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## Determination Of Trace Levels Of Pb(II), Cd(II), And Cu(II) In Tilapia Nilotica, Water Hyacinth, And Sediment Samples At Maninjau Lake.

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### ABSTRACT

Maninjau Lake has decreased water quality for their activities outside and inside the lake which can cause pollution, one of which is water pollution by heavy metals. This study aims to find the heavy metal content in tilapia nilotica, water hyacinth, and sediment in the fish cage culture area at Maninjau Lake using Flame Atomic Absorption Spectrophotometer (FAAS). The results showed: (1) Tilapia nilotica has a Pb(II) concentration of 0.6 - 4.3 mg/kg and Cu(II) 0.7 - 5.6 mg/kg; (2) Water hyacinth has a Pb(II) concentrations of 3.1 - 7.6 mg/kg and Cu(II) 7.1 - 18.5 mg/kg; and (3) Sediment has a Pb(II) concentrations of 6.6 - 11.9 mg/kg and Cu(II) 10.6 - 46.1 mg/kg. The concentration of Cd(II) in all samples was not detected, while Pb(II) and Cu(II) has passed the maximum threshold.

**Keywords:** Heavy metals, tilapia nilotica, water hyacinth, and sediment, FAAS

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## INTRODUCTION

As science and technology to enhance the environmental pollution of water, soil, and air. In aquatic systems, heavy metals had been considerably paid attention due to their toxicity and accumulation in biota. Heavy metals generally flow into the aquatic environment through atmospheric deposition, erosion of the geological matrix, and it was due to anthropogenic activities the caused by industrial effluents, domestic sewage, mining wastes, motorization, urbanization by agricultural waste, population density, and industrial activities and by waste net cages.

Water pollution caused by the impact of industrial development should be controlled because if not done earlier will pose serious problems for the survival of humans and the natural surroundings. The need to be done in the control and monitoring of environmental impacts is to analyze elements in tilapia nilotica, water hyacinth, and sediment especially Pb(II), Cd(II) and Cu(II). Contamination of these metals can influence and cause disease in consumers because in the body will experience excessive elements that endanger human detoxification.

Heavy metals generally are toxic to living organisms, although some are needed in small quantities. Through various intermediaries, such as air, food, or water contaminated by heavy metals, these metals can be distributed to parts of the human body and some will be accumulated. If this situation continues, in the long term can reach the amount of harm to human health.

The presence of heavy metals in waters will be difficult to be degraded, even these metals are absorbed in the body of the organism such as the water hyacinth. Water hyacinth (*Eichhornia crassipes*) is one of the plants that have the ability as a biofilter. With the micro bio at the root rhizosphere and supported by the power absorption and accumulation of large specific pollutant exposure, it can be used as an alternative to pollution control in the waters [1].

Heavy metals also can be distributed and accumulated in the human body through the air, food, and water contaminated with heavy metals. Lead, copper, and cadmium, including categories of heavy metals that have a high toxic. The damage caused by the heavy metal is damage to the nerves, kidneys, gastrointestinal tract, skin irritation, skin cancer and other damage [2].

Tilapia nilotica and water hyacinth are used as an indicator of the level of pollution in the waters industrial waste disposal around which the tilapia nilotica and the water hyacinth, such as rivers, lakes, and sea. A number of heavy metals are absorbed and distributed in tilapia nilotica and water hyacinth depends on the of Maninjau Lake. If in the body of tilapia nilotica and water hyacinth contained high levels of heavy metals and beyond normal limits can be predetermined as an indicator of pollution in the environment. According to [3] heavy metal content in tilapia nilotica and water hyacinth closely related to form of compounds and concentrations of pollutants, the activity of microorganisms, sediment texture, as well as the types and elements of fish that live in the neighborhood.

From 1998 to 2016, water color Maninjau Lake already showing a color change from clear to green dense. These changes are thought to be one indicator of disturbance to the ecosystem of the lake as well as the ability to degrade the incoming waste increasingly limited so monitoring the water quality of Maninjau Lake needs to be done in an effort to preserve these waters. Currently, there are on Maninjau Lake tilapia nilotica cage culture of 20,000 units [4-5]. This amount has passed the carrying capacity was estimated 6,000 units, as the result, a mass tilapia nilotica killed happened every year. In Koto Malintang, the cages contained the largest gain in this place. Utilization of the lake for tilapia nilotica through cage culture to trigger all the problems of the quality of the environment around the floating cages, Maninjau Lake [6-7].

