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Determination of borax content in wet noodles circulating in market by uv-vis spectrophotometry method using curcumin reagent

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ABSTRACT

The determination of borax contains in wet noodles done by curcumin reagent as color regent which produced rosocyanin complexes and monitor by using UV-Vis spectrophotometry in maximum wave length at 545.95 nm. Before it was used in a sample which was taken from Ciputat market, this method was validated. The results showed that calibration curve in linear 6.25-50 µg/ml with regression equal y = 0.008 + 0.012x and correlation coefficient (r) = 0.9994. this method gave detection limit at 3.1132 µg/ml and qualification limit at 10.3775 µg/ml. The simulation recovery of wet nodles was 99.767 ± 1.114 %. The result of examination of wet noodles sample which was marketed in Ciputat market that four of five contained 3.76112 ± 0.0451, 108.592 ± 0.02185, 117.9461 ± 0.01455, and 6.275 ± 0.0221 borax.

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1. INTRODUCTION

Borax, which comes from the Arabic language, namely Bouraq, was originally known to have activity as an antiseptic agent used as a cleaning agent, wood preservative, and herbicide. However, today borax is not used as a cleaner, but is commonly used as a food preservative or thickener. With the presence of borax, the dough can be tougher and more elastic, so it doesn't stretch or sag quickly. Borax is widely used by small industries or home industries, in the manufacture of noodle dough, gendar, or gendar crackers (rice crackers). Noodles are a food product that is very popular with the public, both children and adults, made from wheat flour, rice flour or tapioca flour. In the manufacturing process, especially wet noodles which have a moisture content of 51%, borax is often added to extend their resistance to damage and spoilage. (Winarno et al, 1994).

Although the amount added is not too much, borax has a dangerous accumulation effect. In water, borax is a mixture of sodium metaborate and boric acid. Meanwhile, in acidic conditions, borax decomposes into boric acid. Thus, both when processing food with water and because it is eaten and through the stomach which is acidic, boric acid will be found in the body after consuming foods containing borax. Symptoms of borax poisoningAcute includes initial sensations, vomiting, diarrhea, abdominal cramps, blotches on the skin, decreased body temperature, erythematous skin rash resembling measles and damage to the kidneys, restlessness, and weakness can also occur, death

occurs from respiratory collapse. Whereas chronic poisoning can cause fever, anorexia, anuria, kidney damage, depression and confusion (Haddad et al, 1990; Dreisbach, 1974; Gosselin et al).

Cases of poisoning with borax, not from food, were first reported in 1907. According to the report, many young children suffered from oral thrush, and then applied a mixture of honey and borax. It turned out that after applying it to the skin, there was erythema, and the face looked wrinkled. In Indonesia, precisely in Palembang, South Sumatra, there was a case of borax poisoning from food in mid-1994. It was reported that 5 people died and 56 people had to be hospitalized (Goodman, 1975; Akmal, 1995). In 2009 the Health Office of the city of Depok announced the results of testing in elementary school canteens where on average they were selling typical school snacks, it was proven that 30 percent contained borax and 16 percent contained formalin.

The ban on the use of borax is also strengthened by the Republic of Indonesia Minister of Health No. 1168/MENKES/PER/X/1999 states that one of the BTM that is prohibited from being used in food is borax (Ministry of Health, 1999). Because of these things, this research will determine the levels of borax in wet noodles sold in the Ciputat market. Ciputat Market was chosen because of the location of the market which is close to the UIN Syarif campus

Hidayatullah and is a large traditional market so this research was carried out as UIN's dharma towards the surrounding community.

Determination of borax content was carried out using the visible spectrophotometric method, in which there are two reagents that form color complexes, namely Quinalizarin and Curcumin. In previous studies, the widely used reagent is curcumin where the color complex that occurs is rosocyanin which is rosa in color. (Dibble, 1965)

Based on this, the identification and determination of borax content can be carried out using the UV-Vis spectrophotometry method using curcumin reagent and it is necessary to conduct research on some wet noodles sold in the Ciputat market, because it is possible that these noodles also use borax in their manufacture.

