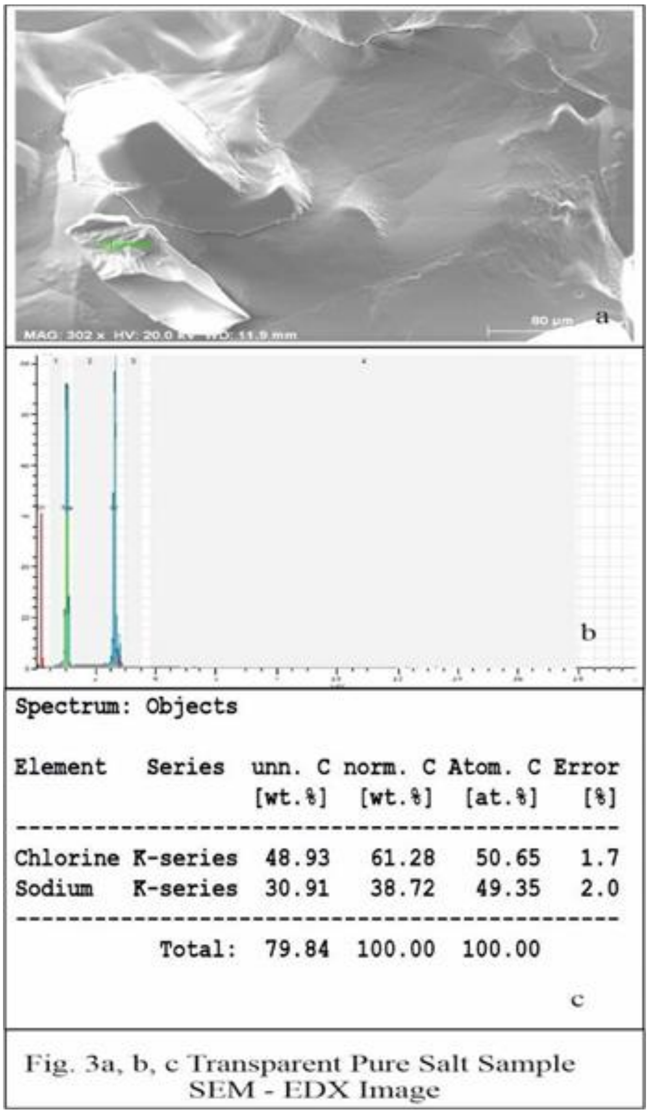
Fig. 2a, b, c. SEM-EDX images and results of a green-gray sample

Figure 2. SEM-EDX images and results of a green-gray sample

4. CONCLUSION

The studies in the study area, the previous studies and the analyzes on the sample taken were evaluated together. It was observed during the field studies that the Upper Eocene-Oligocene aged red-colored sandstones and claystone contain small-sized anhydrite and rock salt lenses, Oligocene aged rock salt deposits over this unit and these rock salts are under a gypsum-clay alternation. In the analysis made in greenish-gray colored salt samples, some foreign elements such as Al, Fe, Cr, Zn, Mn, Sr, and Ba have been determined and in some periods of sedimentation depending on the decomposition, transport, and alteration of the rock groups formed by the volcanism of these elements. moved into the crystal structure. Pure transparent Halite (Salt) bands were detected in the alternating rock salt deposits and no other elements other than Na and Cl were found in the SEM - EDX studies. According to the Turkish Food Codex Salt Communiqué, the heavy metals are below the limit value in the chemical analysis results of these samples. Heavy metals were evaluated in terms of their quantities in the body. The values (table 4) were compared according to the Turkish Food Codex Salt Communiqué. It was observed that the amount of heavy metal was below the limit value.



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Spectrum: Objects

Element  Series   unn. C norm. C Atom. C Error
                  [wt.%]  [wt.%]  [at.%]   [%]
----------------------------------------------
Chlorine K-series  48.93   61.28   50.65   1.7
Sodium   K-series  30.91   38.72   49.35   2.0
----------------------------------------------
           Total:  79.84  100.00  100.00

                                             c
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Fig. 3a, b, c Transparent Pure Salt Sample SEM - EDX Image

Figure 3. Transparent pure salt sample SEM-EDX image

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