Effect of The Amount of KIO_3, Water, and Stirring Time on Salt Quality in The Iodization Process

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Abstract. Currently, IDD (Iodine Deficiency Disorder) is a problem that still requires attention from the Indonesian government. IDD problems can be overcome by adding iodized salt to daily food. However, the quality of consumption salt produced by small industries in Indonesia is still relatively low in terms of the even distribution of iodine in it. This study aims to develop a simple process to increase the uniformity of iodine distribution in salt. The variables studied were the concentration of potassium iodate solution (KIO_3) which was sprayed using a sprayer and the stirring time. This research consists of: (1) raw material analysis; (2) main research and (3) product analysis. The main process is carried out by stirring the salt in the helical ribbon mixer. The observed response was the level of homogeneity of the iodine distribution. The highest degree of homogeneity of iodine distribution was found in the addition of 7.5 mL H_2O_2/kg salt with 60 ppm KIO_3 in it, and the stirring time was 5 minutes. The experimental results show that the amount of KIO_3 used (and its interaction with other factors) affects the degree of homogeneity of iodine distribution in salt products.

Keywords: Helical ribbon mixer, Homogeneity of iodine distribution, Iodine Deficiency Disorder, Salt, Spray Mixing

1 Introduction

1.1 Background

Salt is one of the most widely used food ingredients worldwide. Besides its consumption in food, salt is also utilized in various industries such as chemical, pharmaceutical, and leather tanning [1]. Salt contains essential minerals like sodium and chlorine, which play a crucial role in supporting the body's metabolic processes. Additionally, salt serves as a carrier of iodine in the human body. Adequate iodine intake is essential to prevent severe health issues caused by iodine deficiency.

Iodine deficiency can indeed lead to various health problems, including thyroid gland disorders such as goiter. It can also contribute to the development of hyperthyroidism and result in growth retardation, stunting, and delayed mental development in

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toddlers. It highlights the importance of maintaining sufficient iodine levels in the body to prevent these adverse health effects.

The process of salt iodization conducted by farmers through spray mixing does have limitations, particularly regarding the uneven distribution of iodine in the final product [2]. Despite these limitations, a significant portion of Indonesia's population, especially outside of Java, still relies on salt directly produced by salt farmers for their daily food consumption.

1.2 Literatures Review

Iodized table salt is a product that is regulated by the government, including through the Indonesian Industrial Standards (SNI) as shown in Table 1. The process of making iodized table salt involves fortification, which aims to enhance the nutritional value of the food and maintain adequate nutrition in the body. Several compounds are permitted for iodine fortification, including calcium iodide, calcium iodate, potassium iodate, sodium iodide, and sodium iodate [3]. However, potassium iodate is commonly used due to its good stability, low water solubility, and oxidation resistance [4]. The stability of this compound helps minimize the release of iodine into the environment caused by oxidation, salt moisture, and impurities [3].

There are two main methods of fortification: spray mixing and dry mixing. The difference lies in how potassium iodate is added. In spray mixing, the salt is fortified with a potassium iodate solution, while in dry mixing, potassium iodate powder is used [3]. During the fortification process, challenges arise in achieving uniform iodine distribution and preventing iodine release into the environment [2]. Factors that influence the fortification outcomes include the type of mixer [5], the amount of iodine used [1], the amount of water used [6], and the stirring time. Research by Assey (2009) suggests that using smaller amounts of iodine compounds improves iodine distribution uniformity. Additionally, using larger amounts of water may result in a higher standard deviation in the distribution of iodine within the salt product.

The objective of this study is to enhance the uniformity of iodine distribution, particularly in salt products produced by salt farmers. The aim is to enable farmers to produce iodized salt products that exhibit good iodine distribution uniformity and comply with the Indonesian National Standard (SNI). Through the findings of this study, it is anticipated that improvements can be made in the fortification process, leading to iodized salt products that meet the required quality standards and provide consistent levels of iodine distribution. The development of this research has been carried out quite extensively by various researchers. The study conducted by Widjaja et al. (2019) varied the stirring time and particle size to observe the uniformity of KIO_3 in the resulting salt. The research by McGee et al. (2017) and Modupe et al. (2021) varied the amount of potassium iodate usage. Assey et al. (2009) conducted a study that varied the iodization stirring time and the amount of potassium iodate used. Furthermore, researchers Sangakkara (2011) and Mannar (1995) provided some commonly used values for the duration of stirring time, amount of water, and potassium iodate used, but they have not yet led to results on the uniformity and average level of KIO_3 in the resulting salt product. In this research, there is a novelty where three variables are varied (stirring time, amount of water usage, and amount of KIO_3). Then, the influence of these variables will be examined on the response of the degree of homogeneity of iodine distribution and the average iodine level in the salt product.