Chemical Name : 1,7 – Bis – (4 – hydroxyl – 3 – methoxyphenyl) – 1,6 – diene – 3,5 dione
Molecular formula : $C_{21}H_{20}O_6$ Molecular weight : 368.39
Melting point : 1790 C – 1820 C

Curcuma domestica Val, while curcumin is a natural compound found in the tubers of the Curcuma longa L plant. Another name for the plant is turmeric, turmeric, or temu kuning (Anonymous, 1992).

In the pure state it can be a crystal in the form of rods or prisms, orange yellow. Soluble in ethanol and glacial acetic acid, insoluble in water and diethyl ether. In alkali it is brownish red, and in acid it is yellow (Windholz et al, 1983).

Curcumin is a natural dye, used for food coloring and cosmetics, as well as a borax indicator. Where curcumin will react with boric acid or borax to form a red rosacyanin chelate complex (Roth, 1978).

2. RESEARCH METHOD

Validation of the analytical method is an act of evaluating certain parameters, based on laboratory experiments, to prove that these parameters meet the requirements for their use.

Samples that are often analyzed by the UV-Vis spectrophotometer method are organic compounds. Organic compounds that can provide absorption are compounds that have chromophore and auxochrome groups. The chromophore group is an unsaturated functional group that gives absorption in the ultraviolet or visible region of light. Almost all chromophores have double bonds such as alkenes (C=C), C=O, - NO_2, benzene, and others. Meanwhile, auxochromes are functional groups such as –OH, -NH_2
-X, which is a group that has nonbonding electrons and does not absorb radiation at λ above 200 nm, but absorbs far UV radiation (Harmita, 2006).

The scope of absorption spectroscopy can be expanded using color reactions, which are often accompanied by increased sensitivity or selectivity. The color reaction is used to modify the spectrum

of the absorbing molecule so that it can be detected in the visible region, and is separated from other interfering compounds that have absorption in the UV region. In addition, these chemical modifications can be used to convert non-absorbing molecules into stable derivatives having significant uptake.

From the noodle solution with a concentration of 25 µg/ml, pipetted 1 ml and then put it in a porcelain cup and added 1 ml of 10% NaOH solution, heated over a water bath until dry, then continued heating in the oven at 1000 ± 50 C for 5 minutes, cooled. Added 3 ml of 0.125% curcumin solution, heated while stirring for 5 minutes, cooled again. Then 3 ml of sulfuric-acetic acid solution (1:1) was added, heated while stirring until there was no yellow color, either in the cup or on the stirrer, left for 15 minutes. mark line. Then the solution was filtered with filter paper. The filter results were collected and observed for their absorption at a wavelength between 400 to 600 nm.

From a solution of 6.25 µg/ml pipetted 1 ml then put into a porcelain cup and added 1 ml of 10% NaOH solution, heated over a water bath until dry, then continued heating in an oven at 1000 ± 50 C for 30 minutes, then cooled at room temperature.

Added 3 ml of 0.125% curcumin solution, heated while stirring for 5 minutes, cooled again. Then 3 ml of sulfuric-acetic acid solution (1:1) was added, heated while stirring until there was no yellow color, either in the cup or on the stirrer, and allowed to stand for 15 minutes.

Added a little alcohol then put into a 100 ml volumetric flask, diluted with alcohol up to the mark line. Filtered with filter paper. The filter results were collected and observed for their absorption at a wavelength of 545.95 nm. Do the same for, 12.5 µg/ml, 18.75 µg/ml, , 25 µg/ml, , 31.25 µg/ml, 37 µg/ml, 43.75 µg/ml, 50 µg/ml for the calibration curve and 10 µg/ml, 20 µg/ml, 30 µg/ml for method validation.

