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Dithizone Membrane Manufacture Applicable On Membrane Paper For Early Detection Of Heavy Metal Lead (Pb) Impregnated With Buchner Vacuum

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Abstract. Research on the manufacture of nylon-dithizon membranes for the early detection of lead heavy metal using the vacuum impregnation method has been completed. The purpose of this study was to determine the manufacture of nylon dithizon membranes and test strips as dithizonate complexes as an early detection of lead heavy metal. Making nylon-dithizon membrane by dissolving 50 mg of dithizon with 100 mL of chloroform and the addition of 50 mg of SrCl_2 then stirred using a magnetic stirrer at 100 rpm as the working solution. After that, the solution was impregnated into a 0.20 m nylon membrane using a Buchner vacuum, after which it was carried out for 12 hours in a fume hood. The dithizon strip test was carried out using the colorimetric method, namely observing color changes that occur with the naked eye (qualitative test) with variations in concentration and pH. In this study, the optimum pH in the formation of the dithizone-Pb complex was obtained, namely pH 8 with a dark orange color. The minimum concentration on the indicator strip made was 0.05 with a visible color of faded orange, while the time reached for complex formation at a concentration value of 0.05 ppm was 520 seconds (Faded Orange).

Keywords: *dithizone membrane, membrane paper, heavy metal lead (Pb), buchner vacuum*

INTRODUCTION

The development of science and technology has triggered the expansion of industry which produces a lot of waste that is disposed of without proper treatment. Waste generated without realizing it will cause damage to the environment, one of which is water pollution. Water pollution is the entry of substances, energy, elements, or other components into water so that water quality is disturbed. Water pollution caused by the impact of industrial development must be controlled, because if it is not done early it will cause serious problems for human survival and the natural surroundings (Rizal et al., 2015).

Apart from industrial processes, environmental pollution can also be caused by agricultural activities that tend to use excessive pesticides and chemical fertilizers, indiscriminate displacement of land and settlements as well as domestic waste or household waste also contribute to increasing the amount of pollutants in the environment. The most dangerous problem is the presence of heavy metals contained in these various pollutants. These heavy metals include cadmium (Cd), Cobalt (Co), Copper (Cu), Mercury (Hg), and Lead (Pb) (Rizal, et al., 2015). Several industries that produce heavy metal waste include the gold mining industry which uses excessive mercury for the amalgamation process (Nashukha, 2014).

The main cause of heavy metals being hazardous pollutants is that heavy metals cannot be decomposed or destroyed and are chemical contaminants that are included in the Toxic and Hazardous Materials (B3) group. Heavy metals are easily absorbed and in relatively low concentrations are generally toxic if they enter the human body through various intermediaries, such as air, food, or water contaminated by heavy metals, these metals can be distributed to parts of the human body.

In addition, heavy metals are also cumulative in the soil so that they can exceed the threshold for metal presence in the environment which is very dangerous for the environment as well as organisms (Bugis

et al. , 2012). Therefore, pollution of an environment by heavy metal ions has always been a problem for both developed and developing countries to monitor the presence of heavy metal ions in the environment.

One of the heavy metals that can pollute waters is lead. Lead is a non-essential heavy metal which is very dangerous and can cause poisoning (toxicity) in living things. This poison is cumulative, meaning that its toxic nature will arise if it accumulates in large enough quantities in the bodies of living things. In the human body, lead (Pb) can inhibit the activity of enzymes involved in the formation of hemoglobin (Hb) and a small portion of lead (Pb) is excreted through urine or feces because some are bound by protein, while some accumulate in the kidneys, liver, nails, fat tissue, and hair. The half-life of lead (Pb) in erythrocytes is 35 days in kidney and liver tissue for 40 days, while the half-life in bone is 30 days. The rate of Pb excretion through the urinary system was 76%, gastrointestinal 16%, for hair, nails, and sweat was 8%. Lead is present in water because of the presence of contact between water and soil or air contaminated with lead, water contaminated by industrial waste or due to pipe corrosion (Fardiaz, 1992; Bridiatama, 2014).

