Structural Characterization of Salts Using X-ray Fluorescence Technique: Experiments on Samples Collected from Kurdistan Region of Iraq

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***Abstract*—This study investigates the structure of 21 table salts that were collected from different local markets in the Kurdistan region of Iraq. The major trace elements and iodine concentrations in table salt are analyzed through the X-ray fluorescence (XRF) technique and the titration method, respectively. The study shows that using XRF spectral analysis, the collected table salt samples are rich in chlorine, sodium, and contain a lower percentage of bromine, strontium, tin, tellurium, and iodine. Moreover, these samples have a high percentage of sulfur and sirconium, where the molybdenum is >0.2%. Other elements such as zinc and copper are essential and found in low concentrations <0.0086% and 0.001%. Iodine is a trace element that is necessary nutrients for human life, and it is naturally present in some foods. Iodine deficiency is brought on by a lack of iodine consumption. Iodized salt is highly recommended as a source of iodine to prevent iodine deficiency disease. Iodine is added to table salt in two different ways, either through iodate or through iodine. The results show that only 25% of the salt samples have an adequate level of iodine, while the other samples have low or no iodine content. According to the World Health Organization, quality of salt depends on iodine concentration and other trace elements, which are necessary for human health.**

***Index Terms*—Salts, X-ray fluorescence technique, Iodine concentration, Chemical composition.**

I. Introduction

Natural additives such as table salt are vital for human survival. Table salt consists of 97.4% sodium chloride and a small amount of iodine, which is crucial for metabolic activities and the thyroid gland. To meet the daily iodine requirement, potassium iodate is typically added to table salt. Iodine deficiency diseases (IDDs) are known to exist in a number of nations worldwide, particularly in poor nations. The risk of IDD development affects about 38% of the global population (Wulandari and Rosyida, 2017). The thyroid gland stores of the iodine for a person in good health have 70–80%, or around 15–0 mg (Prodhan, et al., 2014). Thyroid-stimulating hormone (TSH) regulates the production of the thyroid hormones T3 (triidothyronine) and T4 (tetraiodothyronine or thyroxine), which are involved in all essential bodily processes. Iodine is a substrate required for this process (De Nayer, 2001, Kitwa, et al., 2012, Diosady, et al., 1997).

As the human body cannot generate iodine, it is important to consume iodine-rich foods frequently. In general, the salt is added into foods during processing, which is typically the main iodine supplier for people. Seafood, meat, milk, eggs, cereals, fruits, and vegetables are some dietary items that contain iodine (Mohammadi, Azizi and Hedayati, 2018). Table salt must contain within 30–80 ppm of iodine in agreement with standard national iodine (SNI) No. 3556:2010. The iodization technique is used to fortify table salt with either potassium iodide or potassium iodate (Wulandari and Rosyida, 2017). Every stage of a person's life is impacted by iodine deficiency, which can cause a number of serious illnesses (Verma and Raghuvanshi, 2001). In addition, depending on the source and technique of salt production, naturally secondary products are often composed of calcium, potassium, magnesium, sodium sulfates, carbonates, bromides, calcium, potassium, and magnesium chlorides, which may be found in edible salt in varying amounts (Alimentarius, 2001). Salts production with impurities causes absorb moisture and break, making it complicated to use, they must all be minimized throughout

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the purification process to an acceptable standard level (Kumar, et al., 2001, Nerín, Aznar and Carrizo, 2016).

In the US, salt was first infused with iodine in 1924 (Markel, 1987). Universal salt iodization (USI) was suggested as the primary source for reducing IDD by the World Health Organization and UNICEF in 1994. Almost 20% about the iodine within salt is removed during manufacture for home use, and an additional 20% is lost during cooking. Through packaging, storage, and heating, an additional 50% might be lost (Organization, 2007). About 120 nations across the world started salt iodization projects in 2006. In some nations, it has successfully eradicated iodine deficient illnesses by iodizing all salt (USI) (UNICEF, et al., 2008). Around 123 nations approved the iodization of salt between 1942 and 2020 and 124 nations required salt iodization laws in place by 2021(Meena, et al., 2018, Zimmermann and Andersson, 2021). Based on data gathered between 2013 and 2018, UNICEF estimates that 88% of the world's population consumed iodized salt in 2018. East Asia, South Asia, and the pacific had the highest home iodized salt coverage rates, at 89% and 92% respectively, as shown in Fig. 1. Although the coverage of iodized salt in central and western Africa was the lowest, more than three out of every four families had access to it in the area (Andersson, et al., 2007).

