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Determination of iodine species content in iodized salt and foodstuff during cooking

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Abstract: Iodine deficiency disorders (IDD) due to iodine decrease or loss in iodized salt and foodstuff during processing or cooking is still a major public health problem in several areas of the world, especially in developing countries. In this study, the determination of iodine content in iodized salt and foodstuffs was done using ion pair high performance liquid chromatography. The results showed that recovery of iodate content using the dry and wet methods were 97.26% and 71.7%, respectively. The rate of recovery of the iodate content in sour vegetable soup (38.33 mg kg^{-1}) and spinach soup (48.98 mg kg^{-1}) during cooking for 70 min at 100°C showed that the iodate content fulfilled the requirements for bioavailability of iodine which is within the range of 30-80 mg kg^{-1}. The results obtained in this study provide insights on the controversy concerning iodine decrease or loss in iodized salt and foodstuffs.

Keywords: Iodine deficiency disorders, iodine species, foodstuffs, and ion pair – HPLC

INTRODUCTION

Iodine Deficiency Disorders (IDD) is a major public health problem in several areas of the world, especially in developing countries. It has been reported that 2.2 billion people (38% of the world's population) live in areas with iodine deficiency and risk its complications. Iodium Deficiency Disorders (IDD) is one of the major nutritional problems in Indonesia, with an estimated 140 million of the population showing IQ point reduction suspected to be caused in part by iodine deficiency with about 42 million people living in endemic areas. (Diosady *et al.*, 1997; WHO, 1999).

Iodine deficiency can cause goitre, cretinism, reduced of intelligence, mental retardation, brain damage, deaf-mutism, and cause miscarriage in pregnant women and stillbirth as well. For iodine deficiency elimination, the government implemented the program of iodized salt usage by the addition of potassium iodate into salt. Iodine deficiency can be caused by consumption of iodineless salt or less iodine containing salt below the minimum requirement (Diosady *et al.*, 1998; WHO, 1999).

A comprehensive review of the literature by Diosady *et al.* (1998) concluded that the stability of iodine in salt is determined by the moisture of the salt and humidity of the atmosphere, bad packaging, light, heat, impurities in the salt, alkalinity or acidity, and the form of packaging in which the iodine is present. Rates variations between iodine losses reflect impurities, moisture content, and processing methods. Conditions of packaging and storage, such as humidity and temperature also influence the final iodine ingredient of the salt; therefore these factors are not always clearly defined in earlier studies.

Several methods have been applied for determining iodate in iodized salt; however most of the methods does not determine and separate the iodine species specifically. Iodometric titration is often used in analysis of iodate and not only determining potassium iodate but also determining all oxidators in solution that may cause the increase of iodine ingredient in iodized salt. Therefore iodometric method is not quite precise for determination of potassium iodate in iodized salt. There is still controversy amongst the public and even among scientists on the loss of iodine in iodized salt and foodstuffs during processing/cooking, caused by the differences in the methods of analysis. Iodine can also be lost during storage and cooking if it is combined/mixed with the vinegar or chilli. To prove the existence of other iodine species in iodized salt, it requires a specific, accurate and precise analysis. In this study, the iodine species content was determined by ion-pair

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HPLC. This method has better selectivity, more sensitive, and more reliable and it was generally better than other methods for determination of ionic sample (Arhya, 1994; Diosady *et al.,* 1997; Saksono *et al.,* 2001; Marihati *et al.,* 2003).

MATERIALS AND METHODS

Materials

Ion pairing reagent of tetrabuthyl ammonium chloride (TBAC) of 40 mg mL^{-1} in water (Merck, Darmstadt, Germany), KH_2PO_4 p.a. (Merck, Darmstadt, Germany), 5 M potassium hydroxide solution and 6 M phosphoric acid (Merck, Darmstadt, Germany), NaCl of 99.5%, KIO_3 p.a. and KI p.a were used as a standard solution. The water, 0.01 M phosphate buffer containing 0.001 M tetrabuthyl ammonium chloride, methanol and acetonitrile 99.5% (v/v) were used as mobile phase of HPLC grade (JT. Beacker-USA). Simulated iodized salt and some foodstuffs were used as samples.

Instrumentation

High performance liquid chromatography system (Hitachi-Japan) equipped with a UV detector, reversed-phase C-18 column (300 x 3.9 mm, and of 10 µm particle size, Phenomenex, Bondclone). Spectrophotometry UV (Beckmen, Germany), vortex mixer, ribbon blender, vacuum filter, climatic chamber, membrane filter of 0.22 and 0.45 µm, pH meter, micro pipette, volumetric flasks and 20 and 50 µL syringes (Hamilton, USA, and SGA, Australia).