Table 1. Quality requirements for iodized table salt according to SNI

Tested parameter	Unit	Requirement	Ref.
Water	Mass fraction,%	Maximum 7	[7]
Salt (NaCl)	Mass fraction,%	Minimum 94	[7]
Insoluble material	Mass fraction,%	Maximum 7	[7]
Iodine as KIO_3	ppm	30 – 80	[7]
Cadmium	ppm	Maximum 0.5	[7]
Lead	ppm	Maximum 10	[7]
Mercury	ppm	Maximum 0.1	[7]
Arsenic	ppm	Maximum 0.5	[7]
Calcium and magnesium (dry basis)	Mass fraction,%	Maximum 1	[8]

2 Experiment Section

2.1 Raw Materials and Equipments

The salt used for the iodization process is sourced from O'lio village, Kupang Regency, East Nusa Tenggara (NTT), and is commonly known as "boiled salt" among the local community. Boiled salt is produced by dissolving raw salt (K3 quality) and then recrystallizing it using a conventional stove with firewood as fuel. This process allows for the removal of certain soluble impurities (such as calcium, magnesium, and sulfate ions) as well as insoluble impurities (such as dust, sand, or other non-soluble materials) present in the salt.

The potassium iodate used to make the fortificant solution is Pudak Scientific CKA 94/125 125-gm. The potassium iodate used has a minimum purity of 99.5%.

The equipment utilized for iodization/fortification is a helical ribbon mixer. The helical ribbon mixer used has a 50 kg capacity for salt and features an output section (on the left) serving as a product storage area. Additionally, it consists of an inlet section (located on the top side) and is powered by a motor that drives two ribbons with gasoline fuel. The visual representation of the equipment and its dimensions are provided in Figure 1 and Table 2, respectively.

2.2 Experimental Design

There were three variables observed in this study, i.e. (1) the amount of potassium iodate added; (2) the amount of water used and (3) the stirring time. The number of batches conducted was 12 as shown in Table 3.

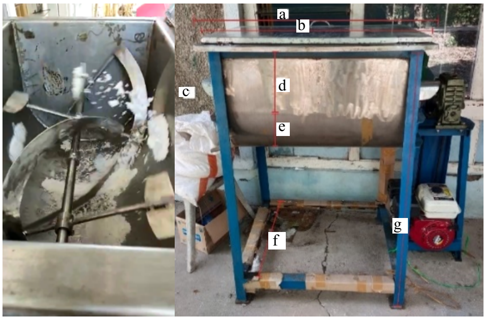

Fig. 1. Ribbon mixer used for iodization process

Table 2. Dimension of ribbon mixer

Part	Dimension
a	90.2 cm
b	57.4 x 86 cm
c	12 x 14 cm
d	29 cm
e	27.7 cm
f	66 cm
g	119.3 cm
Impeller	2 Helical ribbon

Table 3. Experimental design

Variation	Water volume (mL/kg salt)	Stirring time (minutes)	Amount of KIO_3 used (mg/kg salt)
1	2.5	5	40
2	7.5	5	40
3	2.5	15	40
4	7.5	15	40
5	2.5	5	60
6	7.5	5	60
7	2.5	15	60
8	7.5	15	60
9-12	5.0	10	50

2.3 Spray Mixing Method

In the spray mixing method, the salt is introduced into the ribbon mixer. Then, the mixer stirs the salt at a medium speed. The first spraying of the solution is initiated when the salt starts rotating. The spraying continues until the spray solution is completely used up, and there is a delay of about 1 - 1.5 seconds between each spraying cycle.

While the salt is being stirred, the spraying is conducted following the spray pattern illustrated in Figure 2 for all the experimental variations. Once the stirring time specified for each variation is completed, sampling is carried out three times at different positions within the mixer, namely the left (L), center (C), and right (R) positions.

