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Research Article

Analysis of Artificial Sweeteners, Sodium Cyclamate, in Packaged Beverages and Powders by the Gravimetric Method

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ABSTRACT: Most people like instant beverage products because of their practical processing and superior taste and creative packaging, thus attracting consumers to consume them. One of the food additives that is often used in instant beverage products is artificial sweeteners such as sodium cyclamate which has a sweetness level of 3.94 kcal/g or 30 times that of sucrose. This study aims to determine the levels of sodium cyclamate in packaged and powdered drinks circulating on the campus of the Indonesian Muslim University using the gravimetric method. The sampling method used a purposive technique and obtained 6 test samples, qualitative analysis used the precipitation method and obtained all positive samples containing sodium cyclamate. The results of the quantitative analysis obtained the levels of sodium cyclamate for each sample A; B; C; D; E; F as much as 37.07 mg/kg; 30.17 mg/kg; 12.93 mg/kg; 4.31 mg/kg; 340 mg/kg; 258 mg/kg. Thus, these results indicate that all samples do not exceed the levels set by BPOM regulation No. 11 of 2019 concerning food additives, namely 350 mg/kg, so they are safe for consumption.

KEYWORDS: Instant drinks; Packaged drinks; Powdered drinks; Sodium cyclamate; Gravimetry.

1. INTRODUCTION

The emergence of new instant products is a result of the growth of an increasingly modern era. There are many packaged beverage products available in various shapes, types and flavors. Because of its practical packaging and sweet taste, and easy to get it because of its wide availability, it has become one of the favorite drinks among the people (Hana, 2022). In general, food additives are substances that are intentionally added to food processing to produce certain functional properties in food, such as flavor enhancers, bread thickeners, food thickeners, and food preservation (Sujarwo et al., 2020).

Sweeteners are substances added to food and drinks to give them a sweet taste or to help people better tolerate the sweet taste in those foods and drinks. Sweeteners have various purposes, such as improving taste and aroma, improving physical characteristics, functioning as preservatives, increasing chemical characteristics, and providing calories for the body. Natural and synthetic sweeteners are two categories of sweeteners (Gift, 2013). Sweetener classification is divided into two, namely, natural sweeteners and artificial or synthetic sweeteners. Natural sweeteners are produced from plants. Examples of natural sweeteners are sorbitol, mannitol, isomalt, and others (Hadiana, 2018). Meanwhile, artificial sweeteners (synthetic) are food additives with a sweet taste but have no nutritional value. Cyclamate, synthetic sorbitol, aspartame, saccharin, dulsin, and nitropropoxy-aniline are examples of artificial sweeteners. Many factors are taken into consideration in determining the types of artificial sweeteners that are allowed in food products, including caloric value, level of sweetness, toxicity, and their effects on the human body's metabolism (Utomo et al., 2012). The use of artificial or synthetic sweeteners is widely used by traders because it has a relatively cheap price compared to sugar (Tahir and Vitrianty, 2013).

Sodium cyclamate ($C_6H_{12}NNaO_3S$) is a type of artificial sweetener with a sweetness level of 3.94 kcal/g which is approximately 30 times that of sucrose (Melinda et al., 2022). Sodium cyclamate is an artificial or synthetic sweetener that was originally developed to increase the availability of food and beverage products for diabetic patients. In addition, sodium cyclamate has low calories (Dewi and Woelansari, 2018). The World Health Organization (WHO) states that there is a maximum limit for sodium cyclamate that can be consumed per day or Acceptable Daily Intake (ADI), which is 11 mg/kg body weight. According to Permenkes No. 722/MENKES/PER/IX/1988 regarding BTP it is explained that the maximum limit for artificial sweetener sodium cyclamate is 3 g/kg BW. And according to BPOM No. 11 of 2019, cyclamate artificial sweetener is 350 mg (Al-Muqsith and Nadira, 2021).

Excessive use of sodium cyclamate can have negative effects on health. Sodium cyclamate can synthesize cyclohexylamine compounds as a by-product of metabolism in the body. Cyclohexylamine is a carcinogenic and free radical promoter, so excessive use of cyclamate can be harmful to human health. Urinary excretion of cyclamate has been associated with chromosomal and testicular wasting and tumor stimulation (Manoppo et al., 2019).

2. EXPERIMENTAL SECTION

2.1. Population and Sample

The sample population in this study were bottled and powdered drinks, each of which contains sodium cyclamate circulating around the campus of the Universitas Muslim Indonesia, including 2 samples of packaged drinks and 4 samples of powdered drinks. This sampling was carried out using a purposive technique, ie without comparing with other samples.

