Gravitasi Vol. 22 No. 2 (Des 2023) 45-48

Gravitasi

Journal available at: https://bestjournal.untad.ac.id/index.php/GravitasiFisika

e-ISSN: 2654-4318 p-ISSN: 1412-2375

Analysis of Lead Content in Cereal-Based Foods Using Atomic Absorption Spectrometer (AAS)

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Information

Article history:

Received: 02 October 2023

Accepted: 02 June 2024

Published: 09 June 2024

Keywords:

Cereal

GFA AAS

Lead

Vermicelli

Abstract

Cereal-based foods are foods with the highest level of consumption because they have high levels of carbohydrates. Food is also the largest contributor to heavy metal contamination that enters the human body, especially heavy metal of Pb reaching 90%. Pb heavy metals with too high levels in humans can damage human organs and cause death. Research related to the analysis of heavy metal content in cereal foods, especially vermicelli has been carried out. The analysis method uses GFAAAS (Graphite Furnace Analysis Atomic Absorption Spectrometer). Some solutions that must be prepared include vermicelli sample solution, working standards, and aquadem as a diluent. Based on BPOM regulation No. 5 of 2018 states that lead content in vermicelli must be less than 0.25 mg/Kg. There are three samples in this research which are sample 1 is vermicelli from brand A, sample 2 is vermicelli from brand B and sample 3 is vermicelli from brand C. Results indicate that the first sample is not qualified (NQ), but the second and third samples are qualified because they are negative at the time of rough determination of heavy metal lead (Pb).

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DOI: 10.22487/gravitasi.v22i2.16589

1. INTRODUCTION

Food with the highest level of consumption in Indonesian people is cereal-based food, including in toddlers which reaches 99.2% [1], [2]. Cereals-based foods are used as human staple foods because the content of carbohydrates, starches, and sourced from dry raw materials is around 80% [3]. One example of cereal-based food that is widely circulated in the market is vermicelli because it is made from rice raw materials which are processed into rice flour [4]. However, the high consumption of cereal-based foods is not supported by high knowledge of food in the community, especially adolescents. Based on research in the Bangkalan district, it was found that 65% of street children or adolescents have low knowledge related to food [5].

This will have an impact on the consumption of unhealthy foods such as having heavy metal contamination such as lead which has been found in many foods. The percentage of lead metal contamination that enters the human body through food reaches 90% with the four main sources of lead metal contamination in food. The fourth source of lead metal contamination in food is air pollution, equipment used in every manufacturing process, food packaging, and soil conditions that are the source of raw materials [6], [7]. High lead metal content can disrupt the male reproductive system, if the concentration in the blood exceeds 20 µg/dl will cause anemia [8]. Lead content is also very difficult to excrete because it can be stored in soft tissues such as bones, blood, kidneys, lungs, and heart. So it will cause mitochondrial degradation and damage [9]. In addition, WHO also stated that there is no safe concentration of lead for the human body [10]. Meanwhile, analysis of lead content in food has been carried out, for example in fried foods by Umar et al (2021) and Perdana et al (2017) using AAS, and on bran by Tyas (2022) using AAS [11]–[13] but still no one has analyzed the lead content in vermicelli which is part of cereal-based foods. Therefore, there is a need for analysis related to lead content in vermicelli and its relation to lead concentration standards in cereals-based foods.

According to BPOM regulation No. 5 of 2018 states that lead content in cereal-based foods must be less than 0.25 mg/Kg (except wheat flour as a food ingredient of 1 mg/Kg) [14]. Due to the high rate of distribution of cereal foods and also the low level of public knowledge about the dangers of cereal-based foods, the author wants to analyze the content lead in cereal-based foods and their relationship with standard Pb levels in cereal-based foods using AAS instruments and analysis methods using Graphite Furnace Analyzer because has a good level selectivity, sensitivity, besides that it is also suitable for measuring contamination in small amounts [15].