In general, lead analysis is carried out by atomic absorption spectroscopy (Gandjar and Rohman, 2009). The use of atomic absorption spectroscopy in detecting lead contamination must be carried out in the laboratory and cannot be done by the general public as an early detection of lead contamination in beverages. For this reason, it is necessary to use a simple method as an early detection of lead contamination in beverage samples. One of the detections of lead contamination is the formation of complex compounds with ligands. One of the complexing agents capable of reacting with several metals is diphenylthiocarbazone or often known as dithizone. By carrying out a complex formation reaction with dithizone, it is hoped that an early detection of lead contamination in the environment can be carried out in a simple way. This study aims to make a nylondithizon membrane in the form of dithizone which is applied to membrane paper to detect the presence of lead heavy metal contamination in the sample. Impregnation is a method with the principle of removing part or all of the air or liquid in a material and then replacing it with the desired fluid or osmotic solution (Nugraha et al., 2015).

RESEARCH METHODS

1. Location and Time of Research

This research was conducted at the Chemistry Laboratory of the Faculty of Mathematics and Natural Sciences, UNIMED, Jl. Williem Iskandar Pasar V Medan. This research was conducted from August 2021 – October 2021.

2. Tools and Materials

2.1. Tool

The tools used in this research are beaker, erlenmeyer, measuring cup, volume pipette, volumetric flask, all of which are pyrex. Also used are pH meters, Buchner vacuum, magnetic stirrer and analytical balance AND HR-200.

2.2. Ingredient

The materials used in this study were Pb(NO₃)₂, Nylon Membrane Filter 0.22 m(Whatman), concentrated HCl (12 M), concentrated HNO₃ (16 M), SrCl₂, Dithizon, Chloroform, NH₄OH and aquadest. All p.a (pro analyst) quality materials from E. Merck.

3. Work Procedure

3.1. Preparation of Working Solution

50 mg of dithizon was dissolved in 100 mL of chloroform and addition of 50 mg SrCl₂. Then 10 mL of distilled water was added to the solution and stirred with a magnetic stirrer.

3.2. Nylon Membrane Manufacturing – dithizone/detect

Then the solution was injected into 10 mL of water which was stirred at 100 rpm. The detection membrane was made by filtering the dithizone on membrane paper under vacuum. Then the membrane was dried in a fume hood. The membrane is stored in a closed container.

3.3. Preparation of Sample Solutions(AOAC, 2000)

Pb(NO₃)₂ solids were weighed to make heavy metal samples at a certain concentration of 0.1; 0.5; 1; 2 ; and 5 ppm.

3.4. Determination of Optimum pH Formation of Dithizonate complex

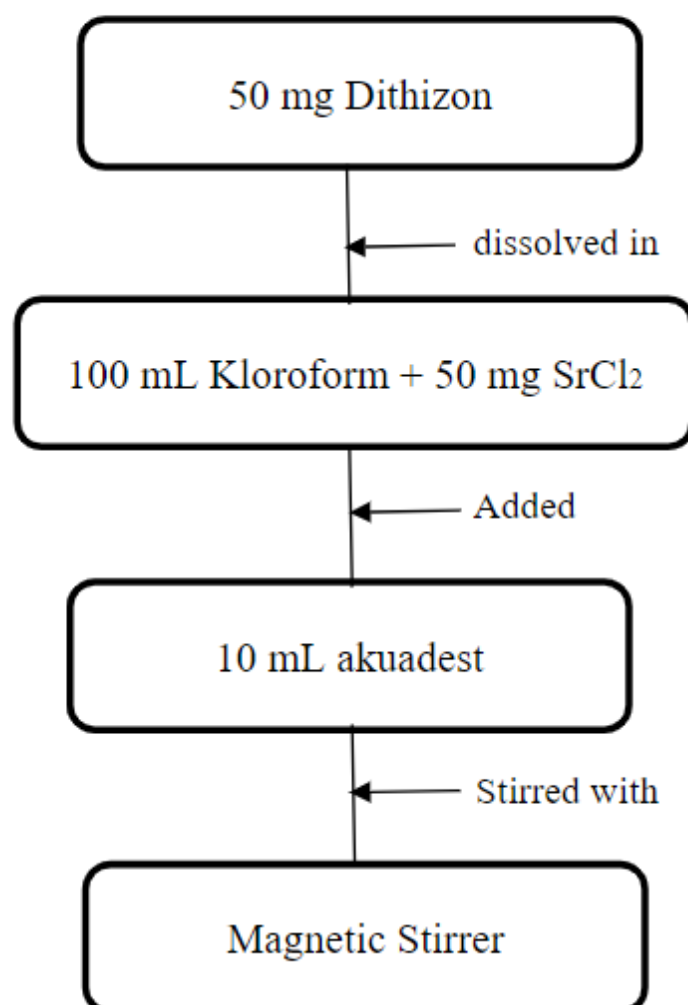
Into a test tube, 5 mL of sample was inserted, adjusted pH = 4, 5, 6, 7, 8 with the addition of 1N ammonium hydroxide or 10% HNO₃, then dithizone strip was inserted. If the strip is orange to red, it means that the sample contains Lead.

