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ANALYSIS OF ACID MIXTURE COMPARISON ON THE PROCESS OF DESTRUCTION OF LEAD METAL (Pb) ON SAMPLE OF CARPET SHELL'S MEAT (*Ruditapes variegatus*)

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ABSTRACT

Analysis of the acid mixture ratio in the lead (Pb) metal deconstruction process in carpet shell's meat samples (Ruditapes variegatus). In the deconstruction agent HNO_3 : HCl (1:3), the average lead metal concentration contained in sweet clam meat is 4.6305 ± 1.066 mg/kg and for the HNO_3 : H_2SO_4 (3:1), the average lead metal concentration contained in the same sweet clam meat was 5.6516 ± 2.556 mg/kg. Statistical test results showed no significant difference in the dissolving power of the acid mixture HNO_3 : HCl (1:3) and HNO_3 acid mixture: H_2SO_4 (3:1) as seen from the p>0.005 value, so both the HNO3 acid mixture: HCl (1:3) and HNO_3 acid mixture: H_2SO_4 (3:1) can be used to deconstruct Pb metal in sweet clam meat (Ruditapes variegatus) because there is no significant difference in the two acid mixtures.

Keywords: Wet Deconstruction, Carpet Shell's, Lead, Statistical Test

ABSTRAK

Analisis perbandingan campuran asam pada proses destruksi logam timbal (Pb) pada sampel daging kerang manis (*Ruditapes variegatus*). Pada zat pendestruksi HNO_3 : HCl (1:3), konsentrasi logam timbal rata-rata yang terkandung dalam daging kerang manis adalah sebesar 4,6305 ± 1,066 mg/kg dan untuk zat pendestruksi HNO_3 :H_2SO_4 (3:1), konsentrasi logam timbal rata-rata yang terkandung dalam daging kerang manis yang sama adalah sebesar 5,6516 ± 2,556 mg/kg. Hasil uji statistik didapatkan tidak ada perbedaan yang signifikan pada kekuatan melarutkan dari campuran asam HNO_3 : HCl (1:3) dan campuran asam HNO_3 : H_2SO_4 (3:1) yang dilihat dari nilai p>0,005, maka baik campuran asam HNO_3 : HCl (1:3) maupun campuran asam HNO_3 : H_2SO_4 (3:1) dapat digunakan untuk mendestruksi logam Pb dalam daging kerang manis (*Ruditapes variegatus*) karena tidak ada perbedaan yang signifikan pada kedua campuran asam tersebut.

Kata kunci : Destruksi Basah, Kerang Manis, Timbal, Uji Statistik

1. Introduction

Maluku is an archipelago province that has abundant marine products, one of which is sweet clam (Ruditapes variegatus). Carpet shell's are marine animals that measure 3-4 cm in size, living by immersing themselves in sandy beach areas and estuary environments. Sweet clams belong to the family Veneridae, class Bivalvia (Poutiers, 1998).

Sea scallops have been utilized for various needs, both economically, ecologically, and other needs. Economically, mussels have been recognized as a food source that is rich in nutrients such as protein (61.74%), carbohydrates (32.64%), fat (14.37%) and several minerals such as N, K, and Mg. Clam shells can also be used for decoration or trinkets. Meanwhile, ecologically, mussels have an important role in an ecosystem and are an integral element of the food chain in the waters. In addition, seashells can also be used as indicators of environmental conditions (Babu et al., 2012; Mikkelsen and Henne, 2011).

Lead (Pb) is one of the most toxic heavy metals. It can be detected in practically all aquatic biological systems. Lead is widely used as an additive in gasoline, is a waste from the industrial sector and coal combustion deposition. Lead is toxic to the nervous system, digestive system, reproduction and affects the kidneys. Symptoms of chronic poisoning with this metal are characterized by nausea, anemia, pain around the abdomen and can cause paralysis. The maximum Pb metal content limit in food set by the Government is 4 ppm, while FAO limits it to 2 ppm (Darmono, 2001).

Carpet shell's is one of the marine biota that can accumulate Pb metal, so if sweet mussels that have accumulated Pb are consumed, it will be harmful to human health. Considering the impact of Pb metal pollution is increasing along with the increase in human activity, it is very necessary to

determine the content of Pb metal in sweet mussels. One of the instruments that can be used for quantitative analysis of Pb metal is atomic absorption spectrophotometer (AAS).

