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ARTICLE

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Abstract

This study was demonstrated to determine the concentration of iodine obtained from different regions of Al-Muthanna Governorate in different labeled salt. 136 samples including 10 commercial brands were collected. The iodine contents of the samples were measured by both the iodometric titration process and spot-testing kit. The results displayed that 36.7% of all samples are inadequate iodine content. The quantitative analysis showed that 31.6% and 46.3% are iodine inadequate salts with content less than 15 ppm of salt samples commercialized in urban and rural regions, respectively. Also, there is a significant difference in iodine concentrations between packaged and bulk salt, besides, all samples of bulk salt were found in rural areas. a comparative study was made between results obtained from iodometric titration and spot-testing kit method, and both methods showed a nearly similar proportion of salt samples with iodine ≥15 ppm (61.8% by kit and 63.3% by titration). These results indicate that the titration technique is essential for quantitative purposes. However, the spot-kit method is dependable for semi-quantitative evaluation of iodine content and it can be utilized for monitoring the quality of iodized salt obtainable in the community.

Keywords: Iodine deficiency disorders, Iodometric titration, Iodine determination, Spot-testing kits

1. Introduction

Iodine is an important element for the normal growth and development of humans. A healthy human body contains (15−20)mg of iodine, of which (70−80)% is stored in the thyroid gland [1]. The thyroid gland utilizes iodine to synthesize the thyroxine and triiodothyronine hormones, which are necessary for maintaining the body's metabolic rate by controlling energy production and oxygen consumption in cells, regulating temperature, improving digestion, maintaining a healthy weight and for neurodevelopment [2]. Since iodine is not produced in the body, daily intake from external sources is required, mainly in the first weeks of life, as the developing fetus does not develop a functioning thyroid gland until about (18−20)weeks of gestation, during which time it is completely dependent on the mother to produce the thyroid hormones needed for growth [3]. Thus, maternal iodine intake is essential for both mother and child, because even mild iodine deficiency can lead to decreased cognitive development [4]. Increased iodine requirements during pregnancy have led the WHO to recommend pregnant increasing iodine intake from 150 mcg to 250 mcg [5]. Iodine therapy is not always available, additional iodine supplementation may be required, and intake should be constantly monitored [6,7]. Iodine sources other than iodized salt can be consumed and may play an important role in the iodine status of individuals [8]. They may include sources such as saltwater fish, seaweed, shellfish, soy sauce, yogurt, cereals, some medications, iodine in processed foods (such as bread or margarine) [9], drinking water [10], cow's milk or infant formula [11] and vegetables [8]. Some

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of these factors may add a significant amount to iodine intake, such as iodine in seafood and processed foods [12].

The titration method is undoubtedly the most common method utilized to quantitatively monitor the adequacy of salt iodization at the level of production, importation and consumption, due to its accuracy, relative ease of operation and low cost. This method is recognized globally as the reference for measuring iodine in iodized salt and should always be considered as a back-up method for any alternative method [5], but it is impractical for many small immunization facilities nor for field use in salt surveys [13]. Rapid-testing kits consist of a starch solution, which causes a blue-purple discoloration when dried over 1 teaspoon of iodized salt. The intensity of the staining is sometimes taken as an indication of the iodine concentration, but the accuracy of these kits at any iodine concentration above the zero point is questionable. Due to their simplicity and quick response, these kits are often utilized in home surveys [14].

The aim of this study is to estimate iodine in a number of dietary salt samples available in the markets of Al-Muthanna Governorate utilizing the iodometric titration and spot-testing kit methods. The other purpose of the study was to assess the performance of the kit in monitoring the iodine content of salt in situations of multiple observers. The goal of monitoring universal salt iodization) USI (is to estimate the availability of iodized salt ($\geq$15 ppm of iodine). The results were divided into two groups and analyzed according to the presence (>0 ppm) or absence (0 ppm) of iodine in the salt. In order to compare the performance of the kit with iodometric titration.

2. Materials and methods

2.1. Samples collection

Samples of table salt (136) were collected from randomly selected markets of Al-Muthanna Governorate, southern Iraq. The samples stored at room temperature away from light and moisture. All the samples were analyzed by the iodometric titration procedure [15] and the spot-testing kits (MBI Kits) [16] in the chemistry lab., college of basic education.