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2. MATERIALS AND METHOD

Time and Place

This research was conducted in December 2022 at the Food Laboratory, Center for Drug and Food Control (BBPOM)

Palembang and Laboratory of Sains Material, Faculty of Mathematics and Natural Science, University of Sriwijaya.

Tools and Materials

The tools used in this study were the Atomic Absorption Spectrometer (AAS) with the limit of detection is 2.4 ppb, measuring flasks 25 mL and 100 mL, *microwave*, vessel and vial. The materials used in this study were 65% nitric acid, aquadem, Pb 1000 ppm, sample 1 is vermicelli from brand A, sample 2 is vermicelli from brand B and sample 3 is vermicelli from brand C.

Research Procedure

Working Standard Solution Preparation

A standard solution of Pb 1000 ppm is taken as much as 1 mL which is then put into the first 100 mL measuring flask, added aquadem to the limit on the measuring flask with the concentration of Pb being 10 ppm. Then another 1 mL of solution is taken from the 100 mL first measuring flask and put into the 100 mL second measuring flask, then aquadem is added to the limit on the measuring flask and homogenized untill the concentration of Pb being 100 ppb.

Sample Preparation

Each sample, namely sample 1, sample 2, and sample 3, was weighed using a digital balance weighing 0.2 grams each in a vessel with each sample being duplo, then the threes samples were destroyed by adding 65% nitric acid (HNO_3) as much as 5 mL and closed tightly. After that, the vessels are put in the microwave for 45 minutes including the cooling time of the sample. The vessels are then taken to the fume hood to open the vessels lid, then the three samples are put into a 25 mL measuring flask for each sample and added aquadem until the limit of the mark on the measuring flask.

Data Retrieval from AAS

Samples and working raw solutions are poured into vials, then vials are placed on AAS according to position, then data is entered by AAS according to the numbering on the AAS container. In addition, blank samples and dilution using aquadem are also needed. Analysis uses a Graphite Furnace analyzer because it is suitable when used with samples with low volumes ranging from 5-15 mL.

Determination of Pb levels in samples

Determination of levels is done manually because the data released by AAS is in the form of absorbance and solution concentrations. Determination of levels can be assisted by Excel to facilitate calculations, in the process of determining levels required sample absorbance values, blanko absorbance, a and b values in the regression formulation, sample weight and measuring flask volume according to Equation (1).

$$\text{Concentration of Pb} = \frac{Sample\ Abs - Blanko\ Abs - a}{b}\ x\ \frac{Measuring\ Flask\ Volume}{Sampel\ Weight\ x\ 1000} \quad (1)$$

3. RESULTS AND DISCUSSION

The results in Table 1 show that the greater the concentration of the working standard solution, the greater the absorbance value. The obtained results are in accordance with the working principle of AAS which provides energy that will be absorbed by atoms. When atoms absorb the provided energy, an excitation process will occur [16]. Then the excited atoms will be vaporized in the AAS and the vaporized atoms are then analyzed by the AAS [17]. The AAS method used is Graphite Furnace Atomic Absorption Spectrometer (GFAAS) because this method is very good for analyzing heavy metal concentrations in very low volumes of sample solutions [15]. The concentration and absorbance of the working standard solution greatly determine the feasibility of sample testing, because the working standard is a representation of the readiness of the AAS system. From Table 1 regression values and their correlations analytically which will affect the calculation of determining lead levels (Pb) in the sample.

Table 1. Measurement data of Pb working raw solution using AAS

No	Concentration Level (µg/L)	Absorbance (y)
1	0	0,0018
2	1	0,0115
3	2,5	0,0206
4	5	0,0514
5	10	0,1124
6	80	0,4515
7	100	0,5406

