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Determination of Boron Concentration at Sausage Samples with Distillation of Ester Borane Method using Fluorescence Spectrophotometry

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Abstract. The study of borax contamination in food samples was successfully analyzed. This study used sausage samples using the Fluorescence Spectrophotometry method. Sausage samples were from Daerah Istimewa Yogyakarta (DIY). This study used 4 sausage samples. The samples were given labels A, B, C, and D. The steps of the research were optimation of distillation time, optimation of catalyst, optimation of solvent, and then followed by analyzing the borax concentration on sausage samples. Based on this study, the optimum distillation time was 24 hours. On the other hand, the best catalysts and solvents were sulphuric acid and ethanol. The A and B samples contained borax contamination and the concentration was under 5 ppm. The C and D samples contained borax contamination and the concentration was above 5 ppm. The conclusions were that A and B samples were not given borax addition and C and D samples were given borax addition.

Keywords: *Borax, Fluorescence Spectrophotometry, Sausage, distillation time, catalyst, solvent*

Abstrak. Penelitian tentang analisis kandungan boraks pada sampel makanan telah berhasil dilakukan. Pada penelitian ini menggunakan sampel sosis dengan menggunakan metode spektrofotometri fluoresensi. Sampel sosis yang dianalisis merupakan sosis yang didapat dari swalayan dan pasar tradisional di Daerah Istimewa Yogyakarta (DIY). Pada penelitian ini, menggunalan dua sampel sosis sebagai bahan tambahan makanan yaitu sampel A dan B, dan sampel sosis siap makan yakni sampek C dan D. Tahapan penelitian ini adalah melakukan uji optimasi waktu distilasi, optimasi katalis, optimasi pelarut, dan terakhir uji kadar boraks pada 4 (empat) macam sampel sosis. Berdasarkan hasil penelitian, diperoleh hasil waktu distilasi yang optimum adalah distilasi selama 24 jam. Sementara itu untuk optimasi katalis, katalis yang optimum pada dsitilasi ester boraks adalah dengan menggunakan asam sulfat, sedangkan untuk pelarut terbaik adalah etanol. Hasil uji kadar boraks dalam sampel sosis didapat hasil sosis A dan B mengandung boraks di bawah 5 ppm, sedangkan sampel C dan D mengandung boraks di atas 5 ppm, sehingga dapat disimpulkan bahwa sampel sosis A da B tidak ada tambahan boraks sebagai bahan pengawet makanan, sedangkan sampel C dan D diberi tambahan boraks sebagai pengawet dan pengenyal makanan.

Kata kunci: *Boraks, spektrofotometri fluoresensi, sosis, waktu distilasi, katalis, pelarut*

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INTRODUCTION

The basic need of humans is food as a nutrition source for the body and to grow and sustain life. If there is no food, human can not do their daily activities (Falahudin et al., 2016). In daily life, snacks can be found everywhere. Snacks contain 36% energy, 29% protein, and 52% another substance. Consuming foods or snacks must have special attention especially the quality of food because bad-quality snacks cause problems for the human body (Berliana

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et al., 2021). There are food additives in some snacks and foods. Food additives are added in the food processing step until the food storage step to get good quality food (Juwita et al., 2021). Technology and scientific development cause foods sold as instant food that can increase food durability. It happens because urban people demand the foods produced in the form of instant food (Bemis et al., 2021). Therefore, the safety food regulation is needed for the food safety consumed by people. The effort to prevent dangerous substance food contamination is food safety and government regulations (Utomoa et al., 2018) (Nopianti et al., 2018).

One of the chicken meat products is sausage. Sausage contains meat, filler, and ingredients (Ismanto A, 2020). In recent decades, to increase food durability, formalin and borax have been added, especially in noodles, sausages, and meatballs (Saputrayadi et al., 2018). Borax and formalin are used as preservatives and spongy substances. The substance for the food coloring of sausage, tempura, fishball, etc is Rhodamin B (Saktiningsih et al., 2023; Hartati, 2017). The foods are sold in traditional markets or supermarkets (Hartati, 2017).

The empirical formula of borax is $Na_2B_4O_7.10H_2O$. Borax is a white powder, odorless, and stable at room pressure and temperature. Borax is a raw material of glass, a preservative substance for wood, and wood antiseptic (Azmi et al., 2018). The other names of borax are sodium piroborate, sodium biborate, and sodium tetraborate. Consuming borax for a long time about 5-10 g/kg causes kidney damage and death (Suseno, 2019).