RESULTS AND DISCUSSIONS

1. Results

From the research conducted, the following results were obtained: a. In determining the wavelength, the maximum absorption wavelength of borax is 545.9 nm. Complete data can be seen in Appendix b. Linearity test results and preparation of a calibration curve c. The calibration curve was made using a concentration series in the range of 6.25-50 µg/ml, and the borax calibration curve was obtained as follows:

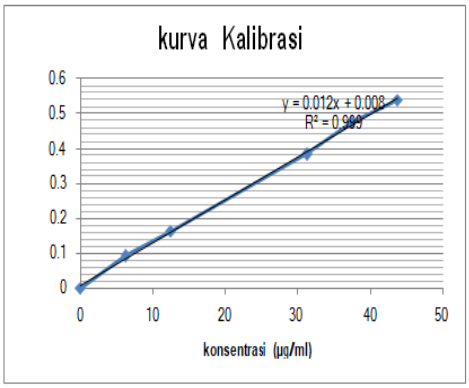

Figure 3. Borax calibration curve on wet noodles

Line equation : y = 0.008 + 0.012x
Correlation coefficient (r) : 0.9994
Complete data can be seen in Appendix 4.

c. In testing the borax detection limit with simulated wet noodles in this experiment was 3.1132 µg/ml while the quantitation limit was 10.3775 µg/ml. Complete data can be seen in Appendix 5 d. The recovery test results (accuracy) of 3 concentrations of borax in simulated wet noodles were 99.767 ± 1.114%. Complete data can be seen in Appendix e. The results of the precision test at 3 concentrations of borax in the simulated wet noodles tested in this experiment gave a coefficient of variation below 2%. Complete data can be seen in Appendix f. In the 2 qualitative tests of the wet noodle samples circulating in the Ciputat market, out of the five samples in circulation, this study was negative for containing borax. Complete data can be seen in attachment 11 g. Determination of the levels of wet noodle samples circulating in the Ciputat market, of the five samples circulating, there were four wet noodles containing borax. The noodles have different levels of borax. Complete data can be seen in the attachment.

2. Discussion

Initially, borax was known as an antiseptic used as a cleaning agent, wood preservative, and herbicide, but now it is widely used as a food additive in noodles, gendar, or kerupuk gendar (rice crackers) as a thickening agent.

preservative. This study used a wet noodle sample, namely noodles containing 51% water content and borax was often added as a chewing agent. Borax absorption repeatedly or excessive absorption can result in toxic (poisoning). In water, borax is a mixture of sodium metaborate and boric acid. Meanwhile, in acidic conditions, borax decomposes into boric acid. Thus, both when processing food with water and because it is eaten and through the stomach which is acidic, boric acid will be found in the body after consuming foods containing borax. Symptoms can include nausea, vomiting, diarrhea, decreased body temperature, weakness, headaches, erythematous rash, and can even cause shock.

The method in this study consisted of several stages so that samples in the form of solids can be used as a solution that can be read on a UV-Vis spectrophotometer. The initial stage was to destroy the wet noodle sample using concentrated H_2SO_4 solution which was covered under reflux and heated at a small temperature of 500-700 C to even out the acid solution for 30 minutes or until the solution turned black and slippery. Then added 30% H_2O_2 oxidizing agent which is useful for reducing organic compounds to C_{02} and H_2O so that the solution will become clear. The use of 30% H_2SO_4 and H_2O_2 is carried out with a ratio of 1:1. Then dilute with aquadest.

This is done because this complex compound is easily hydrolyzed in the presence of water, so efforts are made to remove the existing water by heating.

Borax solution is a colorless solution, this is an obstacle because the solution to be used with UV-Vis spectrophotometry must have a chromophore group marked with color. So in this study borax was reacted with curcumin as a rosocyanin color complex forming which produces rosa color.

Curcumin is a natural dye, besides being used for food coloring and cosmetics, it can also be used as an indicator for the presence of borax in food. By strong acids, borax decomposes from its bonds into boric acid and is bound by curcumin to form a rose colored complex which is often called rosacyanin chelate or Boron Cyanocurcumin Complex. which is a substance that is red, with the reaction as follows.

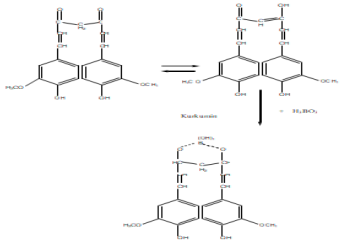

Figure 4. Rosocyanim Complex

The curcumin concentration used was 0.125% based on previous research, that in the range of 0.100% -0.150% curcumin could dissolve completely in acetic acid without a filtering process. The stability of the color complex is 2 hours after the color complex that occurs is dissolved in alcohol in an acidic condition, so that in this experiment the observations on the spectrophotometer were no more than 2 hours after the complex was dissolved in alcohol.