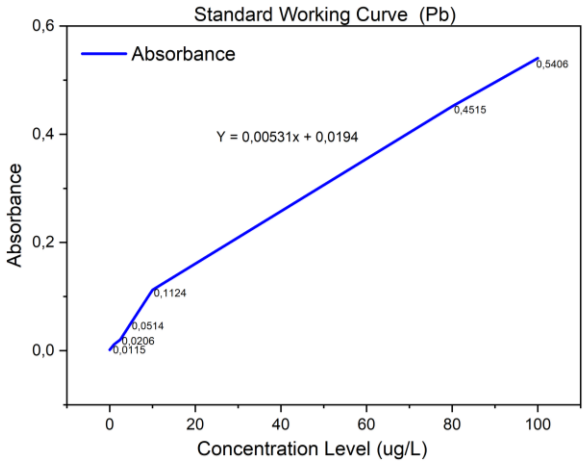

Figure 1. Standard Absorption Curve of Work Pb

Based on Figure 1 it can be seen that the values of absorbance and concentration are always directly proportional and show very good elasticity. Analytically, it is found that the correlation value is 0.9922 and regression values such as *slope* (a) is 0.00531 and *intercept* (b) is 0.0194. The correlation value reaches 0.9922, indicating that there is a very close relationship between the concentration of the analyzed solution and the absorbance value obtained. In addition, the correlation value can also be a form of validation of the method used in analyzing the concentration of Pb in vermicelli. The correlation value obtained has met the requirements of a good correlation threshold of more than 0.98. The correlation value of <0.98 indicates that the absorbance value of the sample is of doubtful validity, conversely, if the correlation value is >0.98 indicates that the validity of the sample does not need to be doubted [18]. After all the values needed to determine the Pb level in the sample are obtained then the calculation of determining the level can be done using Equation 1.

Based on the ethical formula, the absorbance value of the sample is smaller than the sum of the blanko absorbance and the value of a, the level results must certainly be negative. Blanko absorbance is the absorbance value of the blank solution which in this study uses aquadem. After the calculation is carried out based on Equation 1, the results of

lead levels (Pb) in the three samples can be listed in Table 2. BPOM regulation No. 5 of 2018 states that the lead content in vermicelli must be less than 0.25 mg/Kg (except wheat flour as a food ingredient of 1 mg/Kg) [14]. Based on BPOM regulation No. 5 of 2018, it can be determined the feasibility of a sample with Qualified Information (Q) if the yield level of (Pb) is less than 0.25 mg/Kg and Not Qualified (NQ) if lead content (Pb) is more than 0.25 mg/Kg.

Table 2. Results of Determination Pb's Concentration in cereal-based food samples

Solution	Testing	Absorbance	Concentration of Pb (ppm)	Information
Sample 1	Testing 1	0,0560	0,4812	NQ
	Testing 2	0,0490	0,4071	NQ
Sample 2	Testing 1	0,0069	-0,6132	Q
	Testing 2	0,0092	-0,5191	Q
Sample 3	Testing 1	0,0031	-0,6578	Q
	Testing 2	0,0041	-0,6252	Q
Blanko		0,0130		

