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Article Review: Analysis Of Artificial Sweetness In Food And Beverages

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ABSTRACT

Artificial sweeteners are substances added to food and beverages with the aim of giving a sweet taste, having no nutritional value. The types of artificial sweeteners allowed by the Minister of Health are Cyclamate, Saccharin, Aspartame and Sorbitol. The method used in this paper is a literature review study. The total journals used in this article review are 14 journals. In several studies, the levels of artificial sweeteners contained in related foods are still within the limits permitted by the government, but in some studies there are still foods whose levels of artificial sweeteners exceed the permitted limits.

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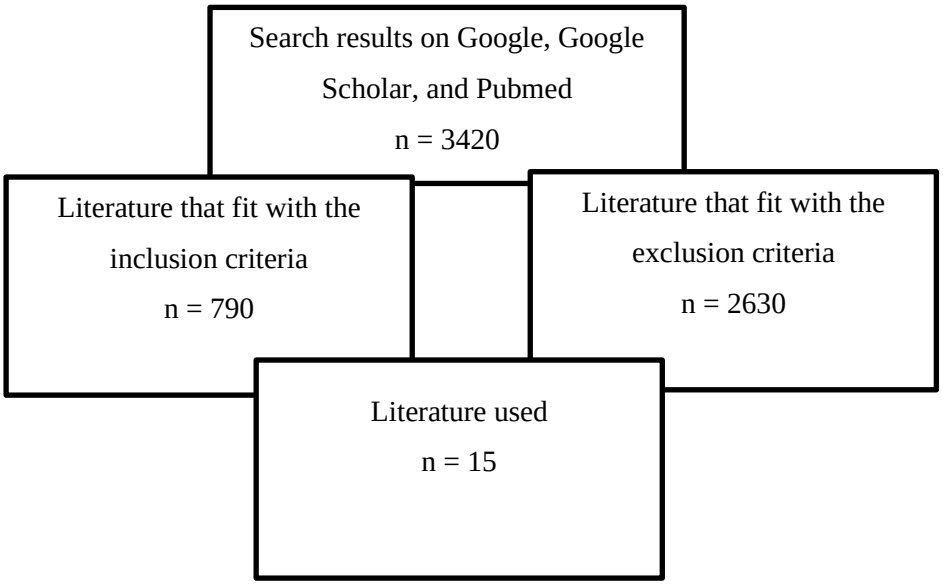
1. INTRODUCTION

Based on the Regulation of the Minister of Health of the Republic of Indonesia No. 722/Menkes/Per/88, Artificial sweetener is a substance added to food and beverages with the aim of giving a sweet taste, has no nutritional value. Examples of artificial sweeteners are saccharin, dulsin, aspartame, synthetic sorbitol. Often artificial sweeteners are added to foods and beverages because they are less expensive. This is often found in manufacturers of snacks and fast food drinks. Along with the development of the era, human needs for food are more and more diverse. Ready-to-eat products in the form of food and beverages are widely available in attractive shapes and flavors. One example of ready-to-eat products that are widely circulated is soft drinks that mix artificial sweeteners. Artificial sweeteners are used as sweeteners made from synthetic chemicals because they are relatively cheap, and the sweetness level of synthetic sweeteners is much higher than natural sweeteners. The types of artificial sweeteners allowed by the Minister of Health are Cyclamate, Saccharin, Aspartame and Sorbitol.

2. METHOD

The method used in this paper is a literature review study. Data search was carried out using search engines Google, Google Scholar, and Pubmed with the keywords "analysis of artificial sweeteners in food and beverages", "saccharin, cyclamate, aspartame in food and drink", "saccharine, siclamate, aspartame in food and drink" . The total journals used in this article review are 14 journals. The inclusion criteria for all literature used in writing this review article are not more than 10 years or the range from 2012 to 2022. The literature exclusion criteria include not originating from theses, theses or dissertations. The review of this study is to determine the method of analysis of artificial sweeteners in food and beverages along with the results compared with the permissible usage threshold. The literature search was carried out with due regard to the inclusion and exclusion data criteria.