Boric acid is of dangerous chemical substance. This compound causes human poisoning. The mechanism is when boric acid is swallowed by humans, it will accumulate in the brain, intestines, testicles, or liver, causing cancer (Muharrami et al., 2015). Based on Indonesian Minister of Health regulations No. 033 2012 about food additives conclude that borax and formalin are forbidden to be used as food additives in Indonesia (PERMENKES RI No. 033 tahun 2012).

The quantitative analysis can use a spectrophotometer UV-Vis and spectrophotometer fluorescence method. Spectrophotometer UV-Vis can be used to analyze organic and inorganic compounds, selectively, with and high accuracy of about 1%-3%, fast analysis, appropriate, and can be used in small samples (Siti Awwalul Amanatur Rohmah, 2021).

Gusfianang Haryarta, *et al* 2021 did research on the analysis of borax concentration in meatball samples using the fluorescence spectrophotometer method. An atom will absorb energy and the electrons move from the ground state to the excitation state. The process emitted light. This phenomenon is called fluorescence. The variation of borax concentration produces different intensities using a fluorescence spectrophotometer. This instrument is a good technology for analyzing very small compounds in samples with high sensitivity (Haryanta et al., 2021).

The determination of borax compounds using the spectrophotometry method needs special treatment, especially for the matrix in the samples. One of the ways is separating the impurities using the distillation method. The distillation ester borane method is more effective and efficient for boron analysis in food because it uses portable distillation and is

environmentally friendly because it uses few ingredients (Adu et al., 2022).

In this study, we analyze the boron concentration in food samples. The food samples in this research are sausage samples from Daerah Istimewa Yogyakarta (DIY). The steps of this research are the optimation of distillation time, optimation of catalyst, optimation of solvent, and analysis of the boron concentration in sausage samples.

MATERIAL AND METHODS

Materials

The materials used in this research were sausage samples from traditional markets and supermarkets, curcumin (Merck), absolute ethanol (Merck), H_2SO_4 (Merck), CH_3COOH (Merck), methanol (Merck), HCl (Merck), oxalic acid dehydrated (Merck), and Acetone (Mallinckrodt).

Instrumentation

This research used a Fluorescence spectrophotometer Shimadzu RF6000, Socorex micropipette 0.5 µL – 10 µL, Socorex micropipette 10 µL - 100 µL, Socorex micropipette 100 µL - 1000 µL, magnetic stirrer, analytical balance (Kern), water bath, mortal and agat, distillation bottle made from Teflon consists of inner teflon and outer teflon are presented in **Figure 1**., and some glassware for analysis.

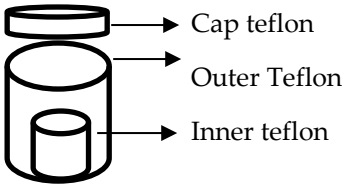

Figure 1. Distillation bottle

Procedure

Optimation of distillation time

The esther borate distillation method was used to determine the optimum distillation time of samples (Adu et al., 2023). The first step was to determine the optimum distillation time. First, 100 mg of boric acid was added into ethanol 0.5 mL and was poured into the inner teflon. Then, 2.5 µL of H_2SO_4 and 10 mL of curcumin were added into the outer teflon. The main steps were distilling the mixture for about 6, 12, 24, 48 and 72 hours. The distillation result was complex curcumin boron and then added into the beaker glass. It was analyzed in triplicate.

Optimation of catalyst and solvent

The second step was optimation of the catalyst and solvent used in distillation of boric acid. First, 100 mg of boric acid poured into ethanol 0.5 mL in the inner teflon. Then, 2.5 µL H_2SO_4, CH_3COOH, and HCl catalyst were added into the mixture solution. Next, 10 mL of curcumin was added to the outer teflon and the mixture solution was distilled at room temperature for about 24 hours. It was analyzed in triplicate.

The steps of optimation of solvent were by adding about 100 mg of boric acid into ethanol, methanol, and propanol for about 0.5 mL. The mixture solution was poured into the teflon that contained of 2.5 µL H_2SO_4. 10 mL of curcumin solution was added to the outer teflon and was distilled for about 24 hours. It was analyzed in triplicate.