II. Methodology

Twenty-one table salts purchased from local stores of Kurdistan Region-Iraq were analyzed using X-ray fluorescence (XRF). The XRF method allowed for the analysis of the chemical composition or elements present in twenty-one samples in <3 h. The X-ray measuring time was only 200 s for the Al target and 100 s for other including RX9, Cu, and Mo secondary targets at room temperature at around 27°C. A detailed description of the XRF technique can be found elsewhere (Chaqmaqchee, Baker and Salih, 2017). Twenty-one salt samples have been collected from different locations in Kurdistan Regions-Iraq. All measurements of salts were carried out under vacuum, using a Rigaku Next-generation Energy dispersive Cartesian Geometry (NEX CG) with four targets. Chemical elements of Na, Cl, I, and other trace elements were calculated with almost the same salt concentration. The $K\alpha$-lines intensities were measured for most salt sample, in which the applied voltage increased in general with the required lines energies. In addition, titration method is one of the common methods used globally to estimation iodine in iodized salt. This is due to easy method and low cost. Titration is a process in which unknown solution's concentration can be determined using a solution with known concentration. Iodometric titration technique was used to determine the iodine content in 21 different samples of iodized salt. An unknown solution's concentration is determined by adding a known volume of the titrant (the known solution) to a known volume of the analyte (the unknown solution) until the reaction is completed. Indicator is used as a signaling to end of the reaction. The endpoint can be detected by observing color change from light brown to colorless as shown in Fig. 1. The endpoint of a titration is the point at which titrant addition should be prevented. The unknown concentration can be determined by knowing the amount of titrant added. A standard solution of 0.005 N sodium thiosulfate as a titrant in addition to its two drops of indicator (1% starch), which changed the solution's color from yellow to light brown then the color of the solution tuned to colorless after the reaction occurs completely.

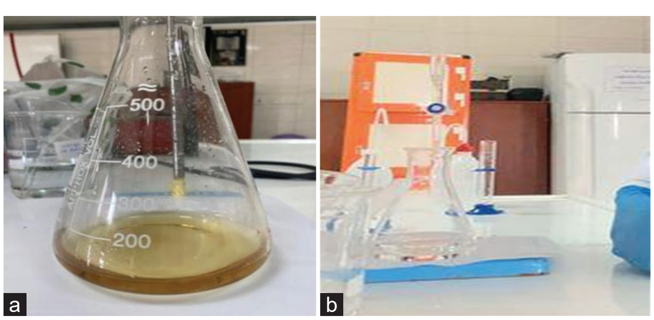

Fig. 1. (a) Before starting titration and (b) after ending titration.

A. Preparation of Standard Solution in Titration Method

Preparation of 0.005 M sodium thiosulfate ($Na_2S_2O_3$)

Dissolve 1.24 g of sodium thiosulfate in 1 litter hot distilled water.

Preparation of 2N sulfuric acid (H_2SO_4)

Add 5.56 ml concentrated sulfuric acid drop-wise into a 90 ml of chilled distilled water and make the final volume up to 100 ml (Sepahvand, et al., 2018).

Preparation of potassium iodide (KI)

Dissolve 10 g of potassium iodide in 100 ml of distilled water.

Preparation of saturated salt solution

Take 100 ml of distilled water in a conical flask and add sodium chloride until the salt is insoluble. Heat the solution till the sodium chloride crystals are formed on the sides of the vessel. After cooling down the saturated salt solution at room temperature, transfer the supernatant to a clean bottle (Sepahvand, et al., 2018).

Preparation of 1% Starch solution

Take 1 g of starch and prepare slurry in 50 ml of water. Add the slurry slowly to 50 ml of boiling water (Sepahvand, et al., 2018).

