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Sodium Cyclamate Identification and Determination of Dawet Ice Sold in Wedi District Indonesia

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ABSTRACT

Sweeteners are food additives that are added to food or beverages to create a sweet taste, improve taste, aroma as well as a source of calories for the body. Sodium cyclamate is a sweetener food additive that is permitted to be used in food products but is often misused by food manufacturers. Excessive consumption of sodium cyclamate can cause bladder cancer, stomach pain, headache, and fever. This study aims to determine the content of sodium cyclamate in dawet ice sold in Wedi District. This study uses descriptive methods for both qualitative and quantitative. Identification of sodium cyclamate content in dawet ice sold in Wedi District using the precipitation test method, determination of sodium cyclamate content was determined quantitatively using UV-visible spectrophotometry. The results of the precipitation test showed that 5 positive samples contained artificial sodium cyclamate, with sodium cyclamate levels of 58.683 mg/L, 79.466 mg/L, 95.066 mg/L, 94.116 mg/L, and 79.5 mg/L, respectively. The conclusion is that the sodium cyclamate content in dawet ice sold in Wedi District from all samples analyzed does not exceed the maximum usage limit set by BPOM Regulation of the Republic of Indonesia No. 4 of 2014, concerning the Maximum Limit for the Use of Sweetener Food Additives, amounted to 250 mg/kg.
Keywords: sodium cyclamate, dawet ice, wedi district

INTRODUCTION

Food safety is an important requirement that must be attached to food that will be consumed by all Indonesian people. Safe and quality food can be produced from household kitchens and the food industry (Siregar et al., 2013). Food is one of the basic human needs. Therefore, the problem of food procurement from the production stage to the consumption stage must be handled from post-harvest until consumption is greatly assisted by food technology which is closely related to the properties of the food itself (Simarmata, 2018).

Today's, food products have various forms, both in terms of type and taste and processing methods. The rapid development on food processing techniques, the addition of addictive ingredients on food products is difficult to avoid. Fresh drinks which are sold on the roadside is an alternative for small and medium-sized traders in Indonesia. The drinks sold on the roadside are suspected of using synthetic sweeteners (cyclamate) to make the drinks taste. Based on instructions from the Ministry of Health of the Republic of Indonesia, the use of cyclamate is only intended for patients or for people who need

low-calorie food, but in fact, the use of cyclamate is increasingly widespread from the small and medium business sector with a variety of snacks. This is because the price is cheaper, and has a sweetness level of 30 times of sugar (Sarumaha, 2019).

Food additives in purer forms and commercially available at relatively low prices will encourage increased use of food additives, which means increasing consumption of these materials for each individual. The development of technology in the production of artificial sweeteners in processed food, both food, and beverages, is very popular with large industries and home industries because the price is cheaper, and the intensity of sweetness is higher than natural sugar(Nurjannah, 2012). The role of food additives is very important in line with advances in technology for the production of synthetic food additives. Many food additives are available in pure form which is commercially available at low prices so that they can encourage increased consumption of food additives, this has started to happen since the middle of the 20th century (Utomo et al., 2012).

The use of chemicals as one of the additives in food and beverages is currently common. Additional ingredients are ingredients that are intentionally added to food and beverages to get better quality. Additional ingredients known as addictive substances in food or beverages can be in the form of dyes, flavorings, aromas, stabilizers, antioxidants, preservatives, emulsifiers, bleaches, thickeners, and sweeteners (Handayani & Agustina, 2015). Sweeteners are food additives that are added to food or beverages to create a sweet taste, improve taste, aroma, improve physical and chemical properties as well a source of calories for the body. Sweeteners based on the source are divided into natural sweeteners and artificial (synthetic) sweeteners (Handayani & Agustina, 2015).