Analysis of borax in the sausage samples

The boron analysis in food samples uses fluorescence spectrophotometer. About 5.00 g sausage sample was added into a double-walled distillation tool and was distilled. Next, the boron-curcumin complex was poured into a

beaker glass and was poured solvent into a measuring flask. The mixture was heated using a water bath at temperature 75 °C until dry and then acetone was added to the complex. The samples were analyzed three times with the same procedure.

RESULT AND DISCUSSION

The Spectra of Fluorescence Spectrophotometry Wavelength

The results of measurements of boron-curcumin solution intensity at 3, 4, and 5 ppm concentrations with 522 nm excitation wavelength and emitted at 593 nm wavelength are presented in Figure 2.

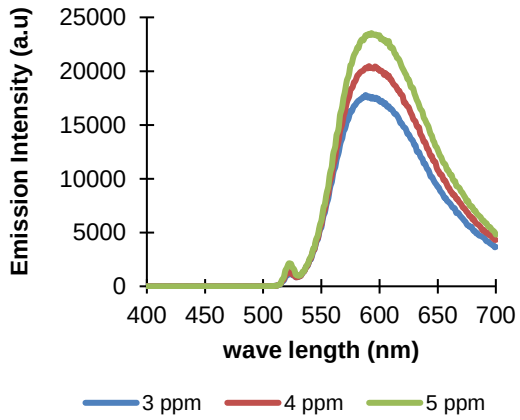

Figure 2. Fluorescence spectrophotometry wavelength of 3, 4, and 5 ppm

The wavelength excitation of boron-curcumin oxalate complex was 522 nm and the wavelength emission was 593 nm, with excitation intensity at a 3 ppm concentration was 1171 and emissions was 17780. At 4 ppm excitation concentration was 1339 and emissions were 20465. At 5 ppm concentration, excitation was 2130 and emissions were 23538. The use of curcumin reagents was to form boron-curcumin complexes. Boron does not have a chromophore group, but after the formation of the boron-curcumin complex, the boron-curcumin complex has a fluorophore group and an auxophore group that can produce fluorescence light.

The Effect of Distillation Time

The distillation time optimation aims to determine the optimum distillation time. The optimum distillation time is indicated by the maximum intensity result.

Table 1. Optimation of distillation time

No.	Distillation time (hours)	Intensity
1	6	2731
2	12	12691
3	24	13725
4	48	11529
5	72	10573

Table 1 shows the distillation time using time intervals. The 6 hours distillation time shows an intensity of 2731, and then the intensity increases when the distillation time is 12 hours followed by 24 hours. On the other hand, when distillations time are 48 hours and 72 hours, the intensities tend to decrease. It happened because the stability of the curcumin oxalate complex has optimum time distillation in 24 hours (Lailatusholihah, I et al, 2022). Therefore the 48 and 72 hours distillation time have lower intensity than 24 hours. The graph is shown in Figure 3.

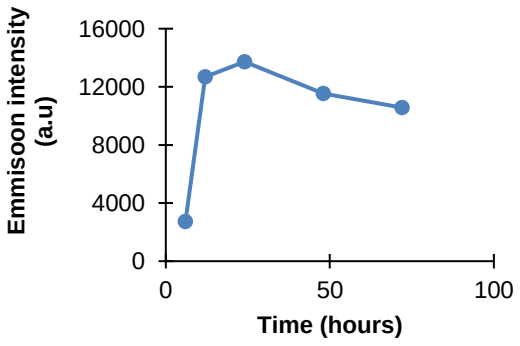

Figure 3. Graph of distillation time in 6, 12, 24, 48, and 72 hours with emission wavelength 593 nm

The Effect of Catalyst

The next research determines the optimum catalyst. The distillation time used in this research is 24 hours. The catalysts used in this research are sulphuric acid, acetic acid, and chloride acid. The catalysts's optimation results are shown in Figure 4.

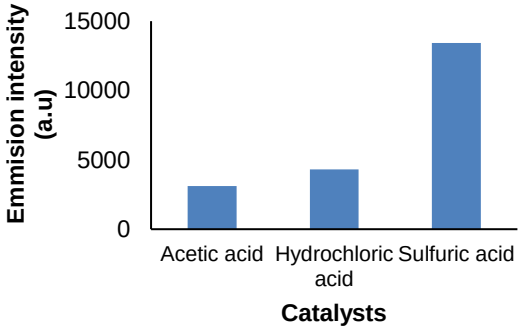

Figure 4. The influence of catalysts in emission wavelength 593 nm

Sulphuric acid and chloride acids are strong acids, in the other hand acetic acids is a weak acid. The H^+ concentration in H_2SO_4 is stronger than HCl. A more H^+ concentration means the more intermediate compound formed (Artati et al., 2012). Intermediate compound or free radical is a very reactive compound, so can be used as a catalyst.