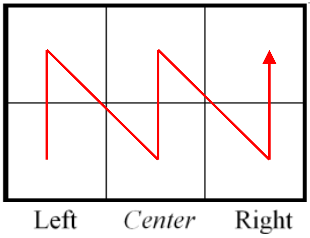

Fig. 2. Spraying pattern in iodization (top view of mixer)

2.4 Analysis

The analysis of NaCl, Ca^{2+}, and Mg^{2+} content in the raw materials was conducted using various titration methods. NaCl analysis was performed through Argentometric titration. For Ca^{2+} and Mg^{2+} ion analysis, a complexometric titration method using EDTA solution was employed. The determination of water content was carried out using the gravimetric method, where the moisture content is calculated based on the amount of water that evaporates during the drying process. The iodine content as KIO_3 was determined using iodometry. To calculate the uniformity index, the standard deviation of 9 samples per batch was used.

For qualitative analysis, the distribution of iodine was observed through black dots that appeared after the analysis. Two reagents were used for the qualitative tests. Initially, the salt sample was spread in a container and then sprayed with a solution containing KI (1%) and HCl (5%) in a 1:1 ratio. Subsequently, the sample was sprayed with a flour solution.

3 Results and Discussion

There are two research results, quantitative and qualitative results. Quantitative results was presented in Table 4 in the form of standard deviation data and average iodine levels for each batch. Meanwhile, the qualitative results was presented in Figure 3. To obtain data from Table 4, each product sample on the left (L), right (R) and center (C) for certain variations that have been sampled will be analyzed for iodine levels with three repetitions. Then all repetitions of the analysis will be averaged for iodine levels and the standard deviation value will be calculated (which represents the level of uniformity of iodine in salt).

3.1 Preliminary Analysis of Boiled Salt

The initial analysis carried out on boiled salt is the analysis of NaCl, Ca^{2+} and Mg^{2+} content, , insoluble materials content and moisture content.. The analysis results show 82.80% NaCl (dry basis), 0.12% Ca^{2+} (dry basis), 0.49% Mg^{2+} (dry basis), and 13.91% moisture content. In addition, there was 7.76% insoluble material (dry basis), along with other content. The analysis results also showed that the salt content was low enough to require drying before iodization.

Table 4. Quantitative results for iodization

Variation	Iodine content at left (L, triplo) side (ppm)	Iodine content at center (C, triplo) side (ppm)	Iodine content at right (R, triplo) side (ppm)
1	32.3581	38.2767	26.4443
	30.9695	37.2318	27.1383
	34.0997	38.6230	26.0960
5	34.6144	37.1340	54.0806
	36.4141	33.8913	49.7540
	35.3334	34.2494	43.9835
6	43.6287	49.6265	45.6284
	46.2936	46.6257	42.2952
	45.2968	47.6281	40.6326
2	31.4134	29.1234	31.4130
	31.0846	30.4317	28.4693
	30.1043	29.1240	29.1230
8	33.7018	32.0669	38.9376
	34.6870	34.3582	41.5569
	34.3567	34.6846	41.8808
7	35.6955	34.6109	36.7759
	37.8567	36.4156	38.9412
	34.6113	40.3799	42.9038
4	29.5758	33.7507	35.1425
	29.9246	28.5331	34.0996
	30.9669	29.5766	38.9713

Table 4. Quantitative results for iodization (continued)

Variation	Iodine content at left (L, triplo) side (ppm)	Iodine content at center (C, triplo) side (ppm)	Iodine content at right (R, triplo) side (ppm)
8	29.1211	35.0127	50.3892
	29.4483	36.9741	54.6462
	30.7587	39.5957	63.4748
3	40.7120	33.0565	64.7209
	41.0602	32.0120	68.8893
	41.7561	33.7510	74.4647
9	34.4477	44.8874	35.8404
	33.0568	44.5393	35.4911
	34.0982	43.4965	35.4925
10	29.1236	36.6502	53.3328
	30.1051	38.6107	52.3573
	30.4319	41.5576	49.0836
11	35.3287	38.5797	37.8581
	32.0876	44.3491	42.1817
	36.0540	38.5769	43.6273
12	36.0556	36.0548	41.1024
	40.3808	37.8583	42.5439
	36.7770	40.3829	37.8587