The preparation of a sample determines the success of the analysis in atomic absorption spectrophotometry. Sample preparation is done through ignition, namely dry deconstruction or wet deconstruction. Shabbering with dry deconstruction is carried out at high temperatures. The ignition temperature for each material is different, depending on the components present in the material (Anderson, 1987).

Sample preparation by the wet destruction method is carried out at low temperatures and with the addition of a mixture of strong acids to destroy organic compounds and other materials in the sample. The wet deconstruction method is more often used for the analysis of volatile samples. The advantage with this analysis method is that the time and process are faster. It's just that with this wet deconstruction method the possibility of error is greater due to the use of more reagents and in the process requires extra attention from the analyst because in its implementation the reaction that occurs takes place strongly and can make the residue come out, so during heating it must be more careful (Rohman, 2007).

2. Methodology

2.1. Materials and Instrumentals

The tools used include aset of glassware (Pyrex), Spatula, knife, blender (miyako), pH meter (Orion mode 710A), Analytical balance (Ohaus Analytical Plus), Atomic Absorption Spectrophotometer (iCe 3000).The materials used include: carpet shell's meat, HCl (E. Merck), HNO_3 (E. Merck), H_2SO_4 (E. Merck), 1000 ppm Pb mother liquor, distilled water, Whatman filter paper (No.42)

2.2. Methods

Sample preparation

Carped shell;s samples were cleaned and removed from their shells, It was then homogenized using a blender.

Wet deconstruction process

A total of 5 g of sample was put into a 250 mL Erlenmeyer and then added a mixture of 10.6 M HNO_3 acid: 10.6 M HCl (1:3) as much as 30 mL, heated on a hot plate until the gas disappears and the solution is clear

On a hot plate until the gas disappeared and the solution was clear. The same treatment was carried out for 10.6 M HNO_3: 6.8 M H_2SO_4 (1:3) after it was cooled and then filtered. The filtrate was diluted with distilled water in a 50 mL measuring flask and made pH 3 and measured the absorbance with AAS at λ 283.3 nm and treated 3 times.

Preparation of lead standard solution

The 1000 ppm lead standard solution was pipetted as much as 10 mL and then put into a 100 mL volumetric flask. The solution was diluted with HNO_3 0.1 N until the limit line then shaken until homogeneous so that a solution with a concentration of 100 ppm is obtained. From the 100 ppm solution, 10 mL was pipetted and then put into a 100 mL volumetric flask and the solution was diluted with 0.1 N HNO_3 to the limit line and then shaken until homogeneous, so that a solution with a concentration of 10 ppm was obtained. From the 10 ppm solution, 5, 10, 15, and 20 mL were pipetted respectively. Each solution was put into 4 different 100 mL volumetric flasks, then diluted with 0.1 N HNO_3 to the limit line and shaken until homogeneous so that a solution with a concentration of 0.5; 1.0; 1.5; and 2.0 ppm was obtained.

Analysis by AAS

The blank solution was introduced into the AAS, then the standard solution was successively introduced into the SSA according to the concentration increment and the absorbance values were recorded.

Data analysis

Based on the measurement results, a graph is made to obtain a straight line on the graph between absorbance and concentration using the linear regression line equation, namely:

$$Y = a + bx$$

Description:
x = Concentration
Y = Absorbance
a = Constant Price
b = Regression Coefficient

3. Result and Discussion

3.1. Determination of Lead (Pb) Metal in Carpet Shell's Meat (*Ruditapes variegatus*)

One of the requirements for metal analysis using Atomic Absorption Spectrophotometry (AAS) is that the sample must be a solution. Sample preparation is an important step in the analysis of micro elements using AAS measurements.

Analysis using AAS can be said to be successful if the selection of the right decomposition method. One of the best methods in metal analysis is the deconstruction method. The deconstruction process needs to be done before analyzing an element in food ingredients, because it greatly affects the results that will be obtained. There are two procedures commonly used to deconstruct organic materials in snippets, namely wet deconstruction and dry deconstruction. The function of deconstruction is to break the bond between organic compounds and metals to be analyzed (Mardiyono, 2009).

Wet deconstruction is used because in general wet deconstruction can be used to determine elements with low concentrations. After the deconstruction process is expected to be left only metals in the form of ions. Solvents that can be used for wet deconstruction include nitric acid, sulfuric acid, perchloric acid, and hydrochloric acid. Of all these solvents can be used either single or mixed. For this reason, the analysis of Pb metal in sweet mussel meat was carried out by comparing different acid mixtures to obtain a wet deconstruction method.