B. Procedure and Working Step

Dissolve 10 g of salt in 50 ml of distilled water, then added 1–2 ml of (2N) sulphuric acid and 5 ml of 10% KI to the solution with constant shaking the solution, it turns to a yellow color. The flask is closed with stopper and keeps it in the dark for about 10 min. Then, the sample is taken out of the dark place and titrated against a sodium thiosulfate solution until it turns a pale yellowish color. Subsequently, add a few drops of 1% starch solution. The solution will turn a deep purple color. Thiosulfate drops are added drop by drop from the burette until the solution becomes colorless, and note the final reading targets.

III. Results and Discussion

The salts were subjected to XRF to analyze the chemical composition or elements present in the sample as summarized in Tables IA and B, for which the salt samples were measured in advanced research laboratory of physics at Koya University of Kurdistan Region-Iraq. Some major and trace elements were identified in 21 salt samples coded between H1 and H21.

The results from the analysis of XRF were based on characteristic Kα lines from the XRF spectral analysis of the salts, the salts contained less percentage of bromine, strontium, tin, tellurium, and Iodine and high percentage of zirconium and molybdenum. Other elements as zinc and copper are essential element and found in low concentrations <0.0086% and 0.001%. However, it can be potential hazard when presented in an over concentration, which can affect both animal's life and human health (Papagiannis, et al., 2004).

When the samples are irradiated with X-rays, the intensity as a function of energy can be calculated over the energy range 1-20 keV at the same computing conditions. Fig. 2 shows two essential peaks in the ranges of ~1.85 and 7.12 keV corresponding to Na- kα and Cl- kα lines for all 21 salt elements. Note that the peak intensity of Cl-k_{α} has a higher intensity than Na-kα intensity due to the salts being comparatively rich in Cl and Na contents that can be interpreted as good sources of Cl and Na as shown in Fig. 2a. In addition, the peak intensities of 11.91, 14.17, 15.76, 17.69, 25.25, 27.41, and 28.57 keV that corresponding to Br-k_{α}, Sr-k_{α}, Zr-k_{α} Mo-k_{α}, Sn-k_{α}, Te-k_{α}, and I-k_{β} lines are indicated in Fig. 2b. It was noticed that Zr element has high intensity than the rest elements.

Fig. 3 shows that the peak intensity of element Cl is higher than that of elements of Na and I. The mass concentrations of salt samples are increasing from around 45–69 mg/cm^2. Where the elements coded between H1 and H21 are indicated in the chart in Fig. 4.

As shown in Table II, the samples labeled from H1 to H21, measuring iodine concentration for all samples, during the titration observed that big different in iodine content in 21 samples. Titrant ($Na_2S_2O_3$) volume increases indicate a higher concentration of iodine present in the solution or in the nature of the salt.

TABLE IA
Chemical Compositions for Salt Samples Coded H1 to H10 Under the Same Measuring Conditions