Determining the absorption value of a sample must be at the maximum wavelength so that the maximum value is obtained. In previous studies, the maximum wavelength of borax was 548 nm. However, due to the different sample preparation conditions, it is necessary to determine the maximum wavelength in this study. The determination was carried out using wet noodle simulation with a borax content of 25 µg/ml. From the measurement results the maximum absorption wavelength of the borax is 545.95 nm based on the highest value from the absorption data.

A research method needs to be validated to prove that the results obtained are accurate and meet the requirements for their use. Validation of the analytical method is an act of evaluating certain parameters that aims to ensure that the analytical method used is accurate, specific, and resistant to the range of analytes to be analyzed and in accordance with the . In this study, the validation parameters were linearity, detection limit and quantization limit, accuracy and precision. Using a noodle sample made by yourself with the addition of borax at a certain level, this is intended to prevent too much deviation.

The first stage is making a calibration curve. The calibration curve is calculated based on the equation obtained from the absorbance values in various concentration series. The concentration series that was created was 6.25-50 µg/ml and this calibration curve was used to obtain test results that were directly proportional to the analyte concentration in the given range. From the calibration curve, we get the regression equation y = 0.012x + 0.008 with a correlation coefficient (r) of 0.9994. The acceptance criterion of the correlation coefficient is (r) of ≥ 0.9995, which means that the result of the curve between absorbance and concentration is linear, that is, if there is an increase in the concentration value, the absorbance value also increases. (Ibrahim, 2009; Harmita, 2006).

Based on the calibration curve data, method validation can be carried out, namely linearity, quantitation limit, and detection limit. Linearity is the ability of an analytical method to provide a response that is directly or with the help of a good mathematical transformation, proportional to the concentration of the analyte in the sample. As a parameter, there is a linear relationship, the correlation coefficient r is used in the linear regression analysis Y = a + bx. An ideal linear relationship is achieved if the value of b = 0 and r ≥ 0.9995 and the value of a indicates the sensitivity of the analysis, especially the instrument used (Harmita, 2006). The results obtained are Y = 0.012x + 0.008 with r = 0.9994, so it can be concluded that the results obtained meet the requirements of the linearity parameter.

The next parameters that use calibration curve data are detection limits and quantity limits. The detection limit is the smallest amount of analyte in a sample that can be detected which still gives a significant response, while the quantitation limit is a parameter in the analysis as the smallest quantity of analyte in a sample that can still meet the criteria carefully and thoroughly (Harmita, 2006).

Limits of detection and quantitation are calculated statistically via the linear line of the calibration curve. In this study, the detection limit value was 3.1132 µg/ml and the quantitation limit was 10.3775 µg/ml. These results stated that the smallest value that could be detected and still gave a significant response was 3.1132 µg/ml and the smallest quantity was 10.3775 µg/ml which still met the criteria of being careful and thorough.

Furthermore, the accuracy test is carried out which is the degree of closeness of the results obtained to the actual analyte levels. The accuracy parameter was determined by measuring the absorbance of three concentrations of borax-containing artificial noodle solutions. The accuracy of the method can be seen from the percent recovery of borax in the noodles. The recovery percentage obtained in this study was 99.767 ± 1.114%. Results that meet the requirements of the recovery test are 98-102% (Harmita, 2006). In the reassignment of borax content using the Uv-Vis

spectrophotometry method, many factors cause the loss of borax content, namely from the process of making noodles to observing it on the spectrophotomer, where the loss of levels cannot be avoided so as to overcome this,

The next test is the exactness test, namely the degree of repeatability of an analytical method. The precision parameter was determined by measuring the absorbance of three concentrations of borax in noodles three times in one day. The accuracy of the method can be measured from the coefficient of variance of the data. The coefficient of variation obtained in this study is 1.12%. The value obtained meets the criteria of the exactness test, which is ≤ 2% (Harmita, 2006).

