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Lead (Pb) and cadmium (Cd) analysis in PVC water pipes using micro wave destruction by atomic absorption spectrophotometry

Magdalena[1*], Meytij Jeanne Rampe[2], Tiene MB Turangan[3]
[1] Student of Master Program, Study Program of Chemistry, Postgraduate Program, Manado State University, Indonesia
[2, 3] Postgraduate Program, Manado State University, Indonesia

Abstract
This study aims to measure the concentration of lead metal (Pb) and cadmium (Cd) in PVC-based water pipe samples with Atomic Absorption Spectrophotometers (AAS) using the MPS Titan 100 microwave preparation system with acid combinations that are varied as destructors. Variations in acid combinations consist of nitric acid (HNO_3), phosphoric acid (H_3PO_4), sulfuric acid (H_2SO_4), and fluoride acid (HF) with a total volume of 10 mL. The optimal acid combination for the destruction of PVC water pipe samples has the composition of HNO_3: H_3PO_4: H_2SO_4: HF = 4: 3: 3: 0 (mL). By using Atomic Absorption Spectrophotometry, the concentration of lead metal analyte (Pb) in a Wavin (6'') PVC sample was 33.8082 ppm and Power (2'') was 9.2151 ppm. For cadmium metal (Cd) in Wavin (6 '') and Power (2'') PVC samples respectively 0.4019 ppm and 0.4185 ppm.

Keywords: acid combination, PVC water pipe, microwave destruction, AAS

1. Introduction
The use of Polyvinyl Chlorida (PVC) in the industrial sector is in high demand, regardless of problems and environmental pollution, it is used as material for making pipes, packaging, electronics, and building construction materials. PVC is a polymer with beneficial basic properties, such as non-flammable, inexpensive, and flexible. In the PVC manufacturing industry, cadmium heavy metal is often used as a stabilizer additive and lead as a plasticizer in the manufacture of water pipes so that it is resistant to corrosion, so the presence of heavy metals that surround the water pipes in its manufacture can allow contact between household water and the metal.
According to Mukono (2002) [1] and Naria (2005) [2] in the United States it was found that lead levels in drinking water reached 50 µg/l caused by the use of tendons and lead-plated drinking water pipes [1,2]. Water channeled through lead pipes contains high levels of dissolved lead in the water [3]. The main source of lead and cadmium contamination in PVC water pipes is additives which is a dangerous category. Tolerable Weekly Intake for Pb is 25 µg/kg body weight, this value refers to Pb which comes from all sources of pollution and applies to all ages. Meanwhile, for the concentration of Pb in the blood, WHO recommends levels of 20 µg/dl for all populations. Additives that are widely used in making Polivinyl chloride water pipes in the form of dyes, fillers, plasticizers from pthalat and stabilizer groups from heavy metals such as Cadmium Stearate and Lead Chromates [4] and when the cadmium content reaches 200 µg/g in the renal cortex will have an impact on kidney failure [5].
Analysis of heavy metal content in laboratories is mostly done using atomic absorption spectroscopy, this method requires the process of sample destruction to produce analytes which are ready to be analyzed. There are a number of commonly used sample destruction methods such as open acid destruction and closed acid destruction. The purpose of the closed acid destruction process is to obtain a solution that is perfectly mixed with the analyte, complete decomposition of the solid and avoid analytic loss or contamination while the open acid destruction method is a mixture of test samples with strong acid reagents heated openly on an electric bath. The advantage of using an open acid digestion method is that the equipment used is relatively simple and inexpensive, while the disadvantage is that volatile elements from the test sample can be lost during the destruction process so that errors in the results of the analysis are possible. The possibility of contamination from the air, and the destruction time is longer than 12 hours [6].
The method used to correct the weaknesses of the method of open acid destruction is the method of closed acid destruction, namely with the help of microwaves in the process of destruction. This method is called the microwave digestion method. In this method the test sample is added with strong acids in a closed system which causes an increase in temperature and pressure. Increases in temperature and pressure as well as conditions in low pH in the test sample cause an increase in the rate of thermal decomposition of the test sample which makes the metal dissolve. After the metal dissolves, then it is possible to do measurements with the instrument [7].
In this study the preparation of PVC water pipe samples was carried out using the microwave destruction method and using a combination of varied acids. The microwave destruction procedure developed is very practical, and is categorized as a preparation method that is easy, fast, accurate, thorough, reliable, and is used as a routine analysis method in laboratories with several types of samples in large quantities. As for the measurement of Lead (Pb) and Cadmium (Cd) heavy metals using Atomic Absorption Spectrophotometry (AAS). This method is suitable for use

in laboratories that analyze metals in many samples. Because it can be tested directly on Atomic Absorption Spectrophotometry [8].

The advantage of atomic absorption spectrophotometry method is that it is more specific in determining the specific metal content in each sample, the low detection limit of the same solution can measure different elements (ppm 10^{-6} and ppb -9) with direct readable output, quite economical, can be applied to many types of elements and the extent of determining the extent of area. In addition, the method is more efficient and effective, it has analytical capacity of both absorption and atomic emission on several matrix parameters such as sensitivity, accuracy, and detection limits. Meanwhile the weaknesses of the spectrophotometric method are chemical influence, ionization effect and matrix effect [9].

2. Research Methods

Tools and materials

The equipment used in this study was the Perkin Elmer PinAAcle 900F Atomic Absorption Spectrophotometer (SSA), Perkin Elmer Microwave Titan MPS, Centrifuge 5810 Eppendorf, micro pipettes of 10 -100 µL, 100-1000 µL and 50 -250 µL, bomb teflon digester, plastic bottles for samples, 50 ml, 25 ml, 10 ml measuring flasks and other laboratory glassware.