No.	Elements	Mass concentration % (mg/cm²)									
		H1	H2	H3	H4	H5	H6	H7	H8	H9	H10
1	Cl	65.9	51.4	68.9	45.6	64.8	67.3	67.5	67.3	67.1	68.0
2	Na	31.7	40.5	29.6	42.0	31.7	30.6	30.2	31.5	30.4	30.6
3	Si	0.766	0.561	0.498	0.484	1.01	0.623	0.551	0.501	0.587	0.542
4	S	0.722	0.769	0.432	1.89	1.00	0.582	0.688	0.232	1.18	0.321
5	Ca	0.578	4.37	0.251	8.28	0.395	0.471	0.595	0.0483	0.154	0.164
6	Zr	0.241	1.22	0.265	1.24	0.301	0.270	0.340	0.299	0.218	0.315
7	K	0.0321	0.656	0.0206	0.270	0.519	0.0332	0.0523	0.0359	0.241	0.0250
8	Fe	0.0167	0.241	0.0063	0.0301	0.0378	0.0074	0.0069	0.0032	0.0038	0.0034
9	Sn	0.0061	0.0312	0.0064	0.0370	0.0081	0.0065	0.0091	0.0078	0.0058	0.0086
10	W	0.0031	--	0.0013	--	0.0028	0.0024	0.0036	0.0039	0.0019	0.0014
11	Ti	0.0030	--	--	--	0.0118	--	--	--	--	--
12	Sr	0.0025	0.0582	0.0027	0.0394	0.0039	0.0026	0.0033	0.0007	0.0018	0.0016
13	Co	0.0019	0.0163	0.0026	--	0.0013	0.0009	0.0021	--	--	--
14	Re	0.0011	0.0040	0.0011	0.0032	0.0016	--	--	--	--	--
15	Au	0.0010	0.0048	0.0009	--	0.0010	0.0007	0.0010	0.0009	0.0006	0.0007
16	Cu	0.0005	0.0057	0.0011	0.0017	0.0007	0.0009	0.0011	0.0010	0.0005	0.0009
17	Br	0.0004	0.0534	0.0050	0.0556	0.0049	0.0024	0.0031	0.0075	0.0038	0.0033
18	SC	--	0.0879	--	--	--	--	--	--	--	--
19	Cr	--	0.0154	--	--	0.0021	--	--	--	--	--
20	Zn	--	0.0086	--	0.0014	--	--	--	--	--	--
21	Ta	--	0.0060	--	0.0046	0.0013	--	--	--	--	0.0011
22	Ni	--	0.0034	--	--	--	--	--	--	--	--
23	Rb	--	0.0033	0.0005	0.0054	0.0009	0.0004	0.0004	0.0010	0.0007	0.0008
24	Fr	--	--	--	0.0451	0.0032	0.0012	0.0019	0.0076	0.0042	0.0044
25	Mn	--	--	--	0.0138	--	--	--	--	--	--
26	V	--	--	--	--	0.0011	--	--	--	--	--
27	Ag	--	--	--	--	0.0006	--	--	--	--	--
28	I	--	--	--	--	0.0090	--	0.0033	0.0029	0.0060	
29	P	--	--	--	--	--	--	0.0960	--	--	--
30	Os	--	--	--	--	--	--	0.0010	--	--	--
31	Hg	--	--	--	--	--	--	0.0009	--	--	--
32	Po	--	--	--	--	--	--	0.0005	--	--	--
33	Te	--	--	--	--	--	--	--	0.0011	0.0011	--
34	Ge	--	--	--	--	--	--	--	--	--	0.0004

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TABLE IB
Chemical Compositions for Salt Samples Coded H11 to H21 Under the Same Measuring Conditions