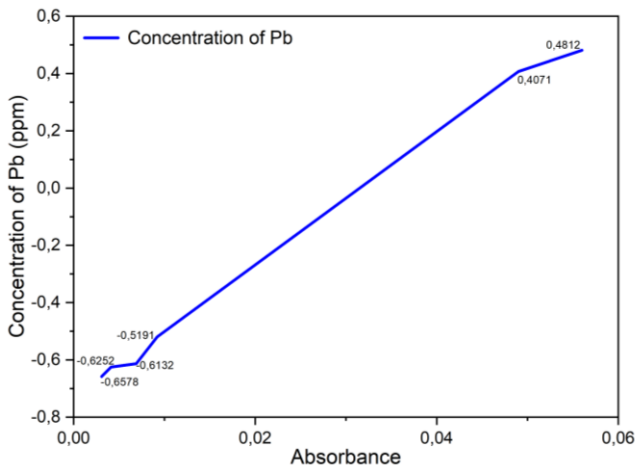

Figure 2. Pb's Concentration Determination Curve

Based on Figure 2 it can be seen that the absorbance value is directly proportional to the Pb content in the sample, because the absorbance value will be subtracted by the blank absorbance variables and a. When the absorbance value of the sample the greater the difference between the absorbance value of blanks and a will be further because the absorbance of blanks and a is constant. The cause of high Pb levels in the first sample may be caused by several factors such as the location of the sample manufacture, tools, materials used to make the sample, or air inhalation during the sample wrapping process. According to Equation 1 the second and third samples have a negative value because the absorbance value of the sample is smaller than the absorbance value of the blank where the blank is an aquadem solution. Aquadem is a dilution solution used in this study so that it can be used as a neutral standard parameter of Pb content. Theoretically, the negative value in the second and third samples are due to the absorbance value being lower than the specified detection limit of 2.4 ppb (µg/L) [19].

4. CONCLUSION

Results show that the three samples are vermicelli. The first sample is not qualified (NQ) because the value is >0.25 mg/Kg, while the second and third samples are qualified (Q) because the value is <0.25 mg/Kg based on BPOM regulation No. 5 of 2018.

ACKNOWLEDGMENT

This research was supported by BBPOM Palembang and Laboratory of Sains Material, Faculty Mathematics and Natural Science, University of Sriwijaya as the place to do this research.

REFERENCES

[1] M. Prijono, N. Andarwulan, and N. S. Palupi, "Perbedaan Konsumsi Pangan dan Asupan Gizi pada Balita Stunting dan Normal di Lima Provinsi di Indonesia," *J. Mutu Pangan*, vol. 7, no. 2, pp. 73–79, 2020, doi: 10.29244/jmpi.2020.7.2.73.

[2] N. H. Utami and R. Mubasyiroh, "Keragaman Makanan Dan Hubungannya Dengan Status Gizi Balita: Analisis Survei Konsumsi Makanan Individu (SKMI)," *J. Indones. Nutr. Assoc.*, vol. 43, no. 1, pp. 37–48, 2020, doi: 10.36457/gizindo.v.

[3] M. Nopriyanti, E. Radwitya, T. Adimarta, and E. Ernayani, "Karakteristik Kimia dan Analisis Sensori Pada Dodol Nanas Dengan Perbandingan Tepung Ketan dan Tepung Beras," *J. Teknol. Pangan Dan Agroindustri Perkeb.*, Vol. 2, No. 2, pp. 183–190, 2022, [Online]. Available: https://jurnal.politap.ac.id/index.php/lipida/article/view/794%0Ahttps://jurnal.politap.ac.id/index.php/lipida/article/download/794/482

[4] Hardoko, A. Chamidah, M. A. P. Panjaitan, and A. N. F. Haryady, "Arakterisitik Fisikokimia Mi Bihun Beras Substitusi Parsial Tepung Rumput Laut Eucheuma cottoni," *J. Fish. Mar. Res.*, Vol. 5, No. 2, pp. 318–328, 2021, doi: 10.33964/jp.v30i3.530.

[5] A. R. 'Aisy, D. Y. Fitranti, R. Purwanti, D. M. Kurniawati, and H. S. Wijayanti, "Faktor-Faktor Yang Berhubungan Dengan Keragaman Pangan Pada Anak Jalanan Di Kota Semarang," *J. Nutr. Coll.*, vol. 8, no. 4, pp. 254–263, 2019, doi: 10.14710/jnc.v8i4.25839.

[6] C. C. Huang, C. M. Liang, T. I. Yang, J. L. Chen, and W. K. Wang, "Shift of bacterial communities in heavy metal-contaminated agricultural land during a remediation process," *PLoS One*, vol. 16, no. 7, pp. 1–17, 2021, doi: 10.1371/journal.pone.0255137.

[7] M. Yulia, H. Febriany, and A. E. Kusuma, "Uji Kandungan Timbal (Pb) Pada Jajanan Bakso Tusuk Di Sekitar Aur Kuning Secara Spektrofotometri Serapan Atom," *J. Pharmacopoeia*, Vol. 1, No. 2, pp. 66–74, 2022.