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In analyzing the concentration of a metal in a sample, it turns out that all elements or components in this case that do not want to be observed can cause an increase or decrease in the concentration of the metal to be analyzed, for this reason it is necessary to dilute the sample solution to reduce the concentration of unwanted metals (Dewi, 2012). Dilution of the sample solution was carried out using distilled water and then quantitatively analyzed with AAS.

3.2. Comparative Analysis of Acid Mixtures in Wet Destrusion of Lead (Pb) Metal

Analysis of the ratio of acid mixture to wet destrusion of lead (Pb) metal in sweet mussel meat was carried out using the calibration curve method. In this method, a series of standard solutions with various concentrations were made and the absorbance of the solution was then measured by AAS. Measurement of absorbance as a function of standard solution concentration (Appendix 1). Figure 1 shows the calibration curve of Pb standard solution measurement. The absorbance measurement of Pb standard solution as a function of concentration is shown by the curve in Figure 1. The function of concentration is shown by the regression coefficient which is close to 1 (0.9983).

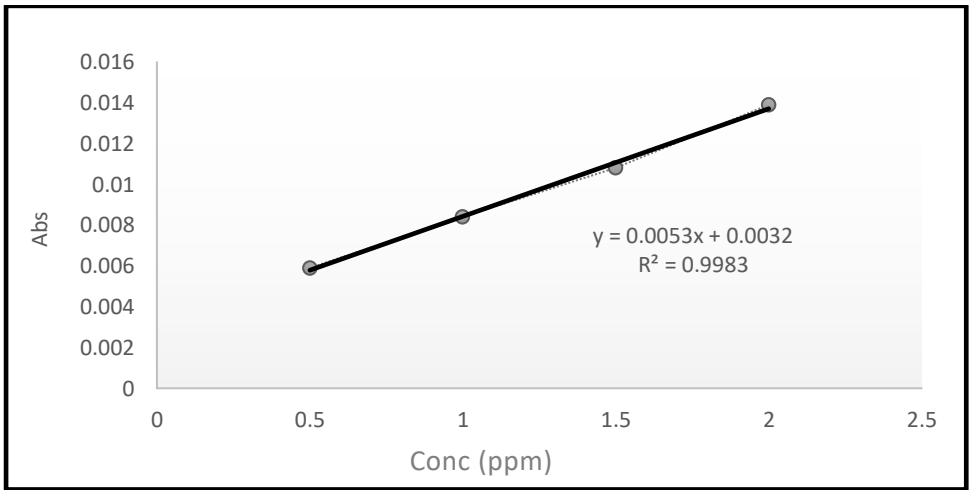

Figure 1. Lead (Pb) metal standard solution curve

The destructive substances used were a mixture of HNO_3 and HCl (1: 3) and a mixture of HNO_3 and H_2SO_4 (3: 1). The addition of each acid has its own purpose. HNO_3 is used as the main oxidizing agent because HNO_3 is a good metal solvent, Pb is oxidized by HNO_3 so that it becomes soluble and

serves to break the bonds of complex compounds, where metals contained in the snippet form complex compounds with organic matter (Setyaningrum and Sukesi, 2013). Lead is easily soluble in HNO_3 and will form nitrogen oxides (Svehla, 1990). The purpose of the variation of the destructive agent in this study is to determine the most effective destructive agent in the analysis of Pb metal in carpet shell's meat samples.

The addition of other acids such as H_2SO_4 and HCl is a catalyst to accelerate the reaction of Pb disconnection from organic compounds in the carpet shell's meat sample. The type of catalyst used here is a catalyst that affects the environment so that this catalyst does not react. According to Dewi (2012), the reaction that occurs in the sample solution when adding an acid mixture is as follows acid mixture is as follows:
Reaction for HNO_3 and HCl oxidizers:

$$3Pb_{(s)} + 8HNO_{3(aq)} + HCl_{(aq)} \longrightarrow 3Pb^{2+}{}_{(aq)} + 6NO_3{}^{-}{}_{(aq)} + 2NO_{(g)} + 4H_2O_{(l)} + H^{+} + Cl^{-}{}_{(aq)}$$

(Reaction for oxidizing HNO_3 and H_2SO_4:

$$3Pb_{(s)} + 8HNO_{3(aq)} + H_2SO_{4(aq)} \longrightarrow 3Pb^{2+}{}_{(aq)} + 6NO_3{}^{-}{}_{(aq)} + 2NO_{(g)} + 4H_2O_{(l)} + 2H^{+}{}_{(aq)} + SO_4{}^{2-}{}_{(aq)}$$