No.	Elements	Mass concentration % (mg/cm²)										
		H11	H12	H13	H14	H15	H16	H17	H18	H19	H20	H21
1	Cl	68.8	65.9	58.3	67.6	66.2	66.5	63.2	66.2	65.1	64.5	65.4
2	Na	29.6	30.3	37.9	31.5	33.0	31.8	34.7	31.2	34.3	32.0	32.5
3	Si	0.569	0.456	0.618	--	0.504	0.491	0.549	0.476	0.481	0.886	0.529
4	S	0.346	1.60	1.50	0.452	--	0.866	0.680	1.19	--	1.55	0.689
5	Ca	0.188	1.11	0.280	--	--	--	0.507	0.219	0.0123	0.295	0.476
6	Zr	0.340	0.305	0.241	0.322	0.247	0.272	0.287	0.327	0.101	0.174	0.264
7	K	--	0.0432	0.278	--	--	--	0.0416	0.249	0.0105	0.325	0.0726
8	Fe	0.0047	0.0031	0.0046	0.0046	0.0017	0.0024	0.0097	0.0044	--	0.0187	0.0085
9	Sn	0.0093	0.0069	0.0068	0.0087	0.0064	0.0068	0.0073	0.0086	0.0024	0.0043	0.0062
10	W	0.0031	0.0026	--	0.0030	--	0.0022	--	0.0096	0.0027	0.0064	--
11	Ti	--	--	--	--	--	--	0.0033	--	--	0.0043	--
12	Sr	0.0030	0.0097	0.0052	0.0037	0.0009	0.0030	0.0029	0.0045	0.0003	0.0024	0.0024
13	Co	--	0.0009	--	--	--	0.0009	--	0.0011	--	--	--
14	Re	0.0019	--	--	--	--	--	--		--	--	--
15	Au	0.0012	0.0010		0.0009	0.0008	0.0006	--	0.0006	--	--	--
16	Cu	0.0007	0.0006	0.0006	0.0006	0.0010	--	--	0.0007	--	0.0004	0.0005
17	Br	0.0047	0.0214	0.0153	0.0048	0.0026	0.0144	0.0006	0.0070	0.0022	0.0026	0.0034
18	SC	--	0.0095	--	--	--	--	--	--	--	--	--
19	Cr	--	--	0.0017	--	--	--	--	--	--	--	--
20	Zn	--	--	--	--	--	--	--	--	--	--	--
21	Ta	0.0013	0.0017	0.0009	0.0012	--	--	--	0.0015	--	--	--
22	Ni	0.0009	--	--	--	--	--	--	--	--	--	--
23	Rb	0.0005	0.0017	0.0015	0.0008	0.0006	0.0014	--	0.0011	0.0004	0.0005	0.0006
24	Fr	--	0.0138	0.0115	0.0052	0.0034	0.0105	--	0.0054	0.0023	0.0016	0.0031
25	Mn	--	--	--	--	--	--	--	--	--	--	--
26	V	--	--	--	--	--	--	--	--	--	0.0011	0.0009
27	Ag	--	--	--	--	--	--	--	--	--	--	--
28	I	0.0062				0.0038		0.0062	0.0032	--	--	--
29	P	0.0940	0.113	--	--	--	--	--		--	--	--
30	Os	--	--	--	--	--	--	--	0.0008	--	--	--
31	Hg	--	--	--	--	0.0007			0.0011	--	--	--
32	Po	--	--	--	--	--	--	--	--	--	--	--
33	Te	0.0012		0.0008	0.0009	--	--	--	--	--	--	--
34	Ge	--	--	--	--	--	--	--	--	--	--	--
35	Mg	--	0.539	--	--	--	--	--	--	--	--	--
36	Al	--	--	0.228		--	--	--	--	--	--	--
38	At	--	--	--	--	--	--	0.0004	0.0005	0.0003	--	--

Titration method is used for determining the trace element such as iodine with a highly accuracy and precise as indicated in Table II. Result show that from 21 samples of iodized salt only seven sample of salts were present iodine and the rest of it not present iodine in salt sample, as well as three samples from these seven samples are not contain the recommended amount of iodine which means the concentration of iodine is less than that recommended to be. However, sample six (H6) showed the highest concentration of iodine, due to reactivity of iodine the rate of iodine decrease with the increasing temperature; moreover, iodine loss from iodine-containing materials is probably influenced by exposure to air and light not only that also inadequate storage might result in loss iodine and humidity affect the iodine, recommendation iodine intake based on the literature is (30–80) ppm, six samples are exiting iodine in the recommendation range, exclusively for the reason of diminish iodine level in salts and foods by increasing heat exposure during cooking 30% of the 20 different household salt are recommended to take advantage. Figs. 5 and 6 show household salt which is iodine is specified with X-ray fluorescence method, which realizes the same result for the titration method.

Most salt samples contain lower amount of iodine compared to standard salt sample, in one of the salt's label indicates that it contains 50–80 mg/kg of potassium iodide.

However, the sample of number six (H6) which is named (Al-Nema salt) contains the highest quantity of iodine (74.02 ppm). The lowest iodine content was 2.11 ppm with in H12 and H13 (Taban table salt and Henan salt), respectively, contain the least quantity of iodine. Iodine content was below 15 ppm in 70% of samples, additionally 30% was significantly contained iodine higher than 15 ppm. The sample number six with the highest concentration of iodine is similar with the result by the XRF analyzed. The main sources of iodized salt imported into the Kurdistan from Iran and Turkey. The salt may refer to coarse rock salt and H1, which is the concentration of iodine, equal to (8.46 ppm). To reduce the range of error, we repeated the titration 2 times.

