

THE EFFECT OF TEMPERATURE AND TIME ON THE MIGRATION OF LEAD METAL (Pb) IONS IN CANNED ORANGE JUICE DRINK USING ATOMIC ABSORPTION SPECTROPHOTOMETRY ANALYSIS

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EFEITO DA TEMPERATURA E DO TEMPO NA MIGRAÇÃO DE ÍONS DE CHUMBO (PB) EM BEBIDAS DE SUÇO DE LARANJA EM LATA USANDO ANÁLISE DE ESPECTROFOTOMETRIA DE ABSORÇÃO ATÔMICA**THE EFFECT OF TEMPERATURE AND TIME ON THE MIGRATION OF LEAD METAL (PB) IONS IN CANNED ORANGE JUICE DRINK USING ATOMIC ABSORPTION SPECTROPHOTOMETRY ANALYSIS****PENGARUH SUHU DAN WAKTU TERHADAP MIGRASI ION LOGAM TIMBAL (PB) PADA MINUMAN JUS JERUK KALENG MENGGUNAKAN ANALISIS SPECTROPHOTOMETRY ABSORPSI ATOM**

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RESUMO

Introdução: A Indonésia é um país que tem um clima tropical ao longo do ano, juntamente com uma diferença de temperatura relativamente menor durante as estações seca e chuvosa. A comunidade móvel influencia o interesse das pessoas por bebidas embaladas, incluindo sucos de frutas em lata. No entanto, o primeiro obstáculo é a condição do local e a migração do metal pesado chumbo (Pb) relacionado à temperatura nas bebidas em lata de suco de laranja. **Métodos:** Este estudo enfocou a relação entre o teor de metal Pb, o tratamento de armazenamento, a temperatura de armazenamento e o tempo de armazenamento. Este estudo utilizou uma bebida de suco de laranja em lata do armazém do distribuidor da cidade de Palembang, Pb padrão (NO₃)₂ (Merck) em pó, soluções concentradas de HNO₃ 0,5 mol / l (Merck), solução concentrada de HCl (Merck), água desmineralizada, pH do tampão 4.7 e 10. A amostra foi conduzida de 5 °C a 40 °C com o período de teste dentro de 1-30 dias. A amostra em determinados intervalos de dias seria então testada para alterar o pH e a concentração de íons Pb usando o teste de Espectrofotometria de Absorção Atômica (AAS). **Resultados e Discussão:** Neste estudo, a amostra de suco de laranja recebeu diferentes tratamentos, sendo que cada tratamento possui um código de análise diferenciado composto por S-SBJ-1-1 (Armazém do distribuidor) para um dia de armazenamento, e os códigos para 10 de tempo de armazenamento são S-SBJ-10-1 (Freezer, temperatura 5 °C), S-SBJ-10-2 (Geladeira, temperatura 20 °C), S-SBJ-10-3 (Homeroom, temperatura 28 °C), S-SBJ-10-4 (Forno, temperatura 40 °C), S-SBJ-10-5 (Espaço aberto, temperatura 22 °C - 38 °C). Além disso, os códigos para 30 dias de armazenamento de amostra são S-SBJ-30-1 (freezer, temperatura 5 °C), S-SBJ-30-2 (refrigerador, temperatura 20 °C), S-SBJ-30-3 (Casa da sala, temperatura 28 °C, S-SBJ-30-4 (Forno, temperatura 40 °C) e S-SBJ-30-5 (Espaço aberto, temperatura 22 °C - 38 °C). **Conclusões:** Temperatura e O tempo tem uma influência significativa na migração do metal pesado chumbo (Pb) da lata para as bebidas de suco de laranja e, para o valor mínimo de pH, há uma alteração menos significativa. Em geral, o suco de fruta em lata deve ser armazenado em temperatura abaixo de 28 °C, e a bebida é protegida da luz solar direta e de alta umidade..

Palavras-chave: adsorção, latas de bebidas, tempo de armazenamento, temperatura

ABSTRACT

Background: Indonesia is a country that has a tropical climate throughout the year along with relatively less difference of temperature during both dry and rainy seasons. The mobile community influences people's interest in packaged drinks, including canned fruit juice. However, the first obstacle is the condition of the place and the temperature-related heavy metal lead (Pb) migration in canned orange juice drinks. **Methods:** This study focused on the relationship between Pb metal content, storage treatment, storage temperature, and storage time. This study used a canned orange juice drink from the distributor warehouse of Palembang city, standard $Pb(NO_3)_2$ (Merck) powder, 0.5 mol/l concentrated HNO_3 solutions (Merck), concentrated HCl solution (Merck), demineralized water, Buffer pH 4.7, and 10. The sample was conducted from 5 °C to 40 °C with the testing period within 1-30 days. The sample at certain day intervals would then be tested to change pH and Pb ion concentration using the Atomic Absorption Spectrophotometry (AAS) test. **Results and Discussion:** In this study, the sample of orange juice was given different treatments, and each treatment has a differentiated analysis code consisting of S-SBJ-1-1 (Distributor warehouse) for one day of storage time, and the codes for 10 of storage time are S-SBJ-10-1 (Freezer, temperature 5 °C), S-SJB-10-2 (Refrigerator, temperature 20 °C), S-SJB-10-3 (Homeroom, temperature 28 °C), S-SJB-10-4 (Oven, temperature 40 °C), S-SJB-10-5 (Open space, temperature 22°C - 38 °C). Furthermore, codes for 30 days of sample storage are S-SBJ-30-1 (Freezer, temperature 5 °C), S-SJB-30-2 (Refrigerator, temperature 20 °C), S-SJB-30-3 (Room house, temperature 28 °C, S-SJB-30-4 (Oven, temperature 40°C), and S-SJB-30-5 (Open space, temperature 22 °C-38 °C). **Conclusions:** Temperature and time have a significant influence on the migration of the heavy metal lead (Pb) from the can to orange juice drinks, and, for the minimum pH value, there is a less significant change. Overall, canned fruit juice should be stored at a temperature below 28 °C, and the drink is protected from direct sunlight and high humidity.