Chromatographic conditions

The column was a 300 mm × 3.9 mm, 10 µm particle size, Phenomenex, Bondclone. The mobile phase was 10: 90 (v/v) methanol/phosphate buffer (0.01 M KH_2PO_4 adjusted to pH 7.0 with H_3PO_4) containing 0.001 M tetrabuthyl ammonium chloride. The flow rate of the mobile phase was 1.0 ml min^{-1}, the wavelength for detection was set at 226 nm, the injection volume was 20 µL, the column temperature was at 27°C, the time was 10 min, and the quantization was based on peak area counts.

Sample preparation

The samples and reagent solutions were sonicated for 15 minutes and stored in glass containers and kept at 4°-5°C before use. The pretreatment of all the samples were filtered by 0.22 and 0.45 micro meter membrane filter and centrifuged for extraction of the salt species from the food matrix. The simulation samples of iodized salt were stored in a cold storage.

Standard solution preparation

Approximately 61.14 mg KIO_3 and 65.35 mg KI were transferred into a 50 ml volumetric flask, added with 0,1 M NaCl solution, and mixed. These were the working standards of iodate and iodide with a concentration of approximately 1000 mg L^{-1}. Each of them were made with concentrations of 0.20, 0.60, 1.00, 1.40, 1.80, 2.20, 2.60 mg/L and 0.20, 0.50, 0.80, 1.10, 1.40, 1.70 and 2.00 mg/L, respectively.

Determination of iodine species content in iodized salt with various methods

a. Dry method

The simulated iodized salt sample was made by mixing potassium iodate (122.30 mg) with 1 kg NaCl p.a. in a ribbon blender and mixed at 24 rpm for 10-15 min, until visual homogeneity was achieved. Approximately 0.1 g of the sample was dissolved in 10 mL aquabidest. The sample was analyzed for iodine species content by ion pair HPLC.

b. Wet method

Approximately potassium iodate (1.223 g) was dissolved in 10 mL aquabidest, the solution was sprayed to 1 kg NaCl p.a. in a ribbon blender and mixed at 24 rpm for 10-15 min, until visual homogeneity was achieved. The sample was heated at 40°-50°C for 30 min in the oven. The sample was cooled in desiccators before analyzed. Approximately 0.1 g of the sample was dissolved in 10 mL aquabidest. The sample was analyzed for iodine species content by ion pair HPLC.

Determination of iodine species content in foodstuffs during cooking

Approximately 5 .0 g iodized salt containing 74.85 mg iodate kg^{-1} were mixed into 500 g foodstuffs (i.e. sour vegetable soup and spinach soup) in 1000 mL of Erlenmeyer flask and cooked before consumed. A 50 mL of the sample was taken every 5 min from the beginning until the end of cooking process. The cooking process was carried out with a simple apparatus shown in Figure 2. The samples were analyzed for iodine species content by ion pair HPLC.

RESULTS AND DISCUSSION

Determination of iodine species content in iodized salt with various methods

The major technology for salt iodization process for both large and small scale in Indonesia are the dry mixing (dry method) and wet mixing (wet method) processes (Marihati *et al.,* 2003). The