3.5. Determination of Nylon/Ditizon Membrane Working Range and Response Time

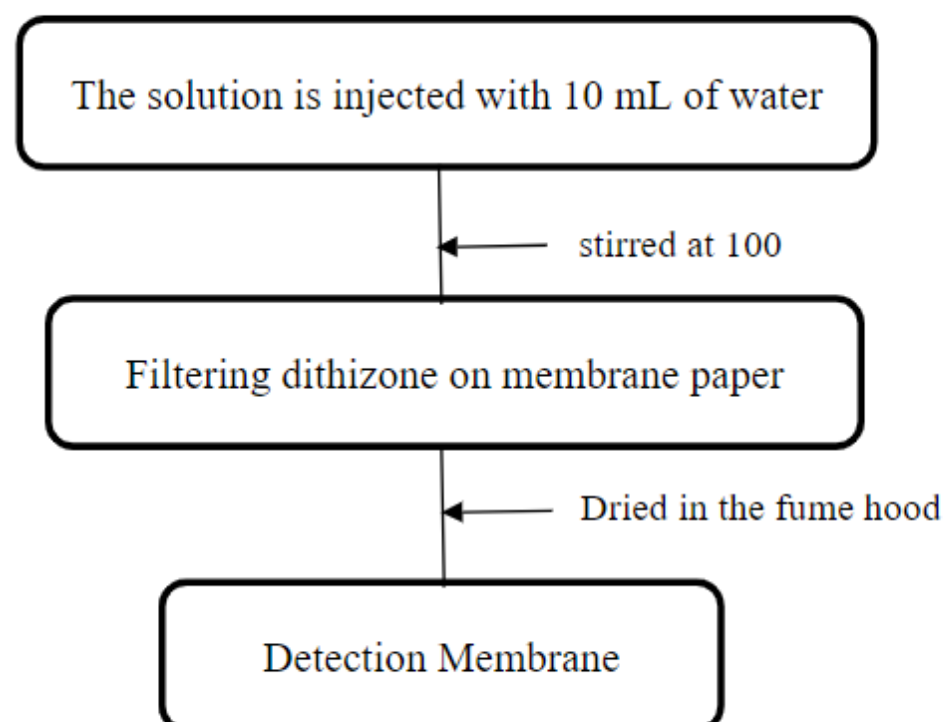
Into the test tube, 5 mL of sample was added with levels varying from 0.1 ppm to 2 ppm, adjusted to pH 8 with the addition of 1N ammonium hydroxide. Then the dithizone strip is inserted. A color change in the dithizone strip was observed. In determining the response time, a stopwatch is used to calculate the response time of the detector for each variation concentration.