2.2. Reagents and solutions

2.2.1. Preparation of a 100 mL solution of 0.1N $K_2Cr_2O_7$

Approximately 0.49g of $K_2Cr_2O_7$ (analytical grade, Fluka) was accurately measured and placed in a 100 mL volumetric flask. Then a small amount of deionized water was added to the flask and shaken well until it dissolves completely. The flask was then filled with deionized water, completed up to the 100 mL mark.

2.2.2. Preparation of 500 mL of 0.1N $Na_2S_2O_3$

At an electronic balance, 12.5 g of $Na_2S_2O_3$ (analytical grade, BDH) was accurately measured and put in a 500 mL volumetric flask. Subsequently, a small amount of deionized water was applied in the flask and then shaken until the solution dissolved. The flask was then filled to the 500 mL mark with deionized water.

2.2.3. Preparation of 1% starch indicator solution

Weighed 1 g of starch (analytical grade, BDH) and placed in a conical flask. The starch was then dissolved into 100 mL of deionized water by heating and stirring at 79 C° for 5 min. Then the solution is cooled to room temperature.

2.2.4. Preparation of 10% potassium iodide

Ten gram of potassium iodide (analytical grade, BDH) was weighed and dissolved in 100 mL of demineralized water in order to prepare 10% Potassium Iodide solution.

2.2.5. Preparation of sulfuric acid (2N)

In a 50 mL volumetric flask, 2.8 mL of sulfuric acid (conc.) (analytical grade, HIMEDIA) was measured and gradually applied to the deionized water. The solution was then filled up to the 50 mL level with water.

2.3. Measurement of iodine concentration in dietary salt

2.3.1. Iodometric titration procedure

The iodine content of salt samples was calculated by standard iodometric titration utilizing the method described by Nath et al. [17]. At first, 10 g of salt was dissolved in 50 mL of deionized water, then 5 mL of 10% (m/v) KI solution and 1 mL of 2 N sulfuric acid were added. The mixture was well shaken and turned yellow/brown color, indicating iodine production. The solution was titrated against diluted $Na_2S_2O_3$ until the color yellow/brown became very pale. In the meantime, three drops of the prepared starch indicator solution were added, resulting in the formation of a dark blue complex. The titration continued until the colorless end point was obtained. The process was repeated two more times and an average value of the volume was determined. The results are expressed as milligram of iodine per kilogram of salt (mg/kg) [15].

2.3.2. Spot-testing kits

To determine iodine content in salt the spot-testing kit, produced by MBI chemicals, is a starch-based test. According to the manufacturer's instructions, the test can be utilized qualitatively to measure iodine in salt at 0, 15, and >30 ppm, (mg/kg of salt) [16,18]. Depending on the level of color obtained, it provides a qualitative estimation of the iodine content. The test indicates the amount of iodine present in the salt. If the test showed "no iodine" on the first testing, the check solution was added again. This was necessary to acidify the salt sample and neutralize the alkali that was present in the salt. It was considered a negative test result if the second attempt at the test revealed no iodine [16].

2.4. Data analysis

The data were processed and analyzed utilizing SPSS software, version 22. Tests for proportion and 95% confidence intervals were estimated as appropriate. To validate the spot-testing kit, sensitivity and specificity were calculated. The agreement was assessed utilizing the k statistic.

3. Result

Iodometric titration is the traditional method for easy and quick estimation of iodine content in dietary salt samples [19], Iodine is titrated with sodium thiosulfate, which involves the reaction of iodine (from salt) with potassium iodide in an acidic medium, where the iodide is oxidized to free iodine [20]. Free iodine reacts with an excess of potassium iodide to form a complex (I^{-5}), then the latter reacts with sodium thiosulfate to form a light green solution of tetraethionate ($S_4O_6^{-2}$), as in the following equations:

$$5I^- + 6H^+ \longrightarrow 3I_2 + 3H_2O + IO_3^- \qquad 1$$

$$I_2 + I_3^- \longrightarrow I_5^- \qquad 2$$

$$I_5^- + 2S_2O_3^{-2} \longrightarrow S_4O_6^{-2} + 3I^- \qquad 3$$

Total reaction equation:

$$IO_3^- + 5I^- + 6S_2O_3^{-2} \longrightarrow 3S_4O_6^{-2} + 6I^{-+} 3H_2O \qquad 4$$