Based on the research, the graph shows that when using a sulphuric acid catalyst, the reaction becomes faster than using chloride acid and acetic acid. Sulphuric acid is a strong acid catalyst and can dehydrate and changes the evaporation when distillation process of ester borate, so the intensity result of sulphuric acid solvent gives the highest intensity compared to acetic acid and chloride acid solvents.

The Effect of Solvent

The next research is to determine the solvent effect. In the boron distillation process, there is needed solvent to separate boron and impurities. This research uses three solvents (ethanol, propanol, and methanol). The solvents optimation results are shown in Figure 5.

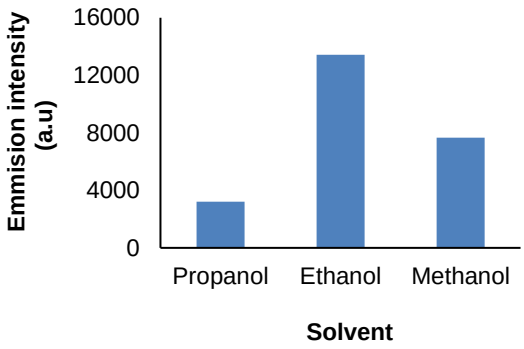

Figure 5. The influence of solvent in emission wavelength 593 nm

The alcohols that used in this research were primary alcohols because primary alcohol will give maximum results compared to secondary and tertiary alcohol. The distillation processes are to separate the methanol at a temperature of 64.5°C, to separate the ethanol at a temperature of 78.4 °C, and to separate the propanol at a temperature of 97 °C. The results show that using ethanol solvent gets the highest intensity among three solvents (ethanol, methanol, and propanol). This happened because of the boiling point differences of each solvent.

The Borax Analysis Result of Sausage Samples

The analysis of boron concentration in this research is to determine the boron concentration in sausage samples. The sausage samples are bought in traditional markets and supermarkets in Daerah Istimewa Yogyakarta (DIY). There are two different kinds of sausage, the A and B sausage are for mixed dishes, and the C and D sausage are for snacks. The analysis data of boron concentration in sausage samples are shown in Figure 6.

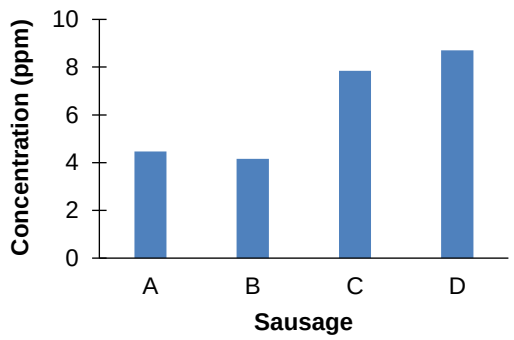

Figure 6. The analysis result of boron in the sausage

The raw materials of sausage are a mixture of meat and flour. The sausage contains boron naturally because boron can be found in animals and plants naturally. In our body, boron has a role in preventing osteoporosis. The human's body needs about 70 – 110 mg a boron every day.

Based on the research, the boron concentration in the A sample is 4.461±0.004 ppm, in the B sample is 4.161±0.001 ppm, in the C sample is 7.839±0.001 ppm, and in the D sample is 8.702±0.002 ppm. In A and B samples, boron concentration is under 5 ppm. This means that there is no boric acid addition in this sample. However, in C and D samples, the concentration of boron is more than 5 ppm. It means that the C and D samples have boric acid addition as a chewing and preservative agent. The result of distillation sausage samples is shown in Figure 7.

Figure 7. The result of distillation sausage samples.

CONCLUSION

The optimum distillation time of boric acid is 24 hours and the best solvent for boric acid distillation is ethanol with sulfuric acid as a catalyst. The distillation ester borane method is more effective and efficient for boron analysis in food because it uses portable distillation and is environmentally friendly because it uses few ingredients. From the 4 sausage samples, we can conclude that A and B samples contain boric acid less than 5 ppm, it means that there is no boric acid addition in the samples. However, C and D samples contain boric acid a more than 5 ppm. C and D samples have boric acid addition and the purpose of boric acid addition in the samples is as a preservative and chewing agent material.

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