3. RESULTS AND DISCUSSION

Table 1. Article Review

References	Name Artificial Sweeteners	Main Findings
(Ramadhani et al., 2018)	Sodium Cyclamate	In the qualitative analysis of the 100 ml sample using a solution of 10 ml of 10% HCl, 10 ml of 10% BaCl2, and 10 ml of 10% NaNo2, it was found that all three samples were positive for cyclamate. The quantitative analysis using spectrophotometry showed that sample A contained 0.4585 g/kg cyclamate, sample B contained 0.8065 g/kg cyclamate, sample C contained 0.3136 g/kg cyclamate. The results obtained are in accordance with the maximum level of sodium cyclamate use.
(Noriko et al., 2011)	Cyclamate and Saccharine	In the qualitative test of artificial sweetener saccharin, 10 grams of 8 types of samples were added with 5 ml of 25% HCl, 25 ml of ether, 15 drops of H2SO4, and resorcinol crystal. The results obtained were negative liquid sugar containing saccharin, positive fruit sugar containing saccharin, negative Orson orange ice containing saccharin, positive purple Orson ice containing saccharin, negative chewing gum containing saccharin, positive ice shake containing saccharin, young coconut ice positive containing saccharin, and negative sugar cane contains saccharin. In the qualitative test of artificial sweetener cyclamate as much as 10 grams of 8 types of samples added 20 ml of distilled water, activated charcoal, filtered. The filter results were added 10 ml of 10% HCl, 10 ml of 10% BaCl2, and 10 ml of 10% NaNo2. The results obtained were negative liquid sugar containing cyclamate, fruit sugar positive containing cyclamate, negative orange Orson ice containing cyclamate, positive purple Orson ice containing cyclamate, negative chewing gum containing cyclamate, positive ice shake containing cyclamate, young coconut ice positive containing cyclamate, and negative sugarcane contains cyclamate.
(Karolina & Rosmiati, 2018)	Saccharine	Saccharin qualitative test by taking 50 mL of sample, added 10% HCl, extracted with 25 mL of ether, the ether layer was evaporated in open air. Then 10 drops of concentrated H2SO4 and 40 mg of resorcinol were added, heated slowly until it turned a cloudy green color. The test tube was cooled and then added with 1 mL