After obtaining a validation value that can meet the criteria, then identification of borax in noodles circulating in the Ciputat market is carried out. Sampling methods are investigative and random methods. Get five big sellers in the market. So that the samples taken were five samples of wet noodles from the Ciputat market. The first identification was carried out qualitatively with two tests, namely the flame test and the curcumin test. In the flame test, 1 ml of concentrated sulfuric acid and 5 ml of methanol were added to the sample which was then burned. An indication of the presence of borax in the sample is the presence of a green flame when it is first burned. The results for the five market samples were negative or they did not produce a green flame when burned. Methanol will burn with a green flame due to the formation of ethyl borate or metal borate (Soetiono, 1985; Basir, 1992). The second test was carried out by adding curcumin with a change in color as an indicator. 20 grams of the market sample was added with sodium carbonate, then charred with a Bunsen flame and then incinerated in a furnace. Add 5 N hydrochloric acid and filter. Add saturated oxalic acid and curcumin dissolved in ethyl alcohol then evaporate over a water bath. The color change as a positive indicator that occurs is cherry red which will change color to blackish green after being given dilute sodium hydroxide. In the market sample, the color that appears is cherry red, but when dilute sodium hydroxide is added, it does not change color or remains cherry red. So it was concluded that there was no borax in the market noodle sample. The second test was carried out by adding curcumin with a change in color as an indicator. 20 grams of the market sample was added with sodium carbonate, then charred with a Bunsen flame and then incinerated in a furnace. Add 5 N hydrochloric acid and filter. Add saturated oxalic acid and curcumin dissolved in ethyl alcohol then evaporate over a water bath. The color change as a positive indicator that occurs is cherry red which will change color to blackish green after being given dilute sodium hydroxide. In the market sample, the color that appears is cherry red, but when dilute sodium hydroxide is added, it does not change color or remains cherry red. So it was concluded that there was no borax in the market noodle sample. The second test was carried out by adding curcumin with a change in color as an indicator. 20 grams of the market sample was added with sodium carbonate, then charred with a Bunsen flame and then incinerated in a furnace. Add 5 N hydrochloric acid and filter. Add saturated oxalic acid and curcumin dissolved in ethyl alcohol then evaporate over a water bath. The color change as a positive indicator that occurs is cherry red which will change color to blackish green after being given dilute sodium hydroxide. In the market sample, the color that appears is cherry red, but when dilute sodium hydroxide is added, it does not change color or remains cherry red. So it was concluded that there was no borax in the market noodle sample. 20 grams of the market sample was added with sodium carbonate, then charred with a Bunsen flame and then incinerated in a furnace. Add 5 N hydrochloric acid and filter. Add saturated oxalic acid and curcumin dissolved in ethyl alcohol then evaporate over a water bath. The color change as a positive indicator that occurs is cherry red which will change color to blackish green after being given dilute sodium hydroxide. In the market sample, the color that appears is cherry red, but when dilute sodium hydroxide is added, it does not change color or remains cherry red. So it was concluded that there was no borax in the market noodle sample. 20 grams of the market sample was added with sodium carbonate, then charred with a Bunsen flame and then incinerated in a furnace. Add 5 N hydrochloric acid and filter. Add saturated oxalic acid and curcumin dissolved in ethyl alcohol then evaporate over a water bath. The color change as a positive indicator that occurs is cherry red which will change color to blackish green after being given dilute sodium hydroxide. In the market sample, the color that appears is

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After conducting qualitative tests, then proceed with quantitative testing. For quantitative analysis, this research was carried out using the UV-Vis spectrophotometry method using curcumin reagent, where boron from the borax compound is bound by curcumin to form a rosa color complex. Of the five samples tested, four samples contained borax of 3.76112 ± 0.0451 in samples originating from production in Ciputat.1, 108.592 ± 0.02185 in samples originating in Ciputat 2, 117.9461 ± 0, 01455 in samples originating from production in Parung, and 6.275 ± 0.0221 in samples originating from production in Tangerang.

CONCLUSION

The validation of the method that has been carried out can meet the requirements seen from the results, including the results of the linearity test in the concentration range of 6.25-50 µg/mL with a correlation coefficient value (r) of 0.9994. The detection limit of the borax noodle solution was 3.1132 µg/mL and the quantitation limit was 10.3775 µg/mL. The recovery percentage of the 3 simulated noodle solutions obtained in this study was 101.09

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