4 Flowchart

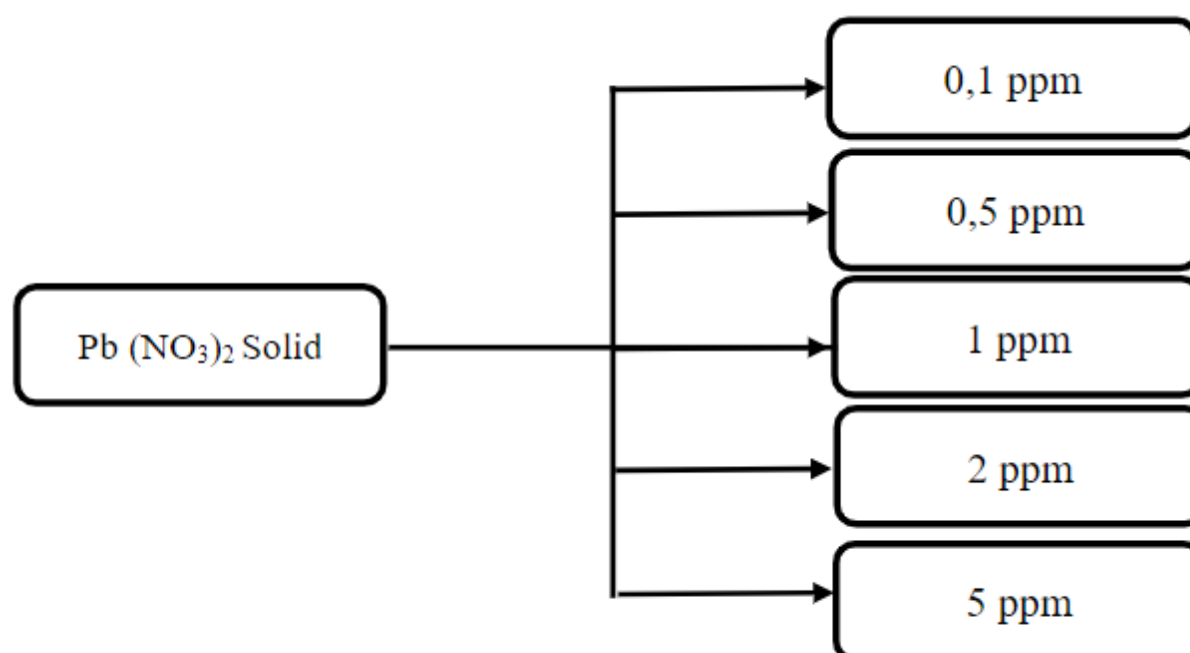
4.1 Preparation of Working Solution



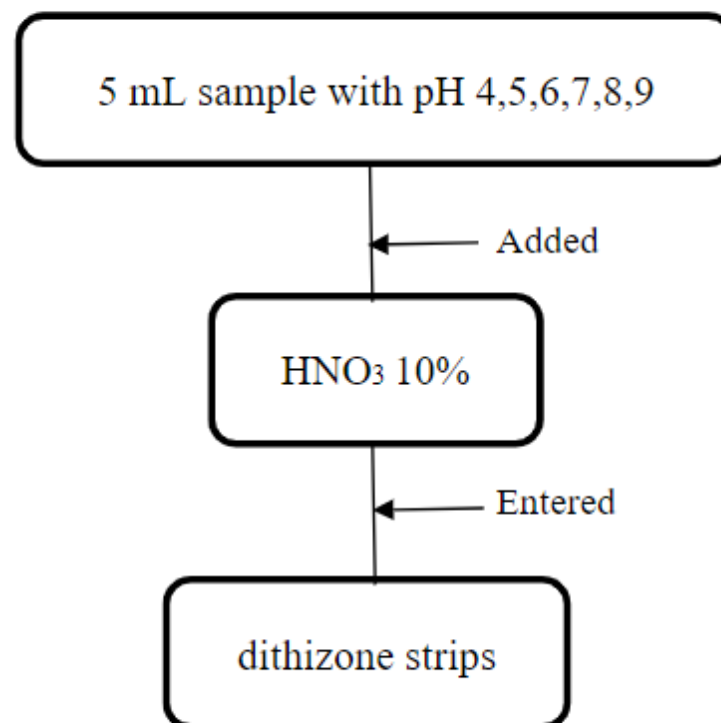
4.2. Nylon–Dithizone Membrane Preparation/ Detect