We have examined the individual and simultaneous determination of trace metals Cd, Cu, Pb, Zn, Fe, Co, Ni, and Cr by Adsorptive Stripping Voltammetry on a hanging mercury drop electrode (HMDE) [8-19]. In this study, we determine the levels of heavy metals Pb(II), Cd(II) and Cu(II). on tilapia nilotica, water hyacinth and sediment using FAAS. Selection FAAS method because it has a high sensitivity, easy, cheap, simple, fast, and samples needed little.

## MATERIALS AND METHODS

### Chemicals, equipment, and instrumentation

Materials used: tilapia nilotica, water hyacinth, and sediments from Maninjau Lake, nitric acid (HNO<sub>3</sub>) 65%, acid peroxide (H<sub>2</sub>O<sub>2</sub>) 20%, the filter paper, and doubly distilled water. Standard solution of Pb(II), Cu(II) and Cd(II) 1000 mg/L.

Tools used: Equipment: FAAS Varian Spectra AA - 240 spectrometers, lamp hollow cathode Pb(II), Cu(II), and Cd(II), analytical balance, oven, muffle furnace, a desiccator, plastic bottles, mortar and glassware.

### Research procedure

#### *Sampling procedures and analysis*

Samples were taken 3 times at a distance of 50 meters respectively by 4 locations. Hyacinth took as many as the 1-2 clump at each sampling location. Samples were stored in plastic polyethylene (PE). Sediments took as many as 500 g by using a shovel and put in a plastic bag of polyethylene. The tilapia nilotica used in this study is ready to harvest tilapia farmed in tilapia nilotica cage culture. Tilapia nilotica samples have an average weight of 250 g and the average nilotica length 21 cm. Tilapia nilotica to be incorporated into polyethylene plastic bags is further cooled with ice and stored before analyzed in the laboratory. Measurements carried out in the laboratory ex-situ includes the determination of Pb(II), Cu(II) and Cd(II) on tilapia nilotica, water hyacinth, and sediment using FAAS.

#### *Samples Destruction Process Tilapia Nilotica*

Samples tilapia nilotica meat, put in porcelain dried and weighed  $\pm$  10 grams by using the analytical balance, then dried in an oven at 105°C for 24 hours and cooled in a desiccator. Then weighed again to determine water content. The dried tilapia nilotica samples crushed to the powder using a mortar. Samples of tilapia nilotica that have been delicately inserted into the furnace at a temperature of 500°C for 16 hours, then cooled in a desiccator. Samples that have been reduced to ashes, added 5 mL HNO<sub>3</sub> 65%. Furthermore, the sample solution is heated on a hot plate at a temperature of 60-70°C until a clear solution was nearly dry. Subsequently cooled to room temperature and add 1 mL HNO<sub>3</sub> 65%, after it is filtered with filter paper Whatman and added to 10 mL volumetric flask, add distilled water up to the mark. Samples were prepared measured using FAAS.

#### *Samples Destruction Process Water Hyacinth*

Samples hyacinth cut into small pieces and blend, then put in a dry and weighed porcelain  $\pm$  10 g using an analytical balance. Then dried in an oven at 105°C for 24 hours. Then the sample is cooled in a desiccator. Then weighed again to determine its mass water content. Samples were crushed into powder using a mortar. Samples that have been finely injected into the furnace at a temperature of 650° C for 8 hours. Samples that have been reduced to ashes, added 5 mL HNO<sub>3</sub> 65%. Furthermore, the sample solution is heated on a hot plate at a temperature of 60-70°C until the solution is clear and almost dry. Then cooled to room temperature and add 1 mL HNO<sub>3</sub> 65% and stirring gently. After that filtered with Whatman filter paper and put in a 10 mL volumetric flask, add distilled water to the mark. Samples were prepared measured using FAAS.