		of distilled water and 1-5 drops of 10% NaOH solution. If it turns into a green fluorescence, it indicates the presence of saccharin. The results obtained in samples A, B, C, D, and E were declared positive because a green fluorescence color was formed. Saccharin quantitative test using alkalimetric titration method. 25 mL of the sample was put into a separating funnel, added 1 mL of HCl, extracted 5 times using a mixture of chloroform and 95% ethanol in a ratio of 9:1, the extract was filtered using filter paper. The filtrate is then evaporated. The residue was dissolved in 70 ml of hot water then cooled and titrated with standardized NaOH using 1% phenolphthalein indicator until the color changed to pink. Results obtained. Sample A 250 mg/kg material, sample B 160 mg/kg material sample C 160 mg/kg material, sample D 250 mg/kg material, and sample E 580 mg/kg material. Samples A, B, C, and D were still within the allowable level of saccharin, which was 300 mg/kg of material, while sample E exceeded the limit.
(Handayani & Agustina, 2015)	Sodium Cyclamate	In the qualitative testing, the step is 100 ml of sample solution added 2 grams of BaCl2, precipitated. The filter results were added with 10 ml of HCl and 10 ml of 10% NaNO2, heated. The presence of a white precipitate indicates the presence of cyclamate. Quantitative testing of sodium cyclamate used the alkalimetric method with 3 repetitions. The levels of sodium cyclamate obtained include sample 1 containing 0.4029, sample 2 containing 0.3425, sample 3 containing 0.0514, sample 4 containing 0.2529, sample 5 containing 0.3492, sample 5 containing 0.3492, sample 6 contains 0.4096, and sample 7 contains 0.3268.
(Dali et al., 2013)	Aspartame	In this study, 7 samples were tested quantitatively by the HPLC method. The mobile phase used is sodium dihydrogen phosphate buffer (pH 2.6) and acetonitrile (82.5 : 17.5) with a flow rate of 1.2 ml//min, UV detector with = 210 nm. The stationary phase used was octadecylsilane (ODS or C18) with 5 m particles. The results obtained sample A 7.5658 mg/kg, sample B 198.3445 mg/kg, sample C 258.2797 mg/kg, sample D 226.5515 mg/kg, sample E did not contain aspartame, sample F 45.5389 mg/kg, sample G 140.37488 mg/kg. This indicates that the aspartame content contained in all samples is still below the established standard, which is a maximum of 600 mg/kgBB
(Tahir & Vitrianty, 2013)	Saccharine and cyclamate	Saccharin qualitative test was carried out with resorcinol test. The sample was added with 10 drops of 10% HCl, and extracted with 25 ml of ether 3 times. The ether extract was collected and evaporated in a test tube in open air to dry. Add 10 drops of concentrated H2SO4 and 40 mg of resorcinol then slowly heated until a dirty green color, cooled and added 10 ml of distilled water and 10% NaOH. If a fluorescent green color occurs, it means that the saccharin is positive in the sample. Qualitative Analysis of Cyclamate with precipitation test Added 10 ml of 10% HCl solution into the sample filter results and then added 10 ml of 10% BaCl2 solution. Leave for 30 minutes and then filter again with Whatman 42 filter paper, then add 10 ml of 10% NaNO2. Heated on a water bath for 30 minutes. If a white precipitate appears from BaSO4, it means that the sample contains cyclamate. The results of the qualitative analysis on samples A, B, C, and D were all positive for saccharin and cyclamate. Quantitative Analysis of Saccharin with UV-Visible Spectrophotometry. The results of the saccharin content obtained included sample A 1021.1 mg/kg, sample B 4829 mg/kg, sample C 1761.91 mg/kg, and sample D 629.59 mg/kg. Quantitative Analysis of Cyclamate Using Gravimetric Method by weighing 15 grams, adding aquadest up to 100 ml. Then added 10 ml of concentrated HCl and 10 ml of 10% BaCl2. If there is sediment, filter and then wash the filter paper