3.2 Discussion of Qualitative Test Results

Based on the qualitative tests carried out, it can be seen that some of the stirring was quite good. Almost the entire surface of the salt turns blackish after spraying the two solutions and shows the presence of iodine in the relevant area. Sample number 7 has fairly good uniformity characterized by a fairly homogeneous distribution of black color. This suggests that the iodine content was evenly distributed in this sample. Based on the quantitative results, sample run 7 also showed a relatively small standard deviation value 1.1184 (indicating good iodine uniformity). In addition, samples number 5, 15 still have some areas that are still white and indicate that there is no iodine in that area (uniformity has not been achieved). This indicates that iodine distribution in these samples was not uniform and certain areas lacked iodine, resulting in non-uniform iodine content. Samples number 3, 11 have some areas of salt that are solid black in color. This indicates that these areas have a higher iodine content compared to other salt areas. This is also depicted through quantitative data where run 3 and 11 have relatively large standard deviation values of 16.7268 and 9.5925, respectively.

Table 4. Quantitative results for iodization (continued)

Varia-tion	No. Run	Average iodine content (ppm)	Standard deviation	Minimum value (ppm)	Maximum value (ppm)
1	5	32.3597	5.0550	26.0960	38.6230
5	16	39.9394	7.5123	33.8913	54.0806
6	1	45.2951	2.7522	40.6326	49.6265
2	7	30.0319	1.1184	28.4693	31.4134
8	8	36.2478	3.5906	32.0669	41.8808
7	14	37.5768	2.7680	34.6109	42.9038
4	6	32.2823	3.4346	28.5331	38.9713
8	10	41.0467	12.3133	29.1211	63.4748
3	3	47.8247	16.7268	32.0120	74.4647
9	4	37.9278	4.8720	33.0568	44.8874
10	11	40.1392	9.5925	29.1236	53.3328
11	13	38.7381	4.0486	32.0876	44.3491
12	15	38.7794	2.3784	36.0548	42.5439

3.3 Discussion of Quantitative Test Results

Each left, right and middle product sample for a particular variation that has been sampled will be analyzed for iodine content with a repetition of three times. Then all repetitions of the analysis will be averaged (iodine content) and calculated the standard deviation value (which represents the level of uniformity of iodine in salt). The smaller the standard deviation value, the better the uniformity of iodine distribution, and vice versa.

Based on Table 4, there are several runs that especially have KIO_3 levels below 30 ppm including runs 5, 7, 6, 10, 11. Of these runs, three of them (runs 5, 7, and 10) are runs that use variations of KIO_3 usage of 40 ppm. This can be caused by the possibility of the spray solution falling/sticking to the mixer wall so that it does not hit the salt part.

3.4 Average Iodine Content

The effect of the amount of KIO_3 usage factor on the average iodine level can be seen through the data of the upper (60 ppm) and lower (40 ppm) limits of the 12 trials that have been carried out. Figure 4 below shows how the effect of KIO_3 on the average response of iodine content in salt products. The more KIO_3 used, the higher the average iodine content in the salt product. The margin of error in Figure 4 is also not too large so this trend is acceptable. This is because the amount of KIO_3 used will be proportional to the iodine content in the salt products.