#### *Samples Destruction Process Sediment*

The sediment samples which have been smoothed with mortar, put in a porcelain dry and weighed  $\pm$  1 gram using analytical balance, then dried in an oven at 105°C for 24 hours and cooled in a desiccator. Then weighed again to determine water content. Samples were dried put in the kjehdahl flask then added 10 mL HNO<sub>3</sub> 65%. Pumpkin heated over a hot plate while putting 2 mL of H<sub>2</sub>O<sub>2</sub> 20% drop by drop until the solution is clear and nearly colorless dry, then cooled to room temperature and added 1 mL HNO<sub>3</sub>. After it is filtered with filter paper Whatman and diluted in 10 ml flask. Samples were prepared measured using FAAS.

RESULTS AND DISCUSSIONS

The Concentration Pb(II), Cu(II) and Cd(II) Samples in the First Week.

The content of heavy metals Pb(II), Cu(II) and Cd(II) on tilapia nilotica, water hyacinth and sediment in the 4th locations at first week can be seen in Figure 1.

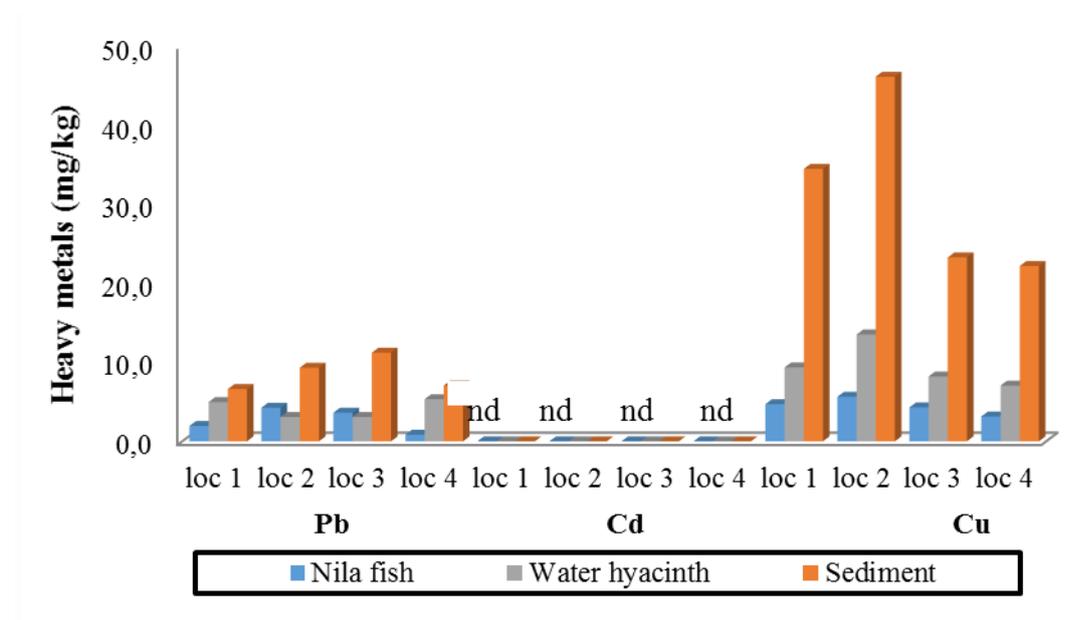


Figure 1. Concentrations of Pb(II), Cu(II) and Cd(II) in tilapia nilotica, water hyacinth and sediment in the first week (mg/kg)

Legend :  
 nd: not detected  
 Loc: location

At the first week, concentration of Pb(II) in tilapia 0.8 - 4.2 mg/kg, in water hyacinth 3.1 - 5.3 mg/kg, and in sediment 6.6 - 11.2 mg/kg. While the concentration of Cd(II) were not detected in all samples because Cd(II) very small, below the detection limit of the FAAS, and a source of Cd(II) metal is very little that goes into the lake. Concentration of Cd(II) usually sourced from the plastics industry and electroplating [8]. The concentration of Cu(II) at the first week in tilapia nilotica 3.1 - 5.6 mg/kg, in water hyacinth 7.1 - 13.5 mg/kg, and in sediment 22.2 - 46.1 mg/kg. Therefore the concentration of Cu(II) has a higher concentration than the concentration of Pb(II) and Cd(II).