		with water. The filtrate was added with 10 ml of 10% NaNO2 solution and then stirred. Heated for 2 hours. Filter the sediment then transfer the sediment along with filter paper into a crucible that has been weighed. Incandescent to constant weight, cooled in a desiccator and then weighed. The results of the cyclamate levels obtained include sample A 2.71 gram/kg, sample B 2.88 gram/kg, sample C 1.51 gram/kg, and sample D 2.37 gram/kg. The average level of saccharin in passion fruit juice exceeds the limit set by the Minister of Health and cyclamate meets the requirements allowed by the Minister of Health.
(Hernaningsih & Jayadi, 2021)	Cyclamate	Quantitative testing of the cyclamate content in the three syrup samples used the UV-Vis spectrophotometer method. Sample 1 contains 238.78 mg/kg, sample 2 contains 239.65 mg/kg, and sample 3 contains 241.39 mg/kg. All samples did not exceed the standard limit set by BPOM RI Number 4 of 2014 where the maximum level of cyclamate was 250 mg/kg so that all samples were safe for consumption.
(Novitasari et al., 2019)	Natrium Siklamat	Qualitative testing was carried out by dissolving the sample with distilled water up to 100 ml, adding BaCl2, adding 10 ml HCl and 10 ml NaNO2 10%, then heated to boiling and then allowed to stand until a precipitate showed the presence of cyclamate. The results obtained in sample A, sample B, and sample C were in the three positive samples containing sodium cyclamate. Quantitative testing of samples A, B and C was carried out by dissolving 8 grams of sample with distilled water up to 100 ml, then 25 ml of sample was taken, then 2-3 drops of Phenolphthalein indicator were added, titrated with 0.1 N NaOH. Cyclamate content of sample A 0.58% or 0.0058 grams, sample B 0.52% or 0.0052 grams and Sample C 1.03% or 0.0103 grams. The maximum level of use of cyclamate according to the regulations of the Minister of Health is 3 g/kg of material weight. The three samples still meet the requirements for the permissible levels of sodium cyclamate
(Umirestu et al., 2019)	Saccharine and Cyclamate	The results of the saccharin test on 5 cappuccino powders that have been mixed with sugar and 5 cappuccino powders that are not mixed with sugar, it was found that the mixed cappucino sample 1 was positive for saccharin with a concentration of 1096.30 mg/kg, the mixed cappucino sample 2 was positive for saccharin with a concentration of 731.15 mg. /kg, mixed cappucino sample 3 was positive for saccharin with a concentration of 559.37 mg/kg, sampled cappucino mixed 4 was positive for saccharin with a concentration of 408.83 mg/kg, sampled cappucino mixed 5 was positive for saccharin with a concentration of 1154.95 mg/kg , the cappucino 6 sample was positive for saccharin with a level of 222.42 mg/kg, the cappucino 7 sample was positive for saccharin with a level of 386.16 mg/kg, the cappucino 8 sample was positive for saccharin with a level of 229.52 mg/kg, the cappucino 9 sample was positive containing saccharin with a level of 523.73 mg/kg, the sample of Cappucino 10 was positive for saccharin with a concentration of 515.97 mg/kg. The results of testing the sodium cyclamate content include the mixed 1 cappucino sample negative because there was no sediment, the mixed cappucino sample 2 was negative, the cappucino sample mixed 3 was negative, the cappucino sample mixed 4 was negative, the cappucino sample mixed 5 was negative, the cappucino sample 6 was positive containing cyclamate with a concentration of 208 ,11 mg/kg, cappucino 7 samples were negative, cappucino 8 samples were positive for cyclamate with a concentration of 203.34 mg/kg, cappucino 9 samples were positive for cyclamate with levels 1219.44 mg/kg, cappucino 10 samples were positive for cyclamate with a concentration of 361 ,62 mg/kg.