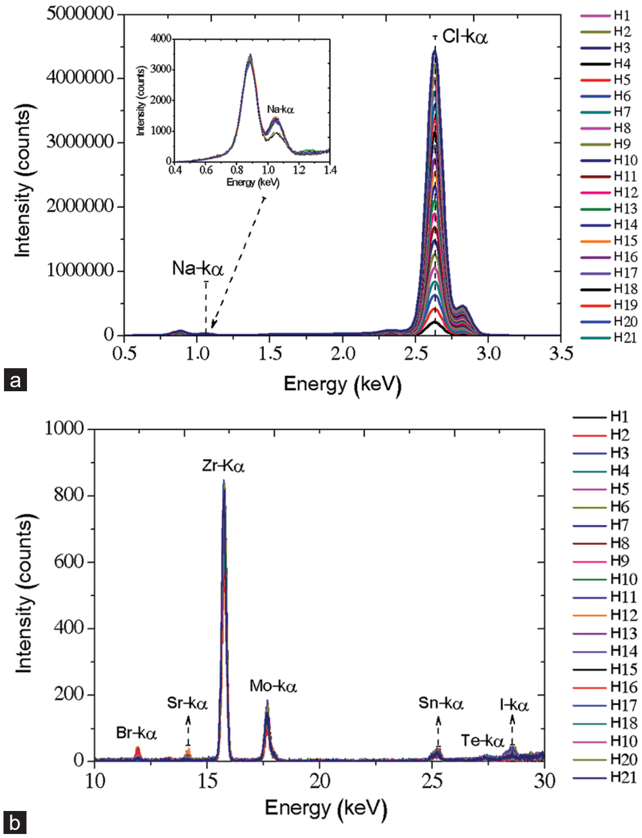

Fig. 2: Energies of X-ray spectra for various salts samples coded from H1 to H12 at (a) high intensity with zoomed Na-kα peak and (b) low intensity using X-ray fluorescence technique.

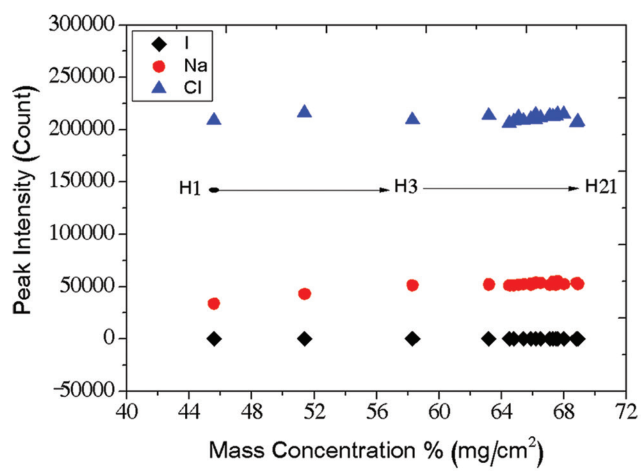

Fig. 3: Dependence of the X-ray fluorescence intensity on the salts concentration coded from H1 to H21.

The result in ppm value of iodine content is displayed in the Fig. 7. However, titration method used commonly but result compared to analytical technique such as XRF not high precise result, may due to technique error and personal error during titration and end point detection.

To investigate the structure of salt and iodine concentration, we used two different methods, such as the titration method and the XRF technique. The main goal of this study is to compare the concentration of iodine in 21 different brands of

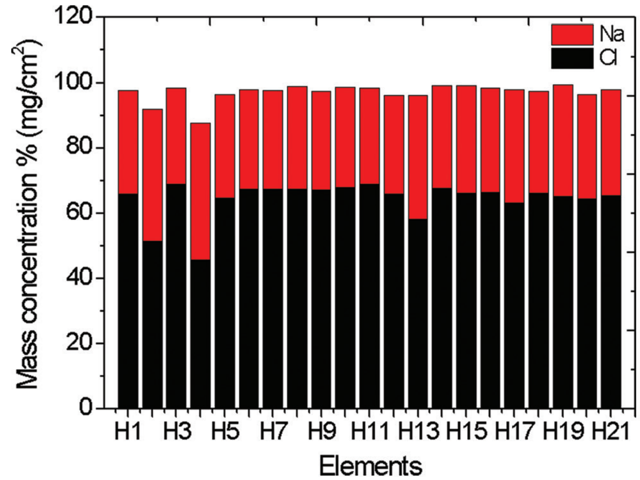

Fig. 4: Mass concentration of Cl and Na elements in the salt samples coded between H1 and H21.