2.2. Materials and tools

The materials used were 2 packaged beverage products and 4 powdered beverage products, 10% hydrochloric acid (HCl), 10% Barium chloride ($BaCl_2$), 10% Sodium nitrite ($NaNO_2$), and distilled water. While the tools used in this study were a spatula, stirring rod, Buchner funnel, beaker glass (Pyrex®), volumetric flask (Iwaki®), glass funnel (Pyrex®), timer, filter paper, Whatman filter paper no.42, dropper pipette, measuring pipette (Pyrex®), volume pipette (Pyrex®), suction cup/rubber, hot plate (Cimarec® 2), desiccator (Pyrex®), oven (Memmert®), analytical balance (Kern®).

2.3. Qualitative analysis

Prepare Whatman 42 paper which has been heated in the oven for 15-20 minutes at a temperature of 100-105^0 C to get an empty filter paper weight. Then the filter paper is cooled using a desiccator. After that the filter paper was weighed using an analytical balance. Heating was carried out repeatedly so that a constant filter paper weight was obtained.

A total of 10 g of powder drink sample was weighed carefully, then added distilled water up to 100 mL. Meanwhile, in bottled drinks, pipette as much as 100 mL. Then as much as 10 mL of concentrated HCl, 10 mL of 10% $BaCl_2$ solution was added to the sample and left for 30 minutes. After that the sample is seen whether there is sediment or not, if sediment occurs then the sample is filtered with filter paper and the filter paper is washed with distilled water.

The filtrate obtained was added to 10 mL of 10% $NaNO_2$ solution and homogenized. Then the filtrate is heated over a water bath or hotplate for 15-20 minutes while stirring occasionally and left for a day. If there is a precipitate, the cyclamate content in the sample studied is positive for sodium cyclamate (Luviriani, 2020).

2.4. Quantitative analysis

Samples that showed positive results for the presence of cyclamate were filtered using Whatman filter paper no.42. Then dried in the oven at 105 °C for 20 minutes. After that, cool using a desiccator for 10 minutes. Then the filter paper is carefully weighed together with the precipitate with an analytical balance. Repeat the process of heating and weighing the filter paper until a constant weight is obtained (Luviriani, 2020).

2.6. Data analysis

Data analysis was carried out by calculating the levels of sodium cyclamate contained in packaged drinks and powders, namely:

For Packaged Drinks:

$$\text{Cyclamate Sodium Content} = \frac{(b-a)}{Sample\ Volume} \times 0{,}862 \times 100\%$$

Information:

a = mass of empty filter paper

b = mass of filter paper + precipitate

For Powder Drinks:

$$\text{Cyclamate content} = \frac{BM\ Natrium\ siklamat}{weight\ BaSO4} \times \text{sediment}$$

3. RESULTS AND DISCUSSION

This research was conducted by analyzing the content of the artificial sweetener sodium cyclamate in packaged and powdered drinks circulating around the Indonesian Muslim University campus, the choice of this location was due to the high consumption pattern of instant drinks such as packaged drinks or powdered drinks among Universitas Muslim Indonesian students. Where this study aims to determine the levels of sodium cyclamate artificial sweetener using the gravimetric method and adjust these levels to BPOM RI regulations no.11 of 2019 (BPOM, 2019).

The gravimetric method is a fixed weight based approach for quantitative analysis (constant weight) (Marlina, 2016). The gravimetric method is one of the most common methods used to determine sodium cyclamate levels in a drink or food with considerations of more economical costs and easier processing.

The drink samples selected in this study were 4 samples of powdered drinks and 2 samples of packaged drinks circulating around the campus area of the Indonesian Muslim University in Makassar city. Sampling using a purposive technique, i.e., without comparing with other samples. Purposive sampling technique is a technique of determining the sample with certain considerations. The reason for using this technique is suitable for quantitative research or research that does not generalize (Sugiono, 2016).

The qualitative analysis was carried out by adding several reagents, the reagents used were 10% HCl solution, 10% $BaCl_2$ solution, 10% $NaNO_2$ solution. The six samples that tested positive for containing sodium cyclamate are shown in **Table 1.**

Table 1. Results of Qualitative Analysis of Cyclamate Artificial Sweeteners in Powder Drinks and Packaging

Samples	Powder drink name and packaging	Test results
Powder drink (vanilla blue flavor)	A	(+)
Powder drink (cappuccino flavor)	B	(+)
Powder drink (apple flavor)	C	(+)
Powder drink (mango flavor)	D	(+)
Packaged drink (orange flavor)	E	(+)
Packaged drinks (grape flavor)	F	(+)