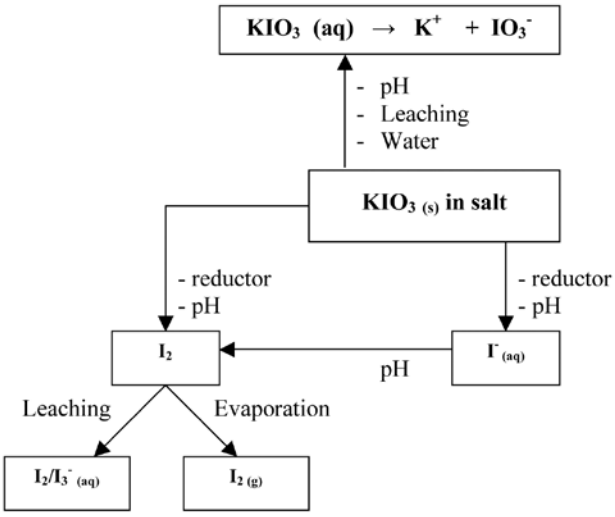

Figure 1: Mechanism of iodate decomposition to iodine and iodide in iodized salt

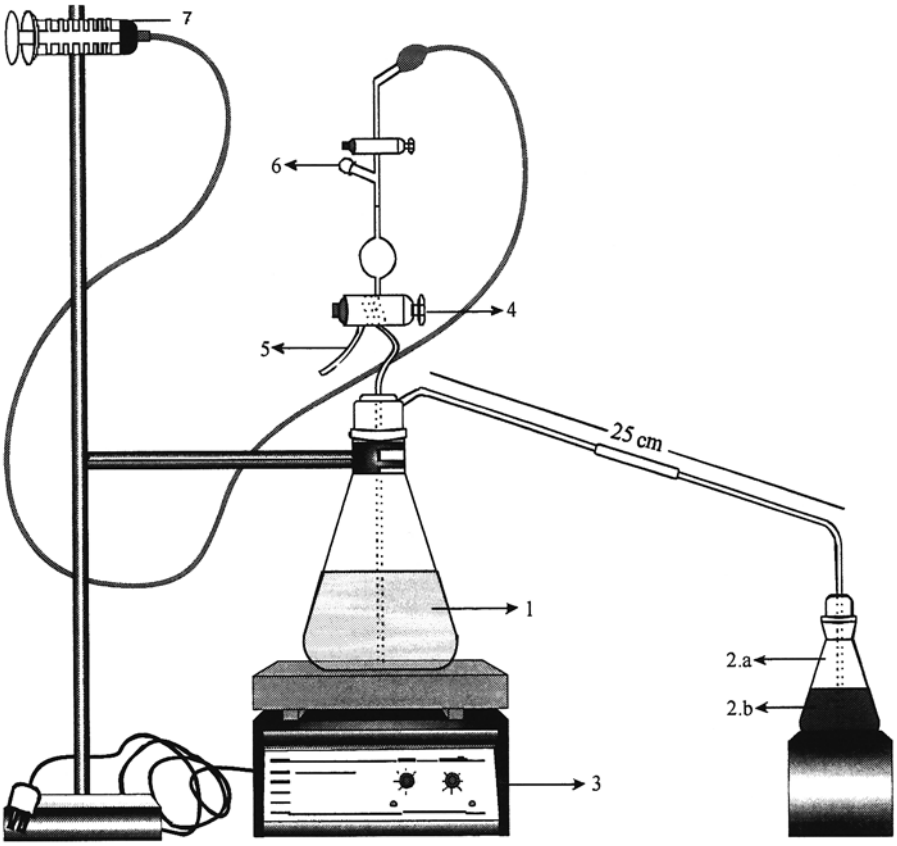

Figure 2: A simple apparatus for determination of iodine (I_2) content in foodstuffs during cooking

Key
1. Sample of foodstuff
2.a. Potassium iodide of 10% (solvent of I_2)
2.b. Accommodate flask of I_2 (g)
3. Magnetic stirrer and heater plate
4. Sample in/out spigot (tap)
5. Output Sample
6. Solvent (water) in
7. Suction pump for sample

results showed that recovery of iodate content using the dry and wet methods were 97.26% and 71.7%, respectively. The dry method was found to be the best process, because it showed the least amount of iodate loss and require no heating. The stability of iodine/iodate can be influenced by heating, moisturing, processing method and humidity of the atmosphere. Iodate content decrease using the wet method could be due to the water content and heating at 40 – 50°C. The stability of iodate in salt can also be determined by the quality and purity of the salt. The effects of purity on iodate retention are shown in Table 1. The retention of salt with purity up to 87% was better if compared with those with less purity (Diosady *et al.,* 1997; Saksono *et al.,* 2001). However it should be noted that many problems can occur in the utilization of iodized salt, including the iodine content of salt being less than the minimum requirement, the loss of iodine from the salt surface, instability of iodine in salt during production and storage, and the food cooking process, the presence of anti-thyroid compounds in food and the interference of ions in analysis by iodometric titration, the traditional method for determining iodine content (Diosady *et al.*, 1997,1998 ; Drobnik and Latour, 1998; Wang *et al.*, 1999).

Determination of iodine species content in foodstuff during cooking

The results showed that the recovery of iodate content in sour vegetable soup was 38.33 mg kg^{-1} and in spinach soup was 48.98 mg kg^{-1} during cooking at 100°C for 70 min, indicating that iodate content have fulfilled the bioavailability requirement which is within the range of 30-80 mg kg^{-1}. The effects of cooking process on iodate stability in certain food (i.e. sour vegetable soup and spinach soup) showed a significant difference in iodate content decrease. Decomposition percentage of iodate into other iodine species and reduction of the highest iodate content occurred in the sour vegetable soup (48.52%), while in the spinach soup (34.62%) during cooking at 100°C for 70 min. The decrease of iodate content and conversion to other iodine species is caused by acidity, moisture content, heating during cooking process, and also influenced by the type of cooking spices and raw materials used. Decrease of iodate content in sour vegetable soup was 56.63% during cooking at 100°C for 35 min, while in *soto* (chicken soup) with coconut milk it was 39.48% at 105°C for 55 min. (Dahro, 1996; Diosady *et al.*, 1997, 1998 ; Arhya ,1998)