TABLE II
Calculation of Iodine Concentration in ppm

Samples number	Volume of 1st titration in ml	Volume of 2nd titration in ml	Average in ml	Iodine conc. in ppm
H1	1	0.6	0.8	8.46
H2	1	1.2	1.1	11.63
H3	0.6	0.5	0.55	5.81
H4	0.5	0.5	0.5	5.2
H5	0.7	0.8	0.75	7.9
H6	7	7	7	74.02
H7	1.5	1.3	1.4	14.8
H8	3	2.8	2.9	30.66
H9	5	4	5.5	58.1
H10	0.2	0.3	0.25	2.64
H11	1.4	1	1.2	12.69
H12	0.3	0.1	0.2	2.11
H13	0.2	0.2	0.2	2.11
H14	1	0.6	0.8	8.4
H15	4	3.8	3.9	41.22
H16	0.5	0.3	0.4	4.22
H17	1.8	1.2	1.5	15.85
H18	2.7	2.5	2.6	27.48
H19	1.5	1.3	1.4	14.8
H20	0.7	0.7	0.7	7.40
H21	0.4	0.5	0.3	4.12

table salt or iodized salt. The sample salt has been iodized, which mean that iodine was added to salt sample. Iodine cannot be produced by human body, despite the fact that it is necessary for a functioning thyroid and other bodily processes. Iodine is used by the thyroid gland to produce thyroid hormones that promote a healthy metabolism, manage growth, and repair damaged cells. There is various sources of iodine but the most common one is iodized salt which is used daily and cheap.

Adequate amount of iodine content in iodized salt should concentration of iodine between (30 and 80) ppm. Unfortunately, the iodized salt available on the local market does not contain adequate iodine, there are many factors that have negative impact on iodine concentration such as, light when sunlight strikes the iodized salt loses between 47.8%

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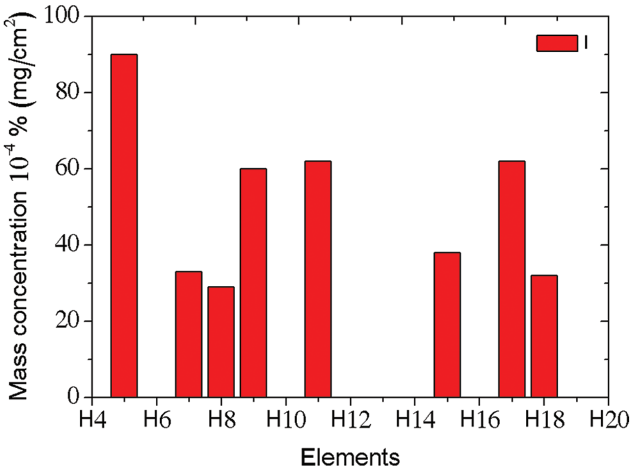

Fig. 5: Mass concentration of I element in the samples coded between H1 and H21.