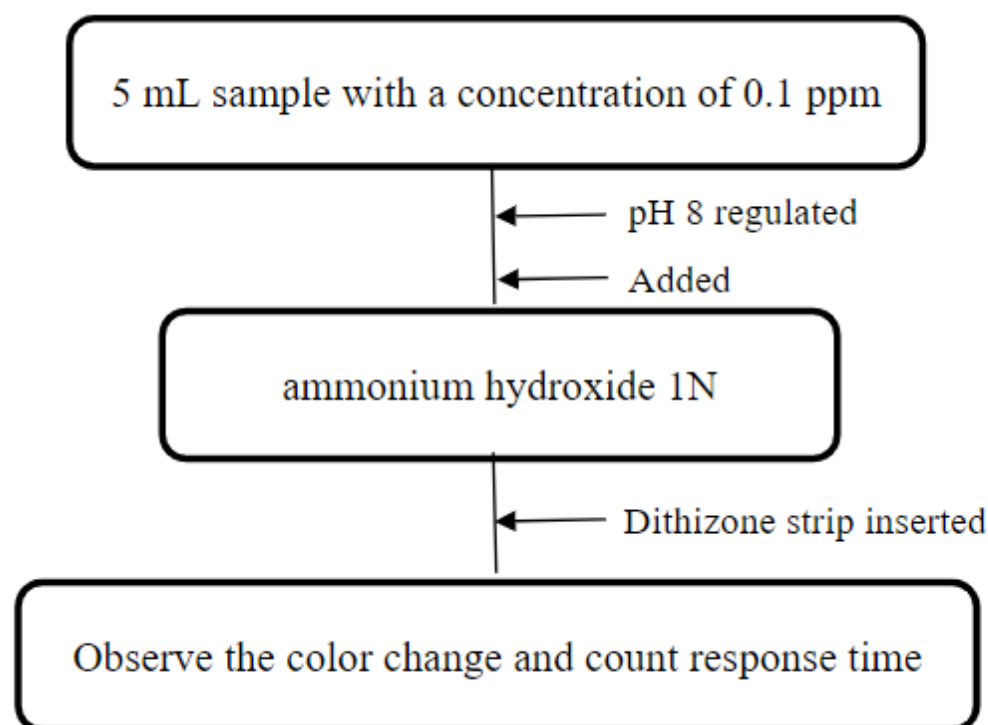
4.3 Pembuatan Larutan Sampel



4.4. Determination of Optimum pH Dithizonate complex formation



4.5. Determination of Nylon/Ditizon Membrane Working Range and Response Time



RESULTS AND DISCUSSION

4.1. Description of Research Results

The results of this study include the manufacture, determination of the optimum pH and the minimum limit for the concentration of complex formation on the dithizon indicator strip.

4.1.1. Preparation of fast reagent for early detection of metal ion Pb²⁺

The manufacture of fast reagents in this study includes the manufacture of The working solution is a dithizon solution which is then impregnated with a Buchner vacuum into a nylon membrane. In this study, we have succeeded in making indicator strips with an identifier, namely dithizon as a metal ion detector Pb^{2+} with nylon membrane as an adsorption medium. In making the working solution, chloroform solution was used to dissolve dithizon. This is because dithizone is a violet-black solid that does not soluble in water, but soluble in ammonia solution and soluble in chloroform and carbon tetrachloride to give a green solution. According to (Jeffery et al., 1989) the solubility of dithizone in CCl_4 is 0.5 mg/mL while that in chloroform is 20 mg/mL.

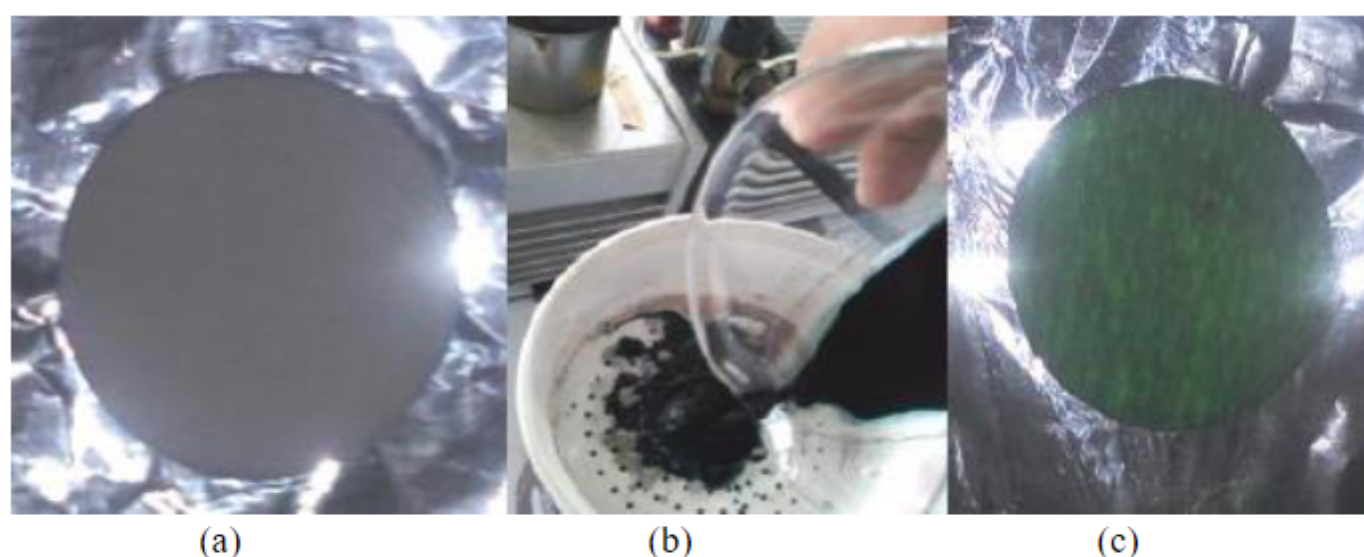


Figure 4.1. (a) Before impregnation, (b) Impregnation, (c) Detection strip

4.1.2. Determination of the optimum pH of dithizon indicator strips

The optimum pH measurement is carried out using a pH meter

by measuring the level of acidity and alkalinity of a solution to determine the pH that affects the performance of the dithizon indicator strip. In determining the optimum pH used a variation of pH 4-10 as the sample determination.