The number of iodine (ppm) in the iodized salt samples was calculated using the following equation from the determined average volume of $Na_2S_2O_3$:

$$\text{Iodine (ppm)} = R * 100 * 1000 * 0.127 * N/6$$

where, R = Average volume of $Na_2S_2O_3$

100 is to convert the reading for 1000g of salt

1000 is to convert gram of iodine to milligram of iodine

0.127 is the weight of iodine equivalent to 1 mL of normal Thiosulphate solution

N is normality of thiosulphate solution (which is 0.005N).

6 is to arrive at the value that corresponds to 1 atom of iodine liberated.

A total of 136 salt samples were collected from three major districts of the Al-Muthanna governorate, where 94% (n = 128) were packaged salt and 6% (n = 8) were bulk salt. The number of samples collected from urban areas was 95 (69.8%) and rural areas was 41 (30.2%).

The results of quantitative analysis with the iodometric titration showed that 31.6% (31/95) of salt samples commercialized in urban areas are iodine inadequate salts with content less than 15 ppm, and 67.4% (64/95) of the latter samples are iodine adequate with content more than 15 ppm of iodine (Table 1). The results showed 46.3% (19/41) of salt samples in rural areas are iodine inadequate salts, and 53.7% (22/41) are iodine adequate by the iodometric titration method (Table 1).

All bulk salt samples (n = 8) are from rural areas, all of these samples had no iodine content utilizing spot-kit method, as well as, the iodometric titration method showed that 100% of bulk salts samples are iodine inadequate salts with content less than 15 ppm. As for the packaged samples the results by iodometric titration method showed that 32.8% (42/128) are salts with less than 15 ppm of iodine concentration, and 67.2% (86/128) of packaged salt samples are iodine adequate (Table 2).

Amongst a total of 136 salt samples collected from three major districts in Al-Muthanna Governorate, 72 (52.9%) were from Al-Samawah city the center of the Governorate. The results showed that 61.1% (44/72) are adequate Iodine samples (≥15 ppm) by both

Table 1. Sample sizes and iodine concentration in salt collected from urban and rural areas.

Origin of salt sample	Sample size	Iodine concentration by iodometric titration (ppm)	
		0.1 to < 15	≥15
Urban	95 (69.8%)	31 (32.6%)	64 (67.4%)
Rural	41 (30.2%)	19 (46.3%)	22 (53.7%)
Total	136	50 (36.7%)	86 (63.3)

Table 2. Sample sizes and iodine concentration in packaged and bulk salt.

Type of salt sample	Sample size	Iodine concentration by iodometric titration (ppm)	
		0.1 to < 15	≥15
Packaged salt	128 (94.1%)	42 (32.8%)	86 (67.2%)
Bulk salt	8 (5.9%)	8 (100%)	0 (0%)
Total	136	50 (36.7%)	86 (63.3)

titration and spot-kit methods. 36 (26.5%) salt samples collected from the second biggest district of Al-Rumaythah northern of Al-Samawah city. The results showed that 63.9% (23/36) are ≥15 ppm of iodine content samples by iodometric titration and spot-kit method. The third major district Al- Khidhir in southern of Al-Muthanna, a total of 28 (20.6%) salt samples were collected, 42.9% (12/28) are iodine adequate samples also by both two methods (Table 3).

Table 3. Iodine content of salt by both iodometric and spot-kit methods at different districts.

District	Salt iodine content									
	a*		b*		c*		d*		Total	
	N	(%)	N	(%)	N	(%)	N	(%)	N	(%)
Al-Samawah	44	(61.1)	3	(4.2)	4	(5.5)	21	(29.2)	72	(52.9)
Al-Rumaythah	23	(63.9)	1	(2.8)	3	(8.3)	9	(25.0)	36	(26.5)
Al-Khidhir	12	(42.9)	1	(3.6)	0	(0.0)	15	(53.5)	28	(20.6)
Total	79	(58.1)	5	(3.7)	7	(5.1)	45	(33.1)	136	

a* ≥ 15 ppm of iodine content by iodometric titration and spot-kit method.
b* < 15 ppm of iodine content by iodometric titration but ≥15 ppm by spot-kit method.
c** ≥ 15 ppm of iodine content by iodometric titration but <15 ppm by spot-kit method.
d* < 15 ppm of iodine content by iodometric titration method.

