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Analysis of Iodine Level (KIO_3) based on the Formation of Amylum-Iodine Complex using UV-Vis Spectroscopy

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ABSTRACT

Iodine is an important component that needed by the body in the synthesis of thyroid hormones in order to maximize humans' growth and development. Besides, iodine also plays a role in the regulation and metabolism system in the human body. Iodine Deficiency Disorders (IDD) is one of the nutritional problems which is an inhibiting factor in the development of human resources because it can cause disruption of mental development and human intelligence. Iodine in food is found from table salt, it found in the form of KIO_3 with a standard level of iodine in table salt that is 30ppm-80ppm. This research aims to determine the iodine levels in the four kinds of table salt using spectrophotometric methods based on the formation of the starch iodine complex. The formation of the starch iodine complex performed by reacting I- with the oxidizer IO_3^- in an acidic atmosphere with starch indicator. The resulting blue color was analysed by visible spectrophotometry and produce absorbance data. The measured iodine content is equal to samples A, B, C, and D showing results less than 30 ppm so it can be concluded that iodine in table salt still does not meet the SNI standards that should be around 30ppm-80ppm.

Keyword: iodine, spectrophotometry, absorbance, table salt

1. INTRODUCTION

The role of iodine is important in the synthesis of thyroid hormones in the human body in order to maximize growth and development. Besides iodine also plays a role in the regulation and metabolism system in the body. Because of the important role of iodine, if iodine levels are lacking in the body it can lead to nutritional problems called IDD (Kim, Yun, Lee, & Kang, 2017). Therefore, human should consume any food that contain iodine to fulfil the standard iodine level in the body.

The natural dietary sources of iodine include vegetables, fruit, milk, spinach, egg, meat, and sea foods. However, natural foods that became the sources of the iodine that consume by human may not meet standard requirement by the body since the iodine also maybe less concentration

(Zimmermann, 2009). The content of iodine in food varies and is influenced by geographical location, season, and how to cook it (Ftwi, Mengistie, & Abdo, 2018). Iodine is not found in pure form but as an iodide compound, iodate, or a combination of both (Subhan, 2014). Adequate intake of iodine can be achieved by consuming the iodized salt. Iodization salt is done by addition of iodate in table salt sample that often consume by human. Then determination of iodate in any table salt samples is important because it may vary the conditions like enviromental conditions, the nature of transport, packing conditions, and cooking methods that is influence about amount of iodate (Brucherseifer, Cripps, Guentay, & Jaeckel, 2003).

The results of the 2013 Basic Health Research shows that the percentage of households in Indonesia consuming salt signified two findings, an adequate iodine content of 77.1% and less iodine of 14.1%. This value has not yet reached the Iodized Salt target for all (Universal Salt Iodization/ USI), that declared at least 90% of households have consumed iodized salt. As a potential food commodity, iodine-containing salt can be made by fortifying iodine (KIO_3) as a source of iodine, with a percentage of 90% coming from food and 10% coming from water. Many survey results circulating in the community of iodine content in salt still do not meet the requirements of SNI No. 01-3556-2000 i.e., 30-80 ppm iodine as KIO_3 (Djokomoelijanto, 2007).

The main strategy in overcoming IDD is through universal salt iodization (USI) (WHO, 2007). Furthermore, WHO (2007) explained that the iodization process was carried out through salt because of the wide distribution of consumption and low iodization costs. Many countries have adopted USI programs and disruption due to iodine deficiency is become increasingly scarce (Prete, Paragliola, & Corsello, 2015). Iodized salt usually contains 20-40 ppm (μg/ g) iodate which turns into iodine 15-30 ppm (Rebary, Paul, & Ghos, 2010), depending on the water content and food supply in an area (Pan, Zhang, & Li, 2015). The amount of iodate that added will affect the salt iodization process and the quality of the salt on the market. The stability of iodized salt is influenced by several factors, including environmental humidity, sun exposure, heat exposure, acid reaction in salt, impurities, and packaging materials (Etsein, et al., 2017).