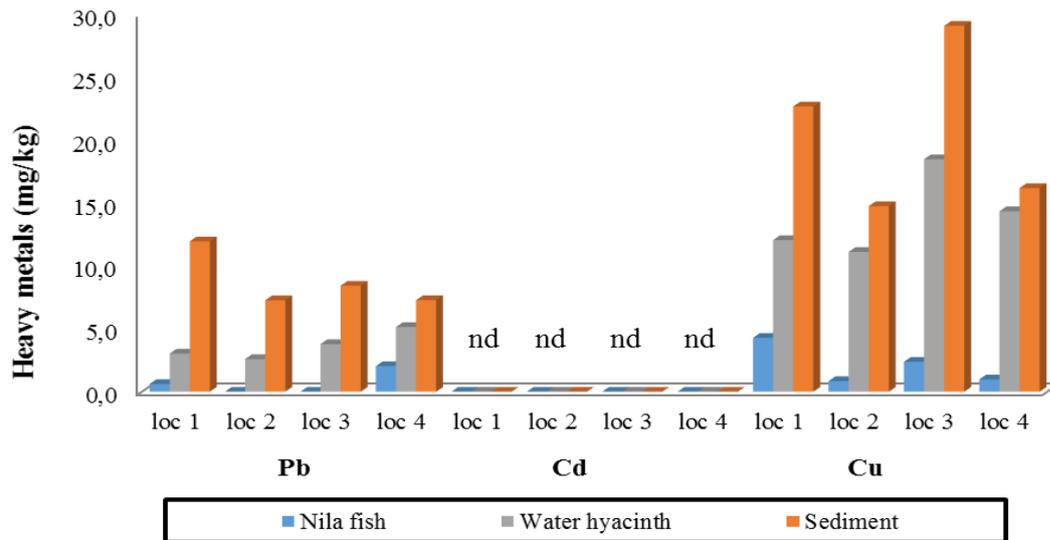
Figure 1, the concentration of metals in samples of tilapia nilotica, water hyacinth and sediments have different concentrations. High and low concentrations of metals contained in the sample is highly dependent on the availability of these metals in an environment. The ability to accumulate a sample in pollutants can also affect, the more waste into the waters, the greater the concentration of pollutants in the water. This allows the metal absorption by the sample metal gradually accumulates in the sample.

Concentration of Cu(II) metal content in the sediment samples was found at the highest location of 2 weeks 0, ie 46.1 mg/kg. Cu(II) natural source derived from erosion events (erosion) mineral rocks, dust and particles in the Cu(II) layer of air, while the non-naturally derived from human activities, among others from the shipbuilding industry, the wood processing industry as well as household waste [20]. Cu(II) metal content in the Maninjau Lake is possible because of the erosion of mineral rocks, household waste, and excessive feeding tilapia nilotica. Excessive feeding can cause the accumulation of food remains on the bottom of the lake and tilapia nilotica feed containing Cu(II) so that the sediments of Maninjau Lake containing Cu(II) [4-5]. Judging from Figure 1 can be seen the results of the concentration of Cu(II) contained in the sediment when

compared to [21] Indonesian Government Regulation No. 82 of 2001, the concentration of Cu(II) were obtained at weeks 0 is above natural levels of sediment should limit is 5-30 mg/kg [22].

**The concentration of Pb (II), Cu (II) and Cd (II) samples of Week – 3**

The content of heavy levels Pb(II), Cu(II) and Cd(II) on tilapia nilotica, water hyacinth and sediment in the 4th location of taking on the 3rd week can be seen in Figure 2. Concentrations of Pb(II), Cu(II) and Cd(II) were measured using the FAAS and is expressed in mg/kg dry weight. Levels of Pb(II), Cu(II) and Cd(II) in samples of tilapia nilotica, water hyacinth, and sediment on the 3rd week in the waters of Maninjau Lake with 4 locations has different levels.