Table 4. Comparison of spot-kit and titration method for the determination of iodine content in salt.

Spot-testing kit (ppm of iodine)	Iodine concentration by iodometric titration (ppm)			Total
	0	0.1 to < 15	≥15	
0	4	28	3	35 (25.7%)
0.1 to < 15	1	12	4	17 (12.5%)
≥15	0	5	79	84 (61.8%)
Total	5 (3.6%)	45 (33.1%)	86 (63.3%)	136

In addition, a comparative study was made between results obtained from iodometric titration and spot-testing kit method, both methods showed a nearly similar proportion of salt samples with iodine ≥15 ppm (61.8% by kit and 63.3% by titration) (Table 4). However, most of the samples found by kit to have 0 ppm did have some iodine in them using the standard titration method. The sensitivity (ability to correctly identify salts with adequate iodine content) of the kit was 91.8% (95% confidence interval (CI) = 90.4–93.2), high sensitivity and specificity (ability to correctly identify salts with inadequate iodine content) was 90.0% (95% CI = 88.0–92.1). The positive predictive value (PPV) was 94.0% (95% CI = 92.3–95.7) while the negative predictive value (NPV) was 86.5% (95% CI = 81.9–91.1) (Table 5). As for the iodine presence, the sensitivity of the kit was 76.3% (95% CI = 74.6–77.91), and specificity was 80.0% (95% CI = 62.0–95.0). The (PPV) was 99.0% (95% CI = 97.0–100), while the (NPV) 11.4% (95% CI = 4.2–18.5) (Table 5).

4. Discussion

This study focused on two aspects, the first is to measure the content of iodine in table salt samples available in the three major districts in Al-Muthanna Governorate southern Iraq, using two methods, the standard iodometric titration and spot-testing kit methods. The results of the two methods showed that more than 60% of the tested samples are adequate iodine (≥15 ppm) salts. This content of iodine can reduce the incidence of iodine deficiency disorders. The difference between the iodine content of salt samples obtained from urban and rural areas is small, but bulk salt samples which all collected from rural areas showed iodine deficiency with less than 15 ppm of iodine content. The difference in the iodine content between the salts used in the districts was clearly noticeable, as the proportion of salts with an adequate iodine content in Al-Samawah and Al-Rumaythah was higher than that in the district of Al-Khidhir, which had the lowest proportion of salts with adequate iodine content among them.

Table 5. Spot-kit validation using a qualitative method.

Interpretation of test	Indicators (%)			
	Sensitivity	Specificity	PPV[a]	NPV[b]
Iodine absence (0 ppm) Iodine presence (>0 ppm)	76.3 (74.6–77.91)	80.0 (62.0–95.0)	99.0 (97.0–100)	11.4 (4.2–18.5)
Iodine deficiency (<15 ppm) Adequate iodine (≥15 ppm)	91.8 (90.4–93.2)	90.0 (88.0–92.1)	94.0 (92.3–95.7)	86.5 (81.9–91.1)

[a] Positive predictive value.
[b] Negative predictive value.

This study also analyses the performance of spot-testing kits in evaluating the status of salt iodization, which is the second aspect of this study. Standard iodine titration is the recommended method for assessing the iodine content of a table salt. However, it requires setting up a laboratory and a trained worker, on the other hand, spot-kit is a simple and a cheap qualitative and semi way to determine iodine content of a table salt, and can be used by anyone. This study amongst others revealed that for quantification, spot-kit method had high sensitivity but low specificity, which means intolerance to small amounts of iodine. These results indicate that spot-testing kit can be used to determine the quality of the salt used, especially to find out whether the salt with adequate iodine content or not, compared to iodometric titration that requires laboratory facilities.

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