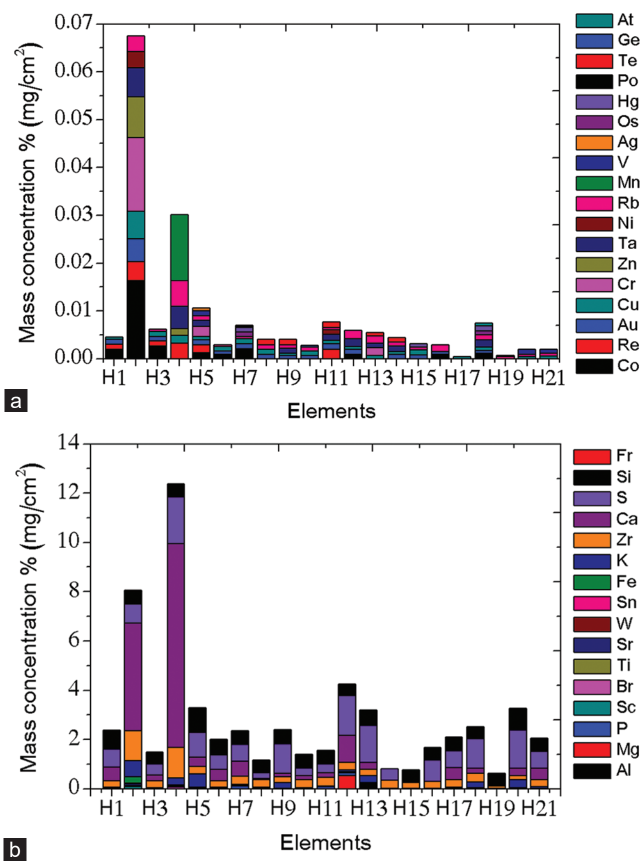

Fig. 6: Mass concentration of trace elements in the samples coded between H1 and H21 from elements between (a) Co and At, and (b) Al and Fr.

and 49.1% of its iodine content, also another factor that might affect iodine content and decrease its concentration in iodized salt is heating and packing. Compared to XRF result, the titration approach is less accurate may due to error at endpoint detection, leading to significant errors in titration process.

XRF result is showed that 70% of samples does not contain enough concentration of iodine, where both techniques proved

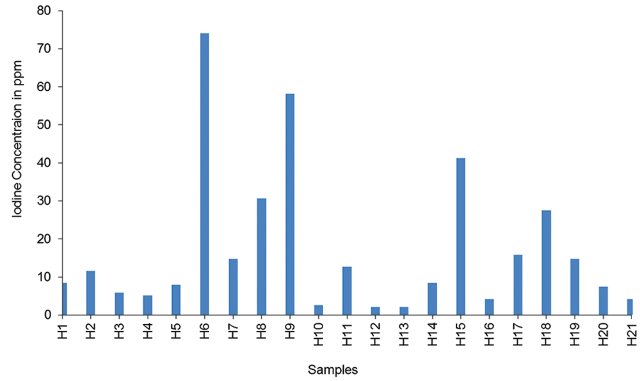

Fig. 7: The concentration of iodine in salts based on the titration method.

that the S.6 (Al-Nema salt) contains the highest concentration of iodine that is equal to 74.02 ppm by titration and 90 ppm by XRF. In addition, the titration method revealed the lowest iodine concentration in H11 and H12.

Based on the literature, the lack of iodine content in iodized salt may be due to various factors, such as the nature of the salt and exposure to direct sunlight. The amount of iodine concentration in iodized salt might also be affected by how it is packaged and stored. Sometimes, iodine is lost from salt samples during cooking. However, each different salt brand had various results. The result from the XRF method detects iodine only in 30% of samples; more than half the samples do not contain iodine.

IV. Conclusion

The purpose of this study is to estimate the major and trace elements in 21 different brands of table salt or iodized salt. To valuate iodine concentration, we used two different methods (the titration method and the XRF technique). The study showed that the table salts using XRF spectral analysis were rich in chlorine and sodium and contained a lower percentage of bromine, strontium, tin, tellurium, iodine and a high percentage of sulfur, sirconium, and molybdenum <0.2%. Other elements such as zinc and copper are essential and found in low concentrations, >0.0086% and 0.001%, respectively. An adequate amount of iodine content in iodized salt should have a concentration of iodine between (30 and 80) ppm. Unfortunately, the iodized salt available on the local market does not contain adequate iodine. Many factors hurt iodine concentration, such as light when sunlight strikes the iodized salt, which loses between 47.8% and 49.1% of its iodine content. Furthermore, another factor that might affect iodine content and decrease its concentration in iodized salt is heating and packing. Compared to the XRF result, the titration approach is inaccurate may be due to error at end-point detection, leading to significant errors in the titration process. The result of the XRF showed that 70% of samples did not contain enough concentrations of iodine, and both techniques proved that sample H6 (Al-Nema salt) contains the highest concentration of iodine which is equal to 74.02 ppm by titration and 90 ppm by XRF. In addition, H11 and H12 showed the lowest concentrations of iodine with the titration method.

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Declaration

We declare that this study is not to defame any product or harm the reputation of the product.

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