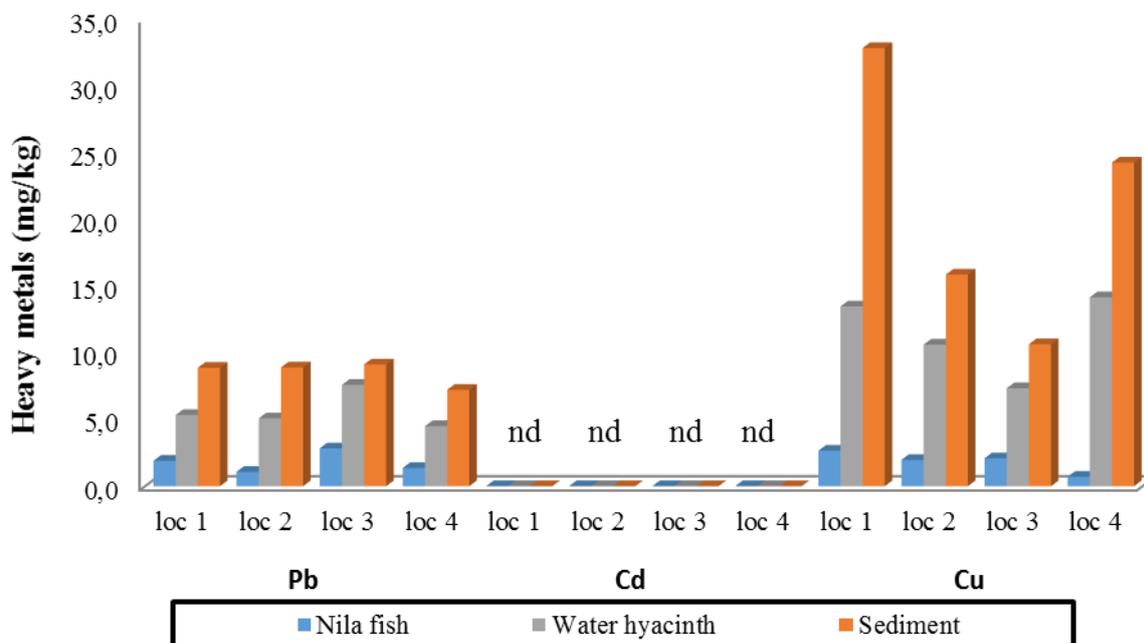
**Figure 2. Concentrations of Pb(II), Cu(II) and Cd(II) metals in tilapia nilotica, water hyacinth and sediment in the third week (mg/kg)**

The concentration of Pb(II) in samples of tilapia in the 3rd week obtained ranged from 0.0 - 2.0 mg/kg, in water hyacinth 2.6 - 5.1 mg/kg, and in sediment samples 7.3 - 11.9 mg/kg. Cd(II) metal concentrations were not detected in samples of tilapia, hyacinth, and sediment. The concentration of Cu(II) in tilapia 0.8 - 4.3 mg/kg, in water hyacinth 11.1 - 18.5 mg/kg, and in sediment 14.7 - 29.1 mg/kg. Therefore the concentration of Cu(II) in all samples has a higher concentration than Pb(II) and Cd(II).

The highest metal content Pb(II) were found in sediment samples one location in the 3rd week is 11.9 mg/kg. In addition to Pb(II), metal contamination comes from motor vehicle exhaust gas emissions and other human activities, a high metal content Pb(II) may also come from the lake itself. Maninjau Lake is a caldera formed by a major eruption that threw a metal material including Pb(II). Moreover, the process of mineral rocks corrosion is also one source path Pb included in waters [23-24].

**The Concentration of Pb(II), Cu(II) and Cd(II) samples in week – 6**

The content of heavy metals Pb(II), Cu(II) and Cd(II) on tilapia, water hyacinth and sediment in the 4th pick-up points at week-6 can be seen in Figure 3.



**Figure 3. Concentrations of Pb(II), Cu(II) and Cd(II) metals in tilapia nilotica, water hyacinth and sediment in the week six (mg/kg)**

The concentration of Pb(II) in samples of tilapia at week-6 obtained 1.1 - 2.8 mg/kg, in water hyacinth 4.5 - 7.6 mg/kg, and in sediment 7.2 - 9.1 mg/kg. The concentration Cd(II) are not detected in samples of tilapia nilotica, water hyacinth, and sediment. The concentration of Cu(II) in samples of tilapia at week-6 ranged from 0.7 - 2.6 mg/kg. The concentration of Cu in samples of water hyacinth on the 6th week ranged from 7.3 - 14.1 mg/kg. The concentration of Cu(II) in sediment samples at weeks 0 ranged from 10.6 - 32.8 mg/kg. The concentration of Cu(II) in samples of tilapia, hyacinth, and sediment at week 6 also have higher concentrations than the concentrations of Pb(II) and Cd(II).