The incompatibility of iodine content in the package with the actual content can be caused by several things among others: (1) Effect of temperature, salt that is stored and exposed to direct sunlight (or exposed to heat) will easily evaporate or melt. It can affect the level of iodine in table salt; (2) Effect of packaging, salt that is packed with clear plastic loses its iodine level at most (14.40 % - 22.60 %). This is presumably because the clear plastic used is from the type of PE. This type of plastic has a high-water vapor penetration (Buckle, 1987). In addition, bright colors of plastic can accelerate the oxidation of iodine more. In addition, this type of plastic is not resistant to oxygen, where oxygen permeability can occur through plastic pores. (Syarief, 1986). This situation causes the oxidation of potassium iodate present in salt which then releases I_2 in the form of gas into the air; (3) Addition of KIO_3, the KIO_3 added is less homogeneous, or is less calculated so that the KIO_3 content in the salt is low iodized consumption. (Amanati, 2017)

There are various analytical methods for determination of iodate in iodized salt samples. Firstly, with kinetic spectrophotometric methods (Ni Y, 2007), secondly with flow injection analysis (Shabani & Ellis PS, 2011), then with microspectrophotometry after liquid-phase microextraction (Pereira , Ferreiro, Lavilla, & Bendicho, 2010) and many other methods can determination it. Therefore, this research aims to determine the iodine levels in the table salt using spectrophotometric methods based on the formation of the starch iodine complex.

2. RESEARCH METHOD

Iodine analysis using a spectrophotometer (WYD Iodine Checker) has the same results with the iodometric titration method. In addition, WYD Iodine Checker is an appropriate, sensitive, affordable and easy-to-use tool according to simple laboratory procedures (Welsey, Makhmudov, Pfeiffer, & Caldwell, 2004). Iodine, iodide, and iodate contain emissions that can be absorbed by the UV-Vis spectra (Kireev & Shnyrev, 2015). Potassium iodide (KI) and potassium iodate (KIO3) are used

as sources of iodine in salt iodization. Fortification (addition) of iodate is much more stable than iodide (Zoysa, Hettarachchi, & Liyanage, 2016). Determination of iodine content using this spectrophotometric method is based on the formation of the starch iodine complex using iodine oxidizing agents. In the formation of amylum-iodine complex using iodate oxidator, it is possible to be influenced by several factors from the results of the formation of complexes that are formed, including the reaction time of the formation of amylum-iodine complex, the use of potassium iodate concentration that used as an oxidizer, and the iodide concentration to be determined (Febrianti, Sulisyanti, & Atikah, 2013).

Determination of iodide levels based on spectrophotometric methods developed by Kolthoff-Sandell used arsenite reagents (As^{3+}) to oxidize iodides. However, this determination requires a complex analysis and a lot of reagents that are expensive, while a fast, simple, accurate, and inexpensive method of monitoring iodine deficiency is needed at this moment. This constraint could solve by determining the iodide using spectrophotometric methods based on the formation of the starch iodine complex using iodic oxidizing agents. This spectrophotometric method is based on the oxidation-reduction reaction and the formation of starch-iodine-colored complexes which produced absorbance values (Wisnu, 2008).

Iodate is one of several oxidizing agents that can oxidize the iodide into iodine in an acidic atmosphere. In this study, the reaction was carried out under acidic conditions since the starch that used as an indicator will be hydrolyzed, in addition to this situation the iodide ion (I^-) that produced can be converted into iodine (I_2) with the presence of oxygen (O_2) from free air, this reaction involves ions (H^+) from acids following this reaction reaction: (Abner Tonu, 2014)

$$IO_3^- + 5I^- + 6H^+ \rightarrow 3I_2 + 3H_2O \quad (1)$$

$$I_2 + \text{Amylum} \rightarrow I_2\text{-Amylum} \quad (2)$$

The iodine and starch will form starch-iodine complex which identified by the formation of a blue color. In this spectrophotometer methods, the absorbance of the iodine-starch complex will be measured.