Wet deconstruction is carried out by decomposing organic matter in concentrated and hot oxidizing acid solutions such as H_2SO_4, HNO_3, and HCl by heating until the volume of the solution is reduced by half, heating until the volume of the solution is reduced by about half. Minerals are in the form of metal cations and chemical bonds with organic compounds have been broken down. To break the covalent bonds between carbohydrates, proteins, and fats with Pb metal, proper destructing must be done with oxidizing agents that are able to clear the solution and are able to maintain the stability of Pb metal because it is undeniable that there are compounds that are insoluble in concentrated nitric acid alone (Dewi, 2012).The results of the analysis of the acid mixture in the Pb metal destruction process contained in carpet shell's meat are shown in Table 1.

Table 1. Pb Metal Levels with Wet Destruction

Deconstructin agent	Pb levels (mg/kg)				
	1	2	3	Average	
HNO_3 : HCl	3,5430	4,6751	5,6734	4,6305	± 1,066
HNO_3 : H_2SO_4	5,2648	3,3108	8,3791	5,6516	± 2,556

Based on Table 1, it can be seen that the concentration of Pb metal in carpet shell's meat with deconstruction using a mixture of HNO_3 acid: HCl (1:3) in each repetition ranged from 3.5430-5.6734 mg/kg with an average concentration of 4.6305 mg/kg, while the concentration of Pb metal by deconstruction using a mixture of HNO_3 acid: H_2SO_4 (3:1) ranged from 3.3108 to 8.3791mg/kg with an average concentration of 5.6516 mg/kg. This same thing can also be seen in Figure 2.

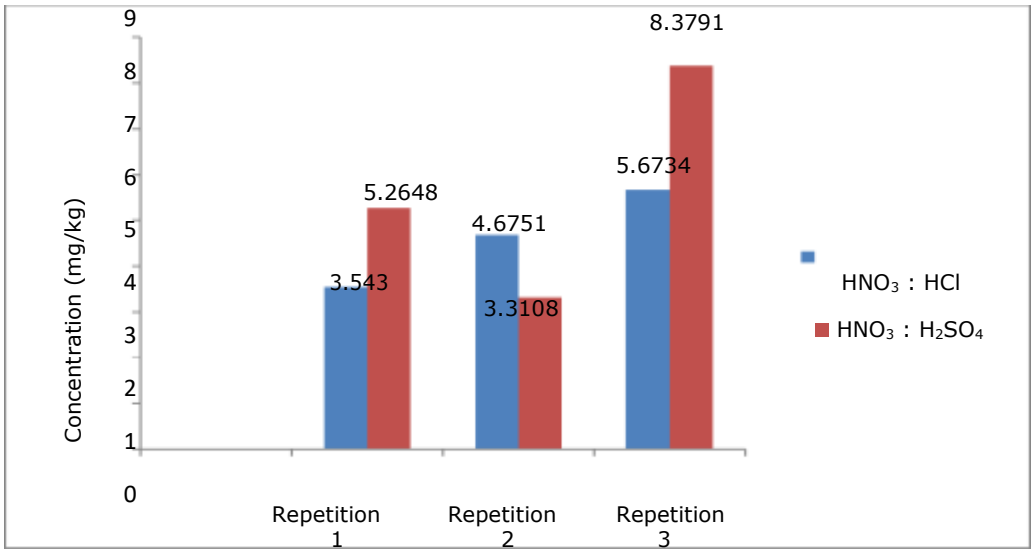

Figure 2. Diagram of Pb concentration in sweet clam meat

From the acid mixture between HNO_3: HCl and HNO_3: H_2SO_4 used in destructing, it was found that the acid mixture between HNO_3 and H_2SO_4 (3:1) was stronger than the acid mixture between HNO_3 and HCl (1:3) in destroying Pb metal as a free metal cation. However, deconstruction with an acid mixture of HNO_3 and H_2SO_4 (3:1) takes longer to dissolve completely or the solution becomes clear yellowish orange, compared to the acid mixture of HNO_3 and HCl (1:3). A mixture of HNO_3 and H_2SO_4 acids has also been used by Dewi (2012) in destroying Pb metal in canned lychee samples. The results showed that the content of Pb in canned lychee amounted to 0.72 ppm or 36mg/kg sample.