The materials used are nitric acid (HNO_3) 65% for analysis EMSURE (Merck), sulfuric acid H_2SO_4 (95%), HF (40%) and Orto Phosphoric Acid H_3PO_4 (85%), aquades (water one), standard solution Pb (II) (Merck-SRM) 1000 mg/L, Cd (II) standard (Merck-SRM) 1000 mg / L. The sample in this study is a water pipe made from PVC or PVC paralon with Wavin brand size of 6 inches and Power size of 2 inches with a length of 4 meters each obtained from a building material store in Tondano City, Minahasa Regency, North Sulawesi.

3. Research Procedure

The sample is cleaned and cut in a circle at one end approximately 2 cm. Pieces of PVC water pipes are cleaned with soap and put in 1 L beaker then rinse with distilled water. Then rinse again with dilute nitric acid and dry. Next, it is dredged using glass to get a smooth and homogeneous sample to be ready for destruction. After that, weigh the sample PVC water pipe 0.1000 g ± 0.0001 g in a Teflon container and put it into the vessel and add 1 mL of $HN0_3$ (65%) concentrated and 4 mL of Sulfuric Acid (H_2SO_4 98%) and 4 mL of H_3PO_4 (85%) as well as 1 mL HF (40%) in the acid chamber. Samples in vessels that have been added to acid combinations are allowed to stand for 15 minutes to react the samples with acids. Next, it is closed and placed in a microwave digestion. Then the digestion program is set, according to the Perkin Elmer Microwave MPS Titan program.

Samples were heated to 170ºC ramp time 2 minutes and holding time 5 minutes at a pressure of 30 bar with a total heating time of 47 minutes. After completion, the vessel is allowed to cool for at least 5 minutes before being removed from the microwave. Inside the acid chamber, each vessel is opened, the sample is transferred to a 25 mL volumetric flask squeeze to the limit, then homogeneous. Subsequently the aqueous sample solution was determined using Atomic Absorption Spectrophotometry.

4. Results and Discussion

The acid used will dissolve the sample to produce a solution containing analytes, namely Pb and Cd. The reaction between the analyte in the sample and the acids used include:

1. Pb reaction with acids

$$3Pb + 8HNO_3 \longrightarrow 3Pb^{2+} + 6NO_3^- + 2NO + 4H_2O$$

The reaction between Pb and nitric acid produces Pb^{2+} ions, colorless NO gases and water. Furthermore, Pb^{2+} ions react with other acids.

Reaction with sulfuric acid

$$Pb^{2+} + SO_4^{2-} \longrightarrow PbSO_4$$

This reaction produces $PbSO_4$ deposits and will dissolve when heated to produce Pb^{2+} ions.

$$PbSO_4 + H_2SO_4 \longrightarrow Pb^{2+} + 2HSO_4^-$$

2. Cd reaction with acids

$$Cd + 2H^+ \longrightarrow Cd^{2+} + H_2$$

The reaction of Cd with acids produces colorless bivalent ions. Cadmium nitrate and sulfate can dissolve in water.

Table 1

Samples		HNO_3 (ml)	H_3PO_4 (ml)	H_2SO_4 (ml)	HF (ml)	Result
Wavin 6’	A	4	3	3	0	dissolved
	B	4	3	3	0	dissolved
	C	4	3	3	0	dissolved
Power 2’	A	4	3	3	0	dissolved
	B	4	3	3	0	dissolved
	C	4	3	3	0	dissolved

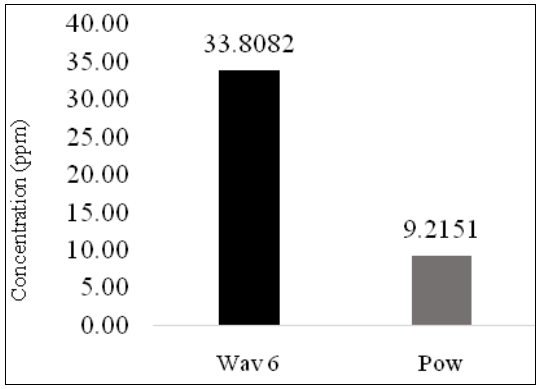

Fig 1: Pb Concentration Diagram in PVC Pipe Samples

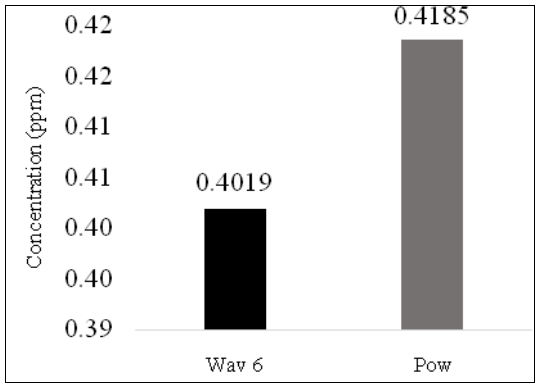

Fig 2: Cd Concentration Diagram in PVC Pipe Samples

From the results of metal analysis, which results in high absorbance, the optimal acid combination can be determined. In general, method validation includes determinations related to tools and methods so that in sample measurements it is shown that, although it is said to be a lead-free PVC water pipe, the lead concentration in the pipe is not necessarily 0 ppm. In addition, the concentration of cadmium metal, detected at concentrations that are not too high.

Conclusion

Pb metal concentration in Wavin PVC water pipe is greater than PVC Power (lead free), which is 33.8082 ppm for PVC Wavin and 9.2151 ppm for PVC Power. Although classified as lead-free pipe, the results of the analysis show that the PVC Power pipe still contains Pb metal. While the concentration of Cadmium (Cd) in PVC Power pipe is 0.4185 ppm higher than the concentration of Cadmium (Cd) in PVC Wavin pipe of 0.4019 ppm.

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