Cyclamate (Cyclamate) is a Food Additive (BTP) sweetener that is permitted to be used in food products with strict regulations but is often misused by food manufacturers and other communities. Cyclamate is heat resistant, so it is often used in foods that are processed at high temperatures, for example in food and beverages (Marlina, 2016). The maximum limit for the use of cyclamate in food is determined based on the food category. Dawet ice drink is included in the dessert food category with a maximum limit of 250 mg/kg (BPOM, 2014). The use of cyclamate in Indonesia is still permitted, but, the product of cyclamate metabolism, namely cyclohexamin, is a carcinogenic compound. Excretion of cyclohexamin through urine can stimulate the growth of bladder tumors in rats. Excessive consumption of cyclamate can also cause cyclamate metabolism in the stomach to produce cyclohexamin compounds which are carcinogens. This compound is capable of causing bladder cancer and is capable of causing atrophy, namely testicular shrinkage and chromosomal damage. The carcinogenic potential of cyclamate occurs when it is converted to cyclohexylamine in the digestive tract. Cyclohexylamine is toxic and is a tumor stimulant (promoter), therefore the Acceptable Daily Intake (ADI) of cyclamate must be determined (Sarumaha, 2019).

Research on the use of cyclamate in food and beverages has been widely carried out. Research conducted by (Simarmata, 2018), in the analysis of cyclamate levels in dawet ice was found to contain cyclamate sweeteners. Meanwhile, the results of research by (Misrawati et al., 2020), in the analysis of artificial sweeteners in snacks sold in traditional markets in Manado City, found cyclamate sweetener in 2 samples with the lowest level of 848.65 mg/kg and the highest level of 931.98 mg/kg. The sample exceeds the set limit.

The addition of cyclamate is usually used by beverage traders, such as dawet ice. Snack drinks such as dawet ice are small-scale industries that generally pay less attention to sanitation and food safety. This drink has an attractive appearance, delicious taste, and fresh making this product much liked by the public. In the manufacturing process, manufacturers often use artificial sweeteners that aim to replace natural sweeteners to reduce production costs (Marliza et al., 2020). Based on this description, research was conducted on the Identification and Determination of Sodium Cyclamate Levels in Dawet Ice Sold in Wedi District. This study aims to determine the content of sodium cyclamate in dawet ice sold in Wedi District.

MATERIALS AND METHODS

Tools and Materials

The materials used in this study were dawet ice samples, 10% hydrochloric acid solution, 10% barium chloride solution, 10% nitrite solution, acetic acid, ethyl acetate, 10N sodium hydroxide, cyclohexane, 30% sulfuric acid, 1% free chlorine, standard solution of sodium cyclamate, concentrated sulfuric acid, 0.5 N NaOH, aquades. The tools used in this research are analytical balance, blender, volume pipette, test tube, measuring cup, filter paper, Erlenmeyer flask, separating funnel, hotplate, water bath, and UV-Vis spectrophotometer.

Method

This research is descriptive research with a total sampling technique. There are 5 samples of dawet ice from 5 trader sold in Wedi District, Klaten, Central Java, Indonesia. Sample preparation was carried out by homogenizing the dawet ice sample, then filtering it with filter paper. The filtrate solution was then used for qualitative and quantitative analysis. Qualitative analysis of cyclamate using precipitation method ([BSN] Badan Standardisasi Nasional, 1996). Quantitative analysis of cyclamate levels using the UV-Vis spectrophotometric method (Kurnia, 2017). The data obtained from the results of research in the laboratory were analyzed descriptively in the form of tables and graphs.

RESULTS AND DISCUSSION

This study aims to determine the content of sodium cyclamate in dawet ice sold in Wedi District. Sampling was carried out based on total sampling. In this study, the precipitation test method was used for qualitative analysis and the UV-Visible spectrophotometric method for quantitative analysis.

The dawet ice sample obtained from Wedi District contained 5 samples, namely sample A, sample B, sample C, sample D, and sample E. The samples obtained were then identified qualitatively with sodium cyclamate by precipitation test. The results of qualitative analysis of sodium cyclamate content can be seen in the Table 1. Based on Table 1, it can be seen that of the 5 samples tested, all positive samples contained sodium cyclamate which was characterized by the formation of a white precipitate.