(Wandira & Rahayu Ilyas, 2018)	Saccharine	The results of the qualitative test on the three syrup samples included sample A, sample B, and sample C that were positive for saccharin. The results of quantitative testing using the titration method obtained saccharin levels in sample A 4.14%, sample B 1.31%, and sample C 1.20%.
(Luviriani & Sari, 2020)	Cyclamate	The results of qualitative testing of the cyclamate content in unbranded milk powder using the precipitation method, namely sample 1, sample 2, sample 3, sample 4, sample 5, and sample 6 were all positive for cyclamate. The quantitative test was carried out by the gravimetric method by weighing the weight of an empty cold incandescent crucible which had previously been heated at 700°C for 1 hour and the weight of an incandescent cup containing precipitated ash after the ashing process at 700°C for 1 hour. The results of the cyclamate levels obtained in sample 1 are 4,500 mg/kg, sample 2 contains 8,800 mg/kg, sample 3 contains 14,300 mg/kg, sample 4 contains 14,600 mg/kg, sample 5 contains 17,200 mg/kg, and sample 6 contains 25,600 mg/kg. All samples containing cyclamate exceeds the limit allowed by BPOM RI number 4 of 2014 where the maximum limit for the use of cyclamate is 250 mg/kg.
(Devitria & Sepriyani, 2018)	Cyclamate	The results of the estimation test with the precipitation reaction for the presence of cyclamate content in the five syrups obtained results in sample A and sample C were negative because there was no precipitate. Sample B, sample D, and sample E were positive because there was a white precipitate. Furthermore, an affirmation test was performed using Thin Layer Chromatography and the average Rf value in sample B was 0.45, in sample D the average Rf value was 0.44, and in sample E the average was 0.56. So that it can be stated that samples B, D, and E are positive for cyclamate.
(Melinda et al., 2022)	Cyclamate	The results of observations of sweeteners in making iced tea at traders showed that samples A, D, and E contained a natural sweetener of granulated sugar. Samples B and C are instant drink powders containing artificial sweeteners. This is in accordance with the results of the qualitative test of precipitation in which samples A, D, and E were negative because there was no precipitation. Samples B and C were positive for sedimentation.
(Hakiki et al., 2016)	Cyclamate	Qualitative testing for the presence of cyclamate on tofu gejrot in the seven samples using the precipitation method showed that all of them were positive for cyclamate due to the formation of a white precipitate. The results of quantitative testing of cyclamate levels in gejrot tofu showed that sample 1 contained 0.24608 g/kg, sample 2 contained 0.1328 g/kg, sample 3 contained 0.2966 g/kg, sample 4 contained 0.26052 g/kg , sample 5 contains 0.28752 g/kg, sample 6 contains 0.225924 g/kg, sample 7 contains 0.291 g/kg.
(Ranny et al., 2017)	Cyclamate	Qualitative testing using the precipitation method showed that samples A, B, C, D, E, F, and G were all positive because of the formation of a white precipitate. Quantitative testing using the gravimetric method showed that sample A1 contains 26 mg/kg, A2 contains 30 mg/kg, A3 contains 22 mg/kg, B1 contains 50 mg/kg, B2 contains 48 mg/kg, B3 contains 44 mg/kg, C1 contains 28 mg/kg, C2 contains 22 mg/kg, C3 contains 26 mg/kg, D1 contains 18 mg/kg, D2 contains 16 mg/kg, D3 contains 24 mg/kg, E1 contains 28 mg/kg, E2 contains 14 mg/kg, E3 contains 20 mg/kg, F1 contains 32 mg/kg, F2 contains 24 mg/kg, F3 contains 26 mg/kg, G1 contains 70 mg/kg, G2 contains 68 mg/kg, G3 contains 66 mg/kg. All samples met the minimum requirements for cyclamate levels issued by BPOM.

Qualitative test of precipitation reaction

Sodium cyclamate (C6H12NNaO3S) is about 30x sweeter than sucrose which has a sweetness level of 3.94 kcal/g. The limit for daily consumption of sodium cyclamate according to WHO (World Health Organization) is 11 mg/kgBW, while according to Permenkes 772/Menkes/Per/IX/88 it is 3 g/kgBW. Basically sodium cyclamate is not dangerous because it is often used for low-calorie diets in diabetics. However, the use of sodium cyclamate in excess of the limit causes adverse effects such as tremors, insomnia, migraines, memory disorders, headaches, allergies, asthma, diarrhea, high blood pressure, sexual disorders, baldness, tumors, disorders of brain and nerve development in children. . The maximum level of use of cyclamate for food and beverage types is 3 g/kg of material weight. From the above provisions, it can be concluded that the threshold price for cyclamate is 3 g in 1 kg of drink (3,000 ppm), so in 1 g of instant powdered drink the threshold price for the use of cyclamate is 0.003 g [1]

Saccharin with chemical compounds (C7H5NO3S) is an artificial sweetener that is added and used for the purposes of processed food products, industry, as well as beverages and food. In addition, saccharin is also widely used to replace sucrose for people with diabetes mellitus or for low-calorie foodstuffs. Excessive consumption of saccharin can cause side effects, including migraines and headaches, memory loss, confusion, insomnia, irritation, asthma, hypertension, diarrhea, stomach pain, allergies, impotence and sexual disorders, baldness, and brain and bladder cancer. . The maximum limit for the use of saccharin based on sugar and other syrup food categories is 500 mg/kg (SNI01-6993-2004). Meanwhile, according to the Minister of Health, the artificial sweetener saccharin is in the threshold of 50-300 mg/kg of ingredients. The permissible aspartame level is a maximum of 600 mg/kg BW [5]