According to Arhya (1994, 1998), the type of cooking spices *i.e.,* chilli, *terasi*, pepper and coriander can contribute to the decrease of iodine content. The occurrence of iodate decomposition into other iodine species (iodine and iodide) in cooking spices was caused by iodate reduction with reductor compounds in cooking spices under acidic conditions, for example chili has low pH of about 4. Therefore chilli has the highest ability to reduce iodate content compared to coriander and pepper (Dahro, 1996; Diosady *et al.*, 1997, 1998; Arhya, 1998; Saksono, 2003)

Table 1: Iodine species content in iodized salt determined with various method

Iodization Process	Theoretical iodate content (mg kg^{-1})	Recovery (mg kg^{-1})	Iodate content decrease (%)	Iodide content (mg kg^{-1})
Wet method	100	66.67	33.33	12.50
		68.18	31.82	16.36
		74.55	25.44	19.09
		75.45	24.54	12.50
		73.64	26.35	19.09
	Mean	71.70	28.30	15.91
	Standard deviation	3.98		
Dry method	100	95.45	4.55	2.67
		96.36	3.64	2.27
		98.18	1.82	1.04
		97.27	2.73	1.18
		99.09	0.91	0.51
	Mean	97.27	2.73	1.53
	Standard deviation	1.44		

Table 2: Iodine species content in sour vegetable soup

Length of cooking process (minute)	Recovery of iodate content (mg kg^{-1})	Formed iodide content (mg kg^{-1})	Ratio of I^- / IO_3^-
0	66.73	0.72	0.0108
5	65.02	0.36	0.0055
10	62.29	0.33	0.0053
15	60.59	0.3	0.0050
20	59.11	0.24	0.0041
25	51.81	0.22	0.0042
30	51.45	0.19	0.0037
35	51.13	0.19	0.0037
40	49.89	0.18	0.0036
45	47.59	0.18	0.0038
50	42.51	0.17	0.0040
55	39.65	0.17	0.0043
60	39.14	0.14	0.0036
65	38.35	0.15	0.0039
70	38.33	0.14	0.0037

Table 3: Determination of iodine content in spinach soup

Length of cooking process (minute)	Recovery of iodate content (mg kg^{-1})	Formed iodide content (mg kg^{-1})	Ratio of I^- / IO_3^-
0	71.73	7.12	0.0993
5	69.02	6.36	0.0921
10	67.89	5.33	0.0785
15	66.29	5.13	0.0774
20	64.19	4.97	0.0774
25	62.78	4.72	0.0752
30	58.65	4.59	0.0783
35	57.43	4.39	0.0764
40	55.93	4.18	0.0747
45	53.29	3.98	0.0747
50	51.87	3.77	0.0727
55	50.86	3.56	0.0700
60	49.74	3.53	0.0710
65	49.35	3.49	0.0707
70	48.98	3.54	0.0723

The iodine formed was analysed by Spectrophotometer at a wavelength of 480 nm for detection. The results obtained showed that the average iodine (I_2) content was 30.17 mg L^{-1} (equal to 20.79 mg L^{-1} as iodate). According to Bhatnagar *et al.* (1997), the dispersion of iodate to other iodine species (iodide and iodine) during heating/cooking shows that iodate is unstable in that condition. Determination of iodine content which evaporates during cooking was conducted in this study and the results showed significant amount of iodine evaporation. However, the mechanism of iodate dispersion to other iodine species bound by other substance in cooked food has not been conducted, and this needs further research for its elucidation.

Iodine is an essential trace element present in nature. In human nutrition, iodine is an integral part of the thyroid hormones that play an important role in controlling the rate of basic metabolism and in reproduction. The iodization of table salt was successful in decreasing markedly the incidence of simple goiter in the supplemented population. The bioavailability of iodide from iodized salt is only 10% of the estimated 0.75 mg iodide in iodized salt consumed per day. This amount, 0.075 mg of bioavailable iodide, represents less than 1% of the amount of iodide used in Marine's (1920) study (i.e., 9 mg) and also less than 1% of the recommended daily intake of iodine from Lugol solution. (Edmundson *et al.*, 1999; Abraham, 2006)

CONCLUSION

The results showed that recovery of iodate content using the dry and wet methods were fairly high with the dry method resulted in the least amount of iodate loss. In addition, the rate of recovery of the iodate content in sour vegetable soup (38.33 mg kg^{-1}) and spinach soup (48.98 mg kg-1) during cooking for 70 min at 100°C showed that the iodate content fulfilled the requirements for bioavailable iodine which is within the range of 30-80 mg kg^{-1}. The results obtained in this study provide insights on the controversy concerning iodine decrease or loss in iodized salt and foodstuffs.

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