The principle of the precipitation method is that the sample which is proven to contain artificial sweetener sodium cyclamate is seen by the formation of a white crystalline precipitate from the reaction between 10% BaCl2 and 10% NaNO2 which produces a BaSO4 precipitate (Sudjadi, 2012). Qualitative testing of sodium cyclamate started with homogenized the sample between solids and liquids then filtered to get the filtrate. Sodium cyclamate reacts with hydrochloric acid to produce primary amines (cyclohexamine), sulfuric acid, and sodium chloride. The sulfuric acid formed reacts with barium chloride to form a precipitate of barium sulfate which is suspended in the mixture. The addition of

10% HCl serves to acidify the solution. The solution is made in an acidic state so that the reaction that will occur can be more easily reacted. The addition of 10% BaCl2 aims to precipitate impurities in the solution, such as the presence of carbonate ions. The addition of 10% NaNO2 serves to break the sulfate bond in the cyclamate. The heating process serves to form nitrogen gas. When the sulfate bond has been broken, the ions will react with sulfate ions and produce a precipitate of barium sulfate (BaSO4) (Rosdayani, 2018). The nitrogen gas produced from this reaction can be identified by the presence of a pungent odor during the heating process. The white precipitate cyclamate in the sample (Suliati, 2020). The 5 samples on this study indicated the positive contain of artificial sweetener sodium cyclamate.

These positive samples, then analyzed quantitatively using UV-Visible spectrophotometry. Quantitative analysis was carried out on positive samples to determine the amount of cyclamate sweetener content in the sample. Quantitative analysis was performed using the UV-Visible spectrophotometric method. This method is used because of the presence of a chromophore group in the chemical structure so that it can be detected by a UV-Visible detector. Solvents and chemical molecular structures containing chromophore groups are the ones that affect the maximum wavelength (Susanti, 2013).

The preparation of standard solutions is the first step in determining the levels of cyclamate sweeteners. A standard solution is a solution that contains a precisely known concentration. Determination of the maximum wavelength using a standard solution with a concentration of 1800 ppm and measured at a wavelength of 200-400 nm. The determination of the maximum wavelength aims to determine the maximum absorption area that can be produced in the form of the absorbance value of a test solution. The maximum wavelength of sodium cyclamate solution is obtained at a wavelength of 252 nm by giving an absorption or absorbance of 0.672 and is still in the range of the optimum absorption area of sodium cyclamate, which is 200-400 nm so that it can be said that the measurement results meet the requirements for use for analysis. The absorbance value of the cyclamate standard solution quantitatively can be seen in the Table 2.

The calibration curve was obtained by measuring the absorbance of standard solutions with concentrations of 1000, 1200, 1400, 1600, 1800 ppm at a wavelength of 252 nm. From this measurement, the regression equation is obtained, namely $y = 0.0006x – 0.3602$ with an R^2 value of 0.9966. The R^2 value indicates that there is a very strong correlation between concentration and absorbance (Hidayat, 2019).

The absorbance value of the sodium cyclamate standard solution above can be used to determine the linearity test with the correlation (r) in the linear regression equation $y = ax + b$. The graph of the calibration curve and the linear regression equation can be seen in Figure 1.

Determination of sodium cyclamate levels is done by first homogenizing the sample between the solid and the liquid. Sodium cyclamate is reacted with H_2SO_4 to convert cyclamate into cyclamic acid, then the cyclamic acid solution is reacted with ethyl acetate to form cyclamic acid in the organic phase and there are two colorless layers. Extraction of cyclamic acid with distilled water aims to bind the cyclamate compound in the sample thoroughly so that it is separated from other sample components. The addition of NaOH solution serves to provide an alkaline atmosphere while the addition of cyclohexane functions as a cyclamate extractor. The extract from the cyclamate was reacted again with H_2SO_4, cyclohexane, and Na-hypochlorite to form

two layers. The cyclohexane layer was taken and washed with NaOH to form a colorless solution. In this cyclohexane layer, the cyclamate has been extracted in it, then diluted with distilled water, and then the absorbance was measured using a UV-Visible spectrophotometer (Padmaningrum & Marwati, 2015).

The absorbance obtained from each sample is then entered into the linear regression equation (y = 0.0006x – 0.3602) to obtain the sample concentration (x). The average levels of sodium cyclamate in the samples were 58.683 mg/L, 79.466 mg/L, 95.066 mg/L, 94.116 mg/mL and 79.5 mg/L.