Cyclamate precipitation reaction

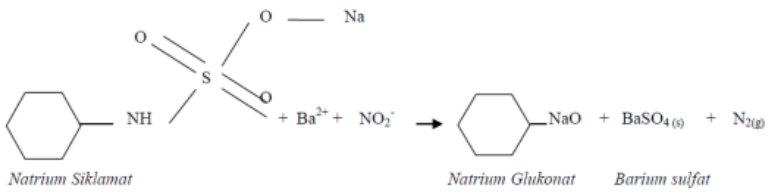


Figure 2. Barium Sulfate Formation Reaction

In the qualitative test of cyclamate with the method of forming a white precipitate. The reason for adding 10% hydrochloric acid is to make the solution acidic, thus facilitating the reaction. Barium chloride is added to precipitate impurities in the test solution. NaNO2 is added so that the sodium cyclamate bond is broken producing nitrogen gas which has a pungent smell when heated.

Qualitative Test with resorcinol

The qualitative test will form a green fluorescence color change when the sample contains saccharin. From the samples obtained the results A, B, C, D, and E formed a green fluorescence color change which indicated that samples A, B, C, D, and E were positive for saccharin. The analysis of the qualitative test of saccharin using the resorcinol method was carried out by pipetting 50 mL of the sample and acidified with 10% HCl in excess and then extracted once with 25 mL of ether, after being separated, the ether layer was evaporated in open air. Then 10 drops of concentrated H2SO4 were added and transferred to a test tube. Then added with 40 mg of resorcinol, and heated slowly with a low flame until it turns a cloudy green color. The test tube was cooled and then added with 1 mL of distilled water and 1-5 drops of 10% NaOH solution. If it turns into a green fluorescence, it indicates the presence of

saccharin. . All samples of beverages that have been tested give a green fluorescence color change which indicates that all samples are qualitatively detected to contain saccharin. Although the qualitative test has identified the presence of saccharin, a quantitative test needs to be carried out to determine the level of saccharin [3].

Quantitative Test Using Alkalimetric Titration Method

The acid-base titration method to determine the levels of saccharin in the sample. This titration method begins with standardization of 0.1 N NaOH solution with 0.1 N KHP solution which has been given phenolphthalein (pp) indicator. The end point is indicated by a color change from colorless to pink. Samples were analyzed quantitatively with NaOH solution, given phenolphthalein indicator (pp) until the sample solution turned pink. Samples were extracted using dilute HCl, chloroform and ethanol so that the saccharin contained in branded soft drinks could be completely bound. After the extraction is complete, two layers will be formed. The layer containing saccharin extract is the lowest layer, namely chloroform because the specific gravity of chloroform is greater than the specific gravity of water and ethanol.

The residue obtained was filtered using filter paper and then tamped in a glass beaker and evaporated in a porcelain cup. The dried residue is dissolved in hot water because saccharin is easily soluble in hot water. The mixture is then titrated with NaOH until the color becomes pink with 1% phenolphthalein indicator. NaOH solution is a secondary standard solution so that before being used for titration it must be standardized with a primary standard solution. The standardization of NaOH in this study was carried out using potassium hydrogen phosphate. The reaction of the PP indicator with NaOH during the titration will form a pink color as the end point of the titration. This study measured the levels of saccharin in all samples by alkalimetric titration method, indicating that there was saccharin in the sample. Based on Permenkes Number 722/Menkes/Per/IX/1988 concerning Food Additives, the saccharin content is 300 mg/kg. Samples A, B, C and D contained saccharin at permissible levels, while sample E contained saccharin exceeding the allowable limit. According to the Food and Drug Administration (FDA) the daily Acceptable Daily Intake of saccharin should not exceed 5 mg/kg body weight [3].

4. CONCLUSION

Artificial sweeteners are substances added to food and beverages with the aim of giving a sweet taste, having no nutritional value. The results of a review conducted on 15 journals are that food in Indonesia contains artificial sweeteners such as cyclamate, saccharin, and aspartame. In several studies, the levels of artificial sweeteners contained in related foods are still within the limits permitted by the government, but in some studies there are still foods whose levels of artificial sweeteners exceed the permitted limits.

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