Several stages were carried out including the manufacture of 100 ppm KIO_3 standard solution, determination of the maximum wavelength (λ), optimization of measurement time, optimization of maximum pH, complex optimization, determination of standard curves, and determination of iodine content in the table salt samples. Optimizations that are carried out before taking measurements are intended in order to obtain the maximum conditions and the high accuracy.

3. RESULTS AND ANALYSIS

3.1. Determination of optimum measurement

Based on the results of the determination of the maximum wavelength complex of amylum-iodine blue, the visible spectrum and complementary colors found within the range of 570nm-590nm. While, the optimum peak obtained at a wavelength of 590 nm with an absorbance value of 0.792. Figure 1 presents the optimum measurement of this research.

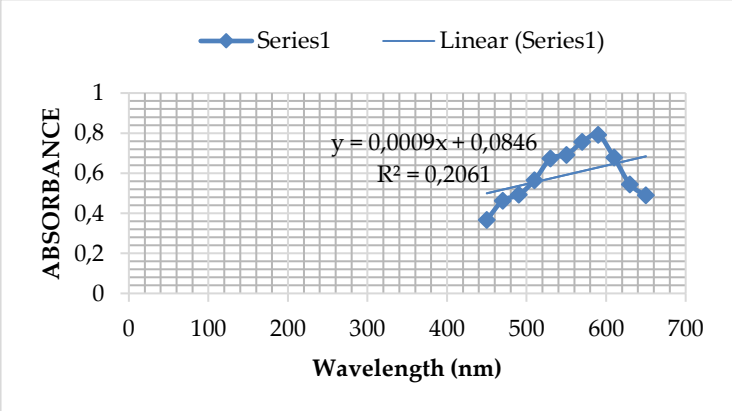

Figure 1. The maximum wavelength of amylum-iodine complex

After obtaining the maximum wavelength, the optimization measurements are then carried out at a wavelength of 590 nm. The next step is determining the maximum time optimization with interval of minute 1 using 590nm wavelength. This activity aims to determine the ideal time needed for producing a colored complex of the starch amylum-iodine solution since it is very important in yielding the absorbance value. The findings on this step seen in Table 1.

Table 1. Determination optimum of time

No.	Time (minutes)	Absorbance
1.	1	0,743
2.	2	0,733
3.	3	0,709
4.	4	0,684

The third measurement is determining the maximum pH after the addition of H_2SO_4 about 1 drop or 0,05 mL. The highest absorbance found to be 0.743 in the pH of 3,2 as seen in Table 2.

Table 2. Determination Optimum of pH

No.	Volume H_2SO_4	Absorbance	pH
1.	0,05 mL	0,743	3,2
2.	0,1 mL	0,629	2,9
3.	0,15 mL	0,588	2,4
4.	0,2 mL	0,533	2

The last optimization carried out is the complexing optimizer using starch in various amounts. The optimal absorbance (1,135) found with the addition of 0,5 mL starch of 1% as presented in Table 3.

Table 3. Determination optimum complexing volume

No.	Volume Amilum 1%	Absorbance
1.	0,2 mL	0,743
2.	0,3 mL	1,033
3.	0,4 mL	1,101
4.	0,5 mL	1,135

3.1. Measurement of the Sample

After all optimization measurement have been made, a calibration curve is carried out. In this study the standard solution has concentration of 20 ppm, 40 ppm, 60 ppm, 80 ppm and 100 ppm measured at a wavelength of 590 nm. After obtaining the absorbance value of the standard solution (see Table 4), the standard curve is drawn and it illustrated in Figure 2.

Table 4. Determination of absorbance of standard solutions

No.	Consentration KIO_3 (ppm)	Absorbance
1.	20	0,442
2.	40	0,667
3.	60	0,735
4.	80	0,804
5.	100	0,943