The addition of concentrated HCl aims to make the sample solution acidic and easily react with $BaCl_2$, HCl will cause the sulfate bond to break in sodium cyclamate, the H atom that binds to HCl will bind to the N atom which forms a primary aliphatic amine bond and sulfuric acid (Fawwaz and Baits, 2016). While the addition of reagent $BaCl_2$ results in a reaction between BaCl and H_2SO_4, Ba^{2+} ions react with SO_4^{2-} ions which will form $BaSO_4$ (Zarwinda et al., 2021). The addition of $NaNO_2$ reagent and heating to break the sulfate bonds in primary aliphatic amine bonds, in the heating process will trigger a characteristic odor. When the sulfate bond is broken, there will be a reaction between Ba^{2+} ions and sulfate ions which then produces a precipitate of barium sulfate ($BaSO_4$) (Zarwinda et al., 2021). The reaction equation for the formation of a precipitate (sodium cyclamate) can be seen in **Figure 1.**

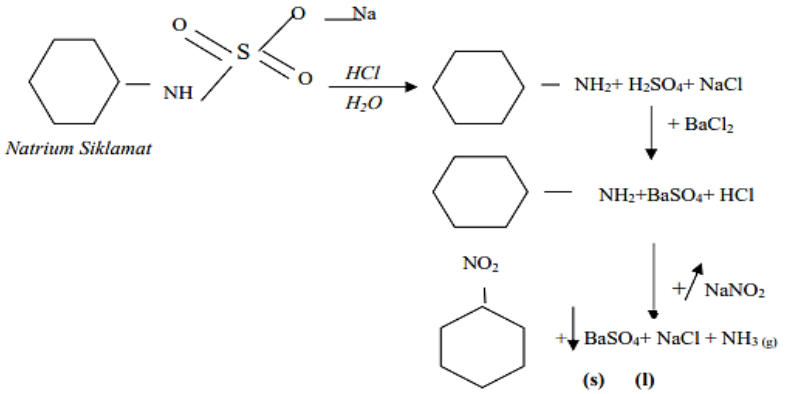

Figure 1. Reaction Equation for the Formation of Barium Sulfate Precipitate

Quantitative analysis was carried out to determine the content of sodium cyclamate artificial sweetener using the gravimetric method. The gravimetric method is carried out by finding the constant weight of a compound (constant weight) (Marlina, 2016). The results of the qualitative and quantitative analysis of the artificial sweetener sodium cyclamate in packaged and powdered drinks can be seen in **Table 2**. In the quantitative analysis the samples that had been heated were then allowed to stand for 24 hours with the aim of causing a reaction between Ba^{2+} ions and sulfate ions which then produced a precipitate of barium sulfate ($BaSO_4$). After the precipitate formed, the sample solution was filtered using Whatman filter paper no.42 with a diameter of 125 mm, previously the filter paper had a constant weight. Furthermore, the precipitate that has been filtered indicates the presence of artificial sweetener sodium cyclamate (Luviriani, 2020). Sodium cyclamate (precipitate) is then heated in an oven at 100 °C for 20 minutes. After 20 minutes, the precipitate was cooled again using a desiccator for 10 minutes and weighed using an analytical balance. This process was replicated several times to obtain a constant weight from filter paper + precipitate (sodium cyclamate) (Luviriani, 2020). The constant weight according to the Indonesian Pharmacopeia VI edition 2020 is the weight on weighing after the substance has been dried for one hour until the difference between two consecutive weighing is not more than 0.25%.

Table 2. Qualitative and Quantitative Analysis of Sodium Cyclamate Artificial Sweetener in Packaged and Powdered Beverages

Sample code	Precipitation reaction	Cyclamate grade
A	(+)	37.07 mg/kg
B	(+)	30.17 mg/kg
C	(+)	12.93 mg/kg
D	(+)	4.31 mg/kg
E	(+)	340 mg/kg
F	(+)	258 mg/kg

Based on BPOM RI regulation No. 11 of 2019 concerning Food Additives (BTP), the maximum limit for using artificial sweetener sodium cyclamate for soft drinks is 350 mg/kg. The results of a quantitative analysis of sodium cyclamate levels in packaged and powdered drinks circulating on the campus of the Indonesian Muslim University

obtained the results, namely the sodium cyclamate levels of each sample A; B; C; D; E; F as much as 37.07 mg/kg; 30.17 mg/kg; 12.93 mg/kg; 4.31 mg/kg; 340 mg/kg; 258 mg/kg. Thus, the results of the levels of artificial sweetener sodium cyclamate found in packaged drinks and powders circulating around the Universitas Muslim Indonesia campus are all in accordance with the regulations set by BPOM RI No. 11 of 2019.

4. CONCLUSION

From the results of this study, it can be concluded that the levels of artificial sweetener sodium cyclamate in packaged drinks and powders circulating around the Indonesian Muslim University campus, each sample A; B; C; D; E; F as much as 37.066 mg/kg; 30.17 mg/kg; 12.93 mg/kg; 4.31mg/kg; 340mg/kg; 258mg/kg. Therefore, all of these samples were below the maximum limit for artificial sweetener sodium cyclamate stipulated by BPOM RI No. 11 of 2019, which is 350 mg/kg, thus they are safe for consumption.

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