[8] D. Y. Pratiwi, "Dampak Pencemaran Logam Berat (Timbal, Tembaga, Merkuri, Kadmium, Krom) Terhadap Organisme Perairan Dan Kesehatan Manusia," *J. Akuatek*, vol. 1, no. 1, pp. 59–65, 2020.

[9] S. Kucukler *et al.*, "Protective Effects of Chrysin Against Oxidative Stress and Inflammation Induced by Lead Acetate in Rat Kidneys: a Biochemical and Histopathological Approach," *Biol. Trace Elem. Res.*, vol. 199, no. 4, pp. 1501–1514, 2020, doi: 10.1007/s12011-020-02268-8.

[10] Z. Guo *et al.*, "Detection of Heavy Metals in Food and Agricultural Products by Surface-enhanced Raman Spectroscopy," *Food Rev. Int.*, vol. 00, no. 00, pp. 1–22, 2021, doi: 10.1080/87559129.2021.1934005.

[11] A. P. Perdana, E. Sy, and E. Yerizel, "Analisis Kandungan Timbal Pada Gorengan yang Dijual Sekitar Pasar Ulakan Tapakis Padang Pariaman Secara Spektrofotometri Serapan Atom," *J. Kesehat. Andalas*, Vol. 6, No. 3, p. 490, 2017, doi:

10.25077/jka.v6i3.727.

[12] R. R. Umar, J. M. L. Umboh, and R. H. Akili, "Analisis Kandungan Timbal (Pb) pada Makanan Jajanan Gorengan di Pinggiran Jalan Raya Kec. Girian Kota Bitung Tahun 2021," *J. Kesmas*, vol. 10, no. 5, pp. 84–93, 2021, [Online]. Available: https://ejournal.unsrat.ac.id

[13] M. W. TYAS, "Analisis Kadar Timbal (Pb) Pada Bekatul Dengan Destruksi Refluks Secara Spektroskopi Serapan Atom (SSA)," pp. 1–80, 2022.

[14] B. P. O. dan M. BPOM, "Peraturan Badan Pengawas Obat dan Makanan Nomor 5 Tahun 2018 tentang Bahan Baku yang dilarang dalam Pangan Olahan," 2018.

[15] D. J. Butcher, "Innovations and developments in graphite furnace atomic absorption spectrometry (GFAAS)," *Appl. Spectrosc. Rev.*, vol. 0, no. 0, pp. 1–18, 2021, doi: 10.1080/05704928.2021.1919896.

[16] Sugito and S. D. Marliyana, "Uji Performa Spektrofotometer Serapan Atom Thermo Ice 3000 Terhadap Logam Pb Menggunakan CRM 500 dan CRM 697 di UPT Laboratorium Terpadu UNS," *Indones. J. Lab.*, vol. 4, no. 2, pp. 67–71, 2021, doi: 10.22146/ijl.v4i2.67438.

[17] Fajar Nugraha *et al.*, "Analisis Kadar Kalium Ekstrak Kombinasi Kulit Pisang (Musa paradisiaca L.) dan Kulit Nanas (Ananas comosus (L.) Merr) Secara Spektrofotometri Serapan Atom," *J. Sains dan Kesehat.*, vol. 3, no. 6, pp. 846–852, 2021, doi: 10.25026/jsk.v3i6.791.

[18] A. H. Mulyati, Sutanto, and D. Apriyanti, "Validasi Metode Analisis Kadar Ambroksol Hidroklorida Dalam Sediaan Tablet Cystelis Secara Kromatografi Cair Kinerja Tinggi," *Ekologia*, vol. 11, no. 2, pp. 36–45, 2011.

[19] X. Zhou, N. Zheng, C. Su, J. Wang, and H. Soyeurt, "Relationships between Pb, As, Cr, and Cd in individual cows' milk and milk composition and heavy metal contents in water, silage, and soil," *Environ. Pollut.*, vol. 255, pp. 1–7, 2019, doi: 10.1016/j.envpol.2019.113322.