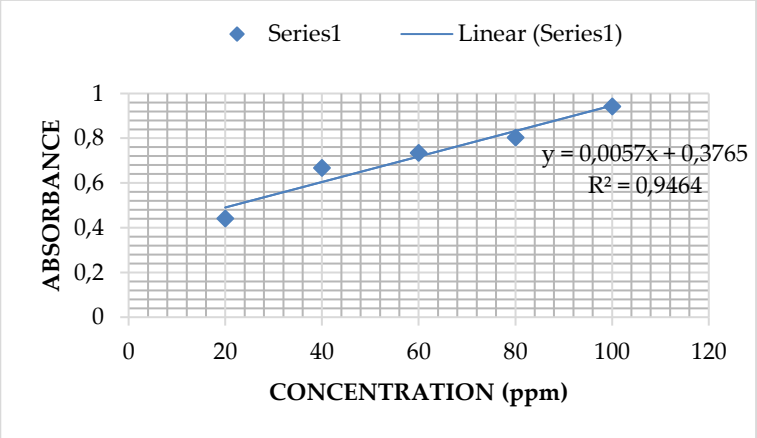

Figure 2. Calibration curve of standard solution

Based on Figure 2, the analysis obtained a linear equation for the standard curve of y = 0.0057x + 0.3765 with a value of R^2 = 0.9464. The X-axis represents iodine concentration and the Y-axis represents the absorbance value measured in UV-Vis spectrophotometry. The curves and regression equations used to determine iodine concentrations in various brands of table salt that used as the samples of this research.

The following steps is the measurement of absorbanse of the four kinds of table salt samples. The sample used was 5 grams of table salt which dissolving in a 25 mL volumetric flask. The measurement conducted in duplo, thus it carried out 2 times. The results of the analysis shown in Table 5.

Table 5. Experimental Data

No.	Sample	Absorbance	Concentration	Average Concentration
1.	Sample A	0,482	18,5 ppm	17,3 ppm
		0,468	16,06 ppm	
2.	Sample B	0,457	14,12 ppm	14, 6 ppm
		0,473	15,08 ppm	
3.	Sample C	0,470	16,4 ppm	15,7 ppm
		0,462	15 ppm	
4.	Sample D	0,467	15,87 ppm	16,3 ppm
		0,472	16,75 ppm	

Based on the linear equation y = 0.0057x + 0.3765, the value of Y or absorbance obtained in sample A is 17.3 ppm, sample B 14.6 ppm, sample C 15,7 ppm, and sample D 16,3 ppm. The measured iodine content is equal to samples A, B, C, and D were less than 30 ppm. Therefore, the iodine in table salt still does not meet the SNI standards since it should be around 30ppm-80ppm. These results are not matched with the amount of iodine written on its packaging that stated the iodine levels above 30ppm. The incompatibility of iodine content in the package with the actual content can be caused by several factors: (1) Effect of temperature, salt that is stored and exposed to direct sunlight (or exposed to heat) will easily evaporate or melt. This can affect the level of iodine in the table salt; (2) Effect of packaging, salt that is packed with clear plastic loses its iodine level at most (between 14.40 percent to 22.60 percent). This is presumably because the clear plastic used is from the type of PE. This type of plastic has a high-water vapor penetration (Buckle, 1987). In addition, bright colors of plastic can accelerate the oxidation of iodine. This type of plastic is not resistant to oxygen, where oxygen

permeability can occur through plastic pores. (Syarief, 1986) . This situation causes the oxidation of potassium iodate present in salt were releases I_2 in the form of gas into the air; (3) Addition of KIO_3, the KIO_3 added is less homogeneous, or is less calculated so that the KIO_3 content in the salt is low iodized consumption (Amanati, 2017).

4. CONCLUSION

In this research, the optimization of the measurement of iodine levels in table salt carried out at a maximum wavelength of 590 nm, a measurement time of 1 minute, volume H_2SO_4 of 1 drop, pH of 3.2, and complexing 1% of 5 drops of starch. The SNI requirements stated the salt consumption should be ranging from 30ppm-80ppm. Unfortunately, the findings on this research of samples A, B, C, and D were contain iodine less than 30 pmm. Therefore, the iodine in table salt still does not meet the SNI standards. These results are not matched with the amount of iodine written on its packaging that stated the iodine levels above 30ppm. Several factors were discussed the rcause of reduction of iodine levels in the table salt include the packaging, storaging and distributing process to the market.

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