Concentrations of Pb(II), Cu(II) and Cd(II) on the waters of Maninjau Lake tilapia on 4 locations within 6 weeks of the acquired Pb(II) concentrations 0.6 - 4.3 mg/kg. Concentrations of Cu(II) ranges between 0.7 - 5.6 mg/kg and Cd(II) metal concentrations undetectable. When compared with a maximum limit of metal contamination in the food specified in ISO 7387: 2009, the contents of Pb(II) contained in tilapia nilotica samples have exceeded the maximum levels. The maximum limit of Pb(II) in tilapia nilotica and their processed products was 0.3 mg/kg (Indonesian Government Regulation No. 82 of 2001). When compared with the maximum limit of the content of Pb(II), Cu(II) and Cd(II) in tilapia nilotica according to the WHO, the concentrations of Pb(II) and Cu(II) in samples of tilapia from this study has passed the maximum threshold. According to WHO, the maximum content of Pb(II), Cu(II) and Cd(II) on each tilapia nilotica is 0.05; 1.00 and 0.03 mg/kg [25].

Similarly, research on heavy metal content in the water of Maninjau Lake has done before, the result of heavy metals Pb ranged from 0.200 to 0.529 mg/L, the content of heavy metals Cd(II) ranged from 0.024 to 0.056 mg/L and the content of Cu(II) ranges 0.352 to 0.946 mg/L [24]. in relation to [21] Indonesian Government Regulation No. 82 of 2001 on Water Pollution Control and Water Quality Management, to limit heavy metal content is Pb(II) of 0.03 mg/L, Cd(II) of 0.01 mg/L and Cu of 0.02 mg/L. Proved that the content of Pb(II), Cu(II) and Cd(II) waters of Maninjau Lake also have exceeded the limits.

## CONCLUSION

It can be concluded that the samples of water hyacinth have the concentration of Pb(II) of 3.1 - 7.6 mg/kg, and Cu(II) of 7.1 - 18.5 mg/kg. Samples of tilapia has concentration of Pb(II) 0.6 - 4.3 mg/kg, and Cu(II) of 0.7 - 5.6 mg/kg. The sediment samples have concentrations of Pb(II) 6.6 -11.9 mg/kg, and Cu(II) 10.6 - 46.1 mg/kg. While, the concentration of Cd(II) in samples of water hyacinth, tilapia, and sediment undetected. The concentrations of Pb(II) and Cu(II) in tilapia, and concentration of Cu(II) in the sediment has passed the maximum threshold.