Table 4.1. Determination of the optimum pH of dithizon indicator strips

pH	Discoloration	pH	Discoloration
4	Stay Green Stay Green Stay Green	7	Orange Orange Orange
5	Stay Green Stay Green Stay Green	8	dark orange dark orange dark orange
6	Orange Orange Orange	9	faded orange faded orange faded orange

In Table 4.1 above, it can be concluded that the optimum pH for observing changes in the color of the impregnated membrane with the naked eye is pH 8. The greater the alkaline nature of a ligand, the more stable the complex will be. The base in question is a Lewis base. In the formation of the chelate complex, the donor group releases H^+ to form the corresponding base.

The metal ion Pb^{2+} in an alkaline environment, the OH^- ion will bind to one of the H^+ ions in the dithizone to form the dithizonate anion. This form of anion will form a stable complex with Pb^{2+} . Meanwhile, under acidic conditions, there is competition between Pb^{2+} and H^+ ions to bind to dithizone. According to (Jeffery et al., 1989) if H^+ binds to dithizone it will form dithizonic acid while when Pb^{2+} binds to dithizone it will form an unstable dithizone- Pb^{2+} complex. Dithizone will form a complex with metal ion Pb^{2+} to form an orange colored complex.

4.1.3. Determination of the minimum limit for the concentration of dithizon indicator strips

The determination of the minimum concentration limit is carried out to determine the minimum concentration of Pb metal ions that can be detected by the indicator strip. The concentration variations used ranged from 0.025 to 2 ppm, with the addition of 1 N ammonium hydroxide as the optimum pH regulator which was previously known through Table 4.1. The minimum concentration limit obtained on this indicator strip is 0.025, which means that there is no change, while at the concentration above it has changed, such as 0.05, the color is faded orange. This is influenced by the higher concentration making the reaction occur faster and the concentration of the strip indicator.

4.1.4. Determination of dithizon . indicator strip response time

The response time was determined to determine how fast the indicator strip works in detecting Pb^{2+} metal ions. This determination was carried out by varying the sample concentration between 0.05 – 2 ppm in optimum pH conditions.

Table 4.2. Determination Of Working Time Of Dithizon Indicator Strip

No	Time (s)	Concentration (ppm)
1	240	2
2	280	1
3	320	0,8
4	340	0,4
5	380	0,2
6	420	0,1
7	520	0,05

In Table 4.2 it can be concluded that the working time is influenced by the optimum concentration and pH in detecting Pb^{2+} metal ions. The greater the concentration of Pb^{2+} metal ions, the faster the indicator strip works. The fast response of the indicator strip is influenced by the optimum concentration and pH.

4.1.5. Determination of Dithizon Indicator Strip Concentration Limits

Determination of the detection limit is carried out to determine the minimum limit for the concentration of Pb metal that can be detected by the indicator strip. The concentration variations used ranged from 0.025 to 2 ppm, with the addition of 1N ammonium hydroxide as the optimum pH regulator which was previously known through table 4.1. The results of determining the detection limit are shown in table 4.3 below.

Table 4.3. Determination Of Detection Limit Of Dithizon Indicator Strip

No	Sample Concentration	Discoloration
1	2 ppm	Dark orange Dark orange Dark orange
2	1 ppm	Dark orange Dark orange Dark orange
3	0,8 ppm	Dark orange Dark orange Dark orange
4	0,4 ppm	Orange Orange Orange

CONCLUSION

From the research results of Nylon-Dithizon Membrane Making for Early Detection of Heavy Metal Lead Using Vacuum Impregnation Method, the following conclusions are obtained:

1. Chloroform solvent was used as dithizon solvent which was then impregnated into a nylon filter membrane. The indicator strip obtained was as expected to be able to detect lead metal by adjusting the pH of the sample.
2. The optimum pH in the formation of the dithizone-Pb complex is at pH 8 with a dark orange color. The minimum concentration on the indicator strip made was 0.05 with a visible color of faded orange, while the time taken for the formation of the complex itself at a concentration value of 0.05 was 520 seconds.

SUGGESTION

Based on the conclusions above, then:

1. It is necessary to do further research on the dithizonate complex using different metals in the indicator strip test.
2. It is necessary to develop research using sensors optics to produce qualitative and quantitative indicator strip tests for more accurate results.

Acknowledgments

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