When viewed from the standard deviation, the acid mixture of HNO_3 and HCl (1:3) has a smaller standard deviation than the acid mixture of HNO_3 and H_2SO_4 (3:1). In statistics, standard deviation is a measure used to measure the amount of variation or distribution of a number of data values. The lower the standard deviation, the closer to the average, while if the standard deviation value is higher, the wider the range of data variation. So the standard deviation is the amount of difference from the sample value to the average (Hasan, 2011).

For this reason, it can be said that the acid mixture of HNO_3 and HCl (1:3) is a better acid mixture in destroying Pb metal because the standard deviation is close to the average Pb metal concentration. This is because acid strength (especially if following the Bronsted-Lowry acid definition) is determined based on the level of ionization of the acid molecule to release protons. Quantitatively this is expressed in Ka or pKa values (Mulyono, 2005) (Table 2).

Table 2. Values (Ka) of Some Acids

Acid	Ka
HNO_3	2.0×10^1
HCl	1.0×10^4
H_2SO_4	1.0×10^3

Table 2. shows that the Ka value of hydrochloric acid is greater than the Ka value of sulfuric acid sulfuric acid, the greater the Ka value of an acid, the greater the strength of the acid.

Statistical test results, showed that there was no significant difference in the dissolving power of the acid mixture HNO_3:HCl (1:3) and HNO_3:H_2SO_4 (3:1) as seen from the $p>0.005$ value. From statistical test results, it can be concluded that both the acid mixture of HNO_3: HCl (1:3) and HNO_3 acid mixture: H_2SO_4 (3:1) can be used to deconstruct Pb metal in carpet shell's meat because there is no significant difference in the two acid mixtures.

4. Conclusion

The results of data analysis of Pb metal in carpet shell's meat samples (Ruditapes variegatus) can be concluded that both:

1. The mixture of HNO_3 acid: HCl (1:3) and HNO_3 acid mixture:H_2SO_4 (3:1) can used to deconstruct Pb metal in sweet clam meat because there is no significant difference in the two acid mixtures.
2. The average concentration of Pb metal with a mixture of HNO_3: HCl (1:3) and HNO_3: H_2SO_4 (3:1) were 4.6305 ± 1.066 mg/kg and 5.6516 ± 2.066 mg/kg, respectively. and 5.6516 ± 2.556 mg/kg.

References

Anderson, R., 1987. *Sample Pretreatment and Separation*. New York: John Willey & Sons.

Babu, A., Venkatesan. V., dan Rajagopal, S., 2012. Biochemical Composition of Different Body Parts of Gafrarium Tumidum from Mandapan, South East Coast of India. *African J Biotech* 111 (7): 1700–1704.

Darmono, 2001. *Lingkungan Hidup dan Pencemaran*. Jakarta: UI Press.

Dewi, D. C., 2012. Determinasi Kadar Logam Timbal (Pb) dalam Makanan Kaleng Menggunakan Destruksi Basah dan Destruksi Kering. Vol. 2, No. 1, Hal: 12–25 .

Hasan, I., 2011. *Analisis Data Penelitian Statistik*. Bumi Aksara, Jakarta.

Hidayati, E. N., 2013. Perbandingan Metode Destruksi Pada Analisis Pb dalam Rambut dengan AAS. *Skripsi*. Jurusan Kimia, Fakultas Matematika dan Ilmu Pengetahuan Alam Universitas Negeri Semarang.

Mardiyono., 2009. *Analisis Cu & Pb pada beberapa Produk Sayur Kacang-kacangan dalam kaleng Secara SSA*. Fakultas Farmasi Univer

Rohman, A., 2007. *Analisis Makanan*. Yogyakarta: UGM

Svehla, G. 1990. *Buku Teks Analisi Anorganik Kualitatif Makro dan Semimikro*, terjemahan Setiono dan Pudjaamatka. Jakarta: Kalman Media Pustaka.

Palar, H., 2008. *Pencemaran dan Toksilogi Logam Berat*. Penerbit: Rineka Cipta. Jakarta.

Poutiers, J. M., 1998*. Bivalves (A Cephala Lameliibranchia, Pelecypoda). In: Carpenter KE, Niem VH (eds).* FAO Species Identification Guide for Fishery Purposes. Living Marine Resources of the Western Central Pacific 1. Seaweeds, Corals, Bivalves and Gastropods*.* FAO,Rome