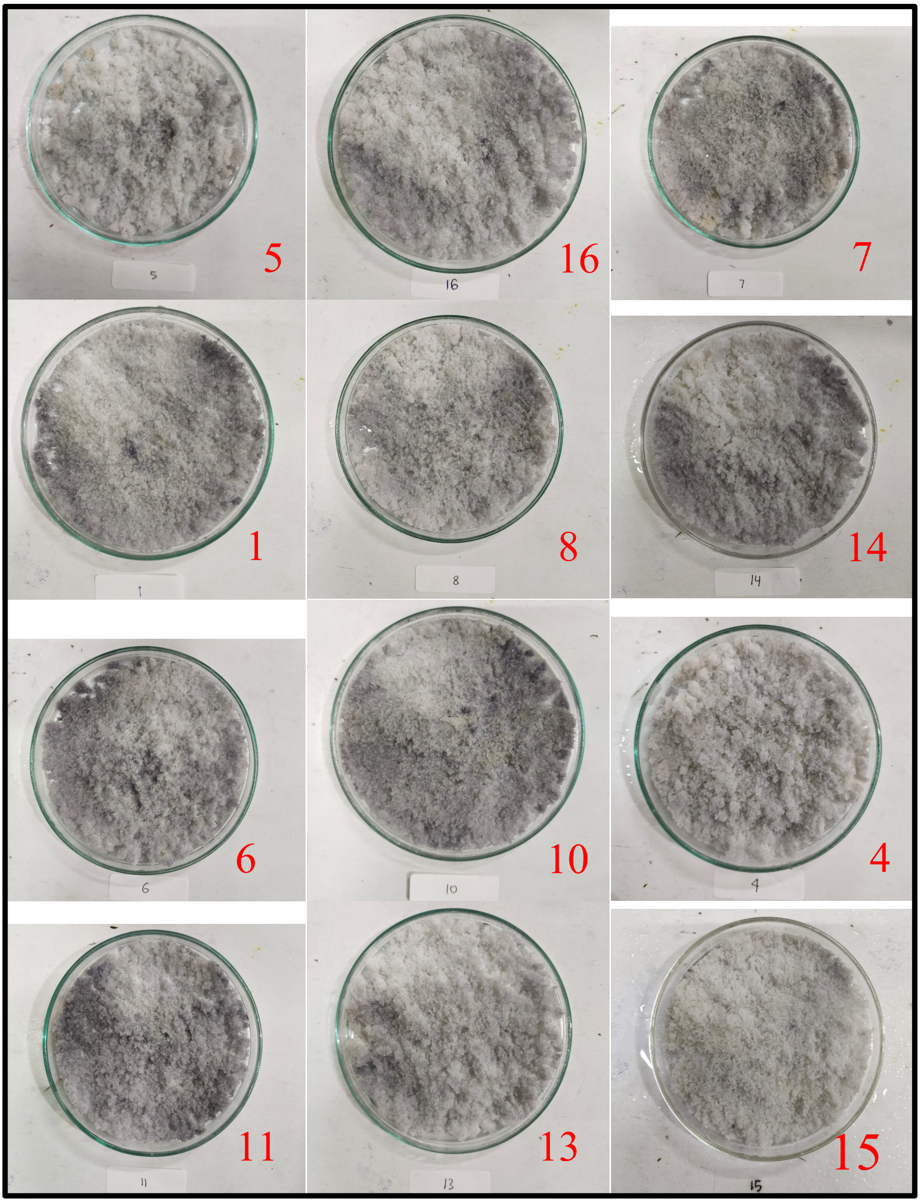

Fig. 3. Qualitative result for all runs

Meanwhile, the interaction effect of the amount of water used and the amount of KIO_3 used also affected the average iodine content response. The trend is shown in Figure 5. With the use of less KIO_3 (black line, 40 ppm), it was found that the use of more water decreased the average iodine content. The interaction effect of stirring time and the amount of KIO_3 on the average iodine content can also be seen with the trend in Figure 6.

3.5 Iodine Distribution Uniformity (Standard Deviation)

Based on the three factors varied, only the factor of using the amount of KIO_3 has an effect on the standard deviation. Through Figure 7, it can be seen that the curve has a certain trend. The trend is that the amount of KIO_3 used during the iodization process will be proportional/linear in relation to the resulting standard deviation. The more KIO_3 used in the iodization process, the higher the standard deviation value (meaning that the uniformity of iodine distribution is not good). This is shown by the trend line with a positive slope that is not too steep. This suggests that there is a possibility that the amount of KIO_3 used will affect the standard deviation but the wide error in Figure 7 may bias the deduction.

The trend of the results obtained is in accordance with the existing trend based on the literature where the amount of KIO_3 usage will be proportional to the standard deviation value [9]. This is because, if during the iodization process with the use of a higher dose of KIO_3 there is uneven mixing, then certain parts of the salt that are iodized will have higher KIO_3 levels than parts of the salt that are not exposed to the spray solution. Based on the data, the use of a higher amount of KIO_3 is more likely to result in a greater difference between the minimum and maximum iodine levels than the use of less KIO_3.