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## REFERENCES

- [1] A. Rani. Application leaf extract water hyacinth (*Eichornia crassipes*) on level and old save on the quality of chicken egg race, Thesis, Hasanuddin University, Makasar, Indonesia. 2014.
- [2] Darmono. Environmental and pollution (relationship with compound metal toxicology), UI-Press Publishers, Jakarta. 2001.
- [3] SJS. Adnan, Determination of mercury, arsenic, and cadmium in fish by neutron activation, *J. Radioanal. Chem.*, 2007, 44-101.
- [4] H. Suyani., Deswati, Refilda, and J. Sutopo, Water quality of post-event up welling and step anticipation in aquaponics location of Maninjau Lake, *Der Pharma Chemica*, 2016, 8(10), 102 -108.
- [5] Deswati; H. Suyani; R. Zein; Refilda, and J. Sutopo, Application of adsorptive stripping voltammetry for the determination of heavy metals Cd, Cu, Pb, and Zn in the culture of fish cage culture in Maninjau Lake, Agam, West Sumatera, Indonesia. *J. Chem. and Pharm. Res.*, 2015, 7(8), 942-947.
- [6] H. Syandri., Junaidi; Azrita, and T. Yunus, State of aquatic resources Maninjau Lake West Sumatra Province, Indonesia. *J. Ecol. and Env. Sci.*, 2014, 5(1), 109-113.
- [7] I. Arlindia, and Afdal. (2015), Pollution analysis Maninjau of value total dissolved solids and Electrical Conductivity. *J. Phys. Andalas Univ.*, 2015, 4(4), 325-331
- [8] Deswati; C. Buhatika; H. Suyani; Emriadi, and U. Loekman, Simultaneous determination of copper, lead, cadmium and zinc by adsorptive stripping voltammetry in the presence of murexide. *Int. J. Res. Chem. Environ.*, 2014, 4(2), 143 -152.
- [9] Deswati; H. Suyani; Safni; U. Loekman, and H. Pardi, Simultaneous determination cadmium, copper and lead in sea water by adsorptive stripping voltammetry in the presence of calcon as complexing agent. *Indones. J. Chem.*, 2013, 13(3), 236 -241.
- [10] Deswati; H. Suyani, and Safni, The Method of the development of analysis Cd, Cu, Pb and Zn in sea water by adsorptive stripping voltammetry (AdSV) in the presence of calcon as complexing agent. *Indones. J. Chem.*, 2012, 12(1), 20 -27.
- [11] Deswati; E. Munaf; H. Suyani; R. Zein, and H. Pardi, Simultaneous determination of trace amounts of iron, cobalt, nickel and chromium in water samples with calcon as a complexing agent by adsorptive stripping voltammetry. *Asian J. of Chem.*, 2015, 27(11), 3978-3982.
- [12] Deswati; E. Munaf; H. Suyani; U. Loekman, and H. Pardi, The sensitive and simple determination of trace metals Fe, Co, Ni and Cr in water samples by adsorptive stripping voltammetry (AdSV) in the presence of calcon. *Res. J. Pharm, Biol and Chem. Sci.*, 2014, 5(4), 990-999.
- [13] Deswati; L. Amelia; H. Suyani; R. Zein, and J. Jin, Applicability of casein in the simultaneous determination of trace metals copper, lead and cadmium by cathodic adsorptive stripping voltammetry. *Rasayan J. Chem.*, 2015, 8(3): 362-372.
- [14] Deswati; H.G. Izzati Rahmi; H. Suyani; R. Zein, and A.Alif, Optimization central composite design for the simultaneous determination of Cd(II) ion in fruit and vegetable samples by adsorptive stripping voltammetry. *Oriental J. Chem.*, 2016, 32(3), 1493-1502.
- [15] HG. Izzati Rahmi; Deswati; H. Suyani, and R. Zein, Design experiment optimization for the determination of copper in water samples by adsorptive stripping voltammetry. *Res. J. Pharm. Biol and Chem. Sci.*, 2016, 7(3), 673-682.

- [16] Deswati; I. Rahmi; H. Suyani; R. Zein, and A. Alif, Application of central composite design for optimization the determination of lead adsorptive cathodic stripping voltammetry. *Rasayan. J. Chem.*, 2016, 9(1), 8-17.
- [17] Deswati; H. Pardi; H. Suyani; R. Zein, and TW. Edelw, The development method for a sensitive simultaneous determination of Pb(II), Cd(II) and Zn(II) by adsorptive cathodic stripping voltammetry using alizarin as a complexing agent. *Anal & Bioanal. Electrochem.*, 2016, 8, 885- 898.
- [18] Deswati; H. Pardi; H. Suyani, and R. Zein, Adsorptive cathodic stripping voltammetric method with alizarin for the simultaneous of cadmium, and zinc in water samples. *Oriental J. Chem.*, 2016, 32(6), 3071- 3080.
- [19] Deswati; H. Suyani; R. Zein; A. Alif, and H. Pardi, Adsorptive stripping voltammetric method for the individual determination of Cd(II), Cu(II), Pb(II), and Zn(II) in vegetable samples using calcein. *Rasayan J. Chem.*, 2017, 10(2), 549-557.
- [20] W. Widowati; S. Astiana, and JR. Raymond, The toxic effects of metal, prevention and control of pollution. Publisher Andi, Yogyakarta. 2008.
- [21] Indonesian Government Regulation No. 82 of 2001 on control of air and water quality management. 2001.
- [22] R. Fitra, Analysis of heavy metal content of Fe, Cu, Pb and Cd in sediments of fish cage culture. Maninjau Lake, Thesis, Andalas University, Padang. 2011.
- [23] MH. Anny; Purwanto, and R.S. Tri, Heavy metal content on water, sediment and tilapia (*Oreochromis niloticus* Linn.) at fish cage culture Rawapening Lake. Proceedings of the National Seminar on Management of Natural Resources and Environment, Semarang, 2012, 95-1001.
- [24] H. Palar, Contamination, and toxicology of heavy metal. Rineka Cipta, Jakarta. 2004.
- [25] FA. Qurrata, Analysis of water quality: Temperature, pH, DO, Pb, Cd, Cu and Zn in Koto Malintang, Maninjau Lake. Thesis, Andalas University, Padang. 2016.