Based on the quantitative tests carried out and shown in Tabel 3, the sodium cyclamate levels in sample A are 58.683 mg/L, sample B is 79.466 mg/L, sample C is 95.066 mg/L, sample D is 94.116 mg/L and sample E is 79,5 mg/L. These data indicate that the levels of the dawet ice samples tested have not exceeded the maximum usage limit set by the BPOM Regulation of the Republic of Indonesia Number 4 of 2014 concerning the maximum limit for the use of sweetener food additives, which is 250 mg/kg. The low levels obtained are still safe for consumption but the use of artificial sweeteners is more specifically for people such as diabetics whose aim is to control excess sugar levels or obese people, but also must be within certain limits and must be supervised by a doctor or health expert (Musiam, 2016).

The results of this study showed lower levels than the results of previous studies. Research by Misrawati et al. (2019), it is known that the results of the cyclamate test on mixed ice snacks circulating in elementary schools in Kendari City, 4 positive samples contained cyclamate artificial sweeteners with the highest cyclamate content of 300 mg/kg. According to (Hadju et al., 2013), showed that the results of testing cyclamate on snack drinks in the traditional market of Manado city that two positive samples contained cyclamate sweeteners with cyclamate levels of 931.98 mg/kg and 848.65 mg/kg. These levels indicate that the sample has exceeded the maximum limit for the use of sodium cyclamate that has been set, which is 250 mg/kg.

Based on the results of the study, consuming sodium cyclamate above the specified limits can trigger health problems, including tremors (neural diseases), migraines, headaches, memory loss, confusion, insomnia, irritation, asthma, hypertension, diarrhea, stomach pain, allergies. , impotence, baldness, and brain cancer (Hadju et al., 2013). Other problems related to sodium cyclamate consumption are cardiovascular and nervous system problems, decreased growth rate, bladder cancer, thyroid adenoma, abnormalities in red blood cells, leukocytes, monolayers, spinal cord, and bacterial infections (Hidayat, 2019). Therefore, it is recommended for the community to consume beverages, especially dawet ice, in moderation.

CONCLUSION

This study concluded that the 5 samples of dawet ice drinks sold in Wedi District are contain the artificial sweetener sodium cyclamate. The content of sodium cyclamate in the sample is under the maximum limit of the regulation which is set by BPOM of the Republic of Indonesia.

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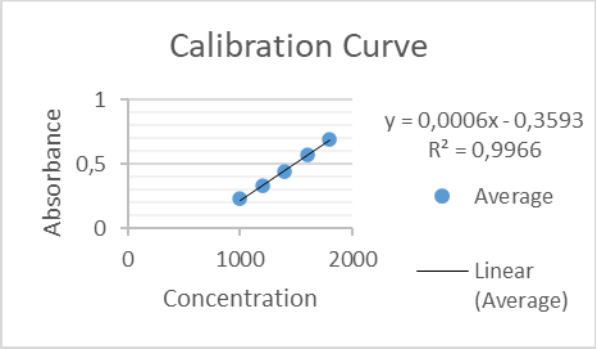

Figure 1. Sodium Cyclamate Standard Solution Calibration Curve

Table 1. Results of Qualitative Analysis of Sodium Cyclamate Precipitation Method

No	Sample	Observation Result	Interpretation
1.	A	Presence of white precipitate	Positive (+)
2.	B	Presence of white precipitate	Positive (+)
3.	C	Presence of white precipitate	Positive (+)
4.	D	Presence of white precipitate	Positive (+)
5.	E	Presence of white precipitate	Positive (+)

Table 2. Absorbance Value of Standard Solution of Sodium Cyclamate at Maximum Wavelength of 252 nm

Concentration (ppm)	Absorbance
1000	0,233
1200	0,331
1400	0,439
1600	0,570
1800	0,694

Table 3. Sodium Cyclamate Levels in Dawet Ice based on UV-Visible Spectrophotometer

No	Sample	Average Absorbance	Average rate ± SD (mg/L)
1.	A	0,344	58,683 ± 0,0577
2.	B	0,593	79,466 ± 0,2753
3.	C	0,780	95,066 ± 0,0577
4.	D	0,769	94,116 ± 0,0288
5.	E	0,594	79,5 ± 0,4582