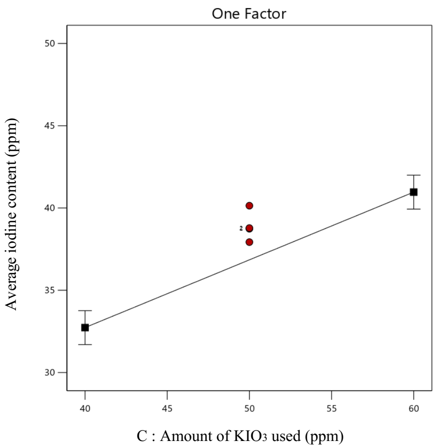

Fig. 4. Effect of the amount of KIO_3 used on the average iodine content in consumed salt products

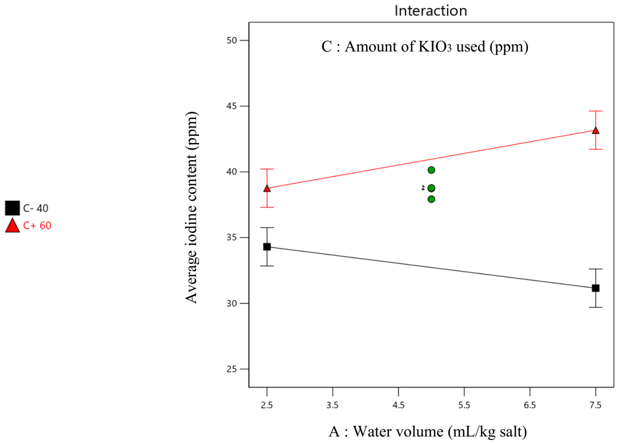

Fig. 5. Effect of interaction of water amount and KIO_3 amount on average iodine content in table salt products

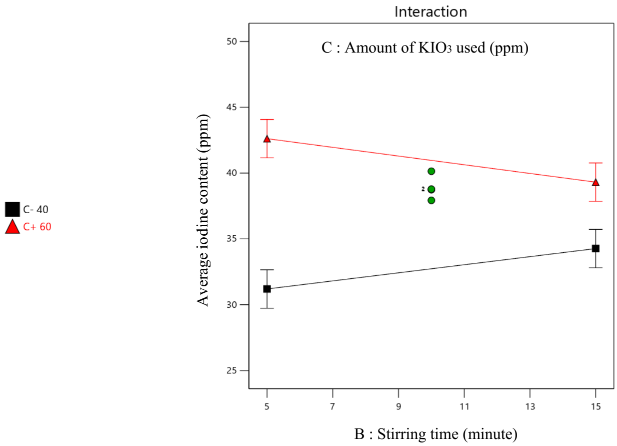

Fig. 6. Interaction effect of stirring time and amount of KIO_3 on average iodine content in table salt products

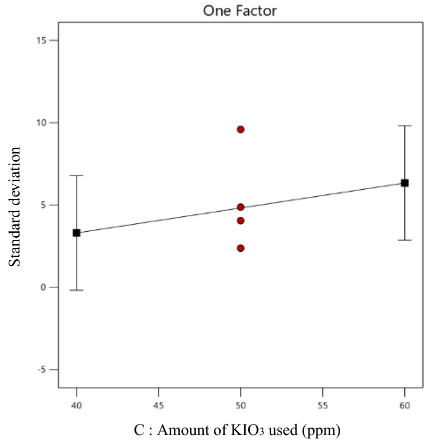

Fig. 7. Effect of the amount of KIO_3 used on standard deviation

4 Conclusions

Based on the research activities that have been carried out, several conclusions can be written as follows.

- The factor of KIO_3 use, the interaction of water amount and KIO_3 use, and the interaction of stirring time and KIO_3 use were the factors that influenced the response of average iodine content in iodized table salt products. The higher the use of KIO_3 for the iodization process, the higher the average iodine content in the consumed salt product.
- The factors of water usage and stirring time did not affect the average iodine content of iodized table salt.
- There are indications that the use of more KIO_3 will reduce the uniformity of iodine distribution in salt products. However, in the ANOVA, because the experimental error was quite large (as shown by the wide distribution of center points) the effect of this factor was not considered significant.
- The factors of stirring time and amount of water used did not affect the response of iodine distribution uniformity in salt products.

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