Keywords: *adsorption, beverage cans, storage time, temperature*

ABSTRAK

Latar Belakang: Indonesia merupakan negara yang beriklim tropis sepanjang tahun dengan perbedaan suhu yang relatif lebih kecil baik pada musim kemarau maupun musim hujan. Komunitas mobile mempengaruhi minat masyarakat pada minuman kemasan, termasuk jus buah kalengan. Namun kendala pertama adalah kondisi tempat dan migrasi logam berat (Pb) terkait suhu pada minuman jus jeruk kaleng. **Metode:** Penelitian ini difokuskan pada hubungan antara kandungan logam Pb, perlakuan penyimpanan, suhu penyimpanan, dan waktu penyimpanan. Penelitian ini menggunakan minuman sari buah jeruk kalengan dari gudang distributor kota Palembang, serbuk standar $Pb(NO_3)_2$ (Merck), larutan HNO_3 pekat 0,5 mol/l (Merck), larutan HCl pekat (Merck), air demineral, Buffer pH 4.7, dan 10. Pengambilan sampel dilakukan dari suhu 5 °C hingga 40 °C dengan periode pengujian dalam waktu 1-30 hari. Sampel pada interval hari tertentu kemudian akan diuji perubahan pH dan konsentrasi ion Pb menggunakan uji Atomic Absorption Spectrophotometry (AAS). **Hasil dan Pembahasan:** Pada penelitian ini sampel sari buah jeruk diberi perlakuan yang berbeda, dan setiap perlakuan memiliki kode analisis yang berbeda yang terdiri dari S-SBJ-1-1 (gudang Distributor) untuk lama penyimpanan satu hari, dan kode untuk 10 lama penyimpanan adalah S-SBJ-10-1 (Freezer, suhu 5 °C), S-SJB-10-2 (Kulkas, suhu 20 °C), S-SJB-10-3 (Homeroom, suhu 28 °C), S-SJB-10-4 (Oven, suhu 40 °C), S-SJB-10-5 (Ruang terbuka, suhu 22 °C - 38 °C). Selanjutnya kode penyimpanan sampel selama 30 hari adalah S-SBJ-30-1 (Freezer, suhu 5 °C), S-SJB-30-2 (Kulkas, suhu 20 °C), S-SJB-30-3 (Ruang rumah, suhu 28 °C, S-SJB-30-4 (Oven, suhu 40 °C), dan S-SJB-30-5 (Ruang terbuka, suhu 22 °C-38 °C). **Kesimpulan:** Suhu dan Waktu memiliki pengaruh yang signifikan terhadap perpindahan logam berat timbal (Pb) dari kaleng ke minuman jus jeruk, dan untuk nilai pH minimum terjadi perubahan yang kurang signifikan. Secara keseluruhan, jus buah kalengan harus disimpan pada suhu di bawah 28 °C, dan minuman terlindung dari sinar matahari langsung dan kelembaban tinggi.

Kata kunci: *adsorpsi, kaleng minuman, lama penyimpanan, suhu*

1. INTRODUCTION:

Canned drinks and beverages are large market sectors following the market of bottled mineral water (Arif, 2020) as their price range can be considered affordable. Specifically, Indonesian consumers spend an average of 2% (percent) of their monthly income on purchasing packaged drinks (Triyono, 2013).

However, using a can as a container for a

packaged drink, especially for long-term storage and longer consumption, would cause heavy metals contamination in canned orange juice drinks (St, 2011) (Paula *et al.*, 2015).

Poisoning is one of the harmful effects if the can damage leading to a possible chemical reaction. This reaction would decrease the drink acidity resulting in reduction and oxidation reactions. This happens to the packaging cans that do not meet the standard requirements. The

occurrence of corrosion, a color change, and the formation of hydrogen gas can damage the can and other harmful microbiological activity. The formation of hydrogen gas (H₂) and carbon dioxide (CO₂), for example, is caused by the growth of anaerobic spore-forming bacteria classified as Clostridium, including poisonous C. Botulinum. The growth of spore-forming bacteria can break down protein and produce hydrogen sulfide (H₂S), so the cans would rot and turn black due to the reaction between sulfides and iron (Amin, 2015)

A can made of metal or metal alloy is definitely not an inert material. Cans that are not properly coated with an inert material (protective layer) may have some defects on their inside part, causing corrosion that can release metal elements from the can to the drink. The metal elements such as Lead (Pb), Iron (Fe), Tin (Sn), Cadmium (Cd), and Zinc (Zn) will negatively affect human health. The presence of these metals, even in small amounts, will still endanger the health of people who get long metal exposure (Sugiasuti *et al.*, 2006, Martini *et al.*, 2020, Roslinda *et al.*, 2013).

Lead (Pb) is included in the metal group IV-A on the periodic table of chemical elements, with atomic number (NA) 82 and atomic weight (BA) 207.2. Lead (Pb) can cause chronic and acute poisoning effects (Ian Tanu, 2016). The exposure threshold for lead and lead arsenate in the air is 0.15 mg per cubic meter. Meanwhile, the exposure limit for tetramethyl lead and tetraethyl lead is 0.07 mg per cubic meter, and the threshold for lead content in food is 2.56 mg/kg (Sartono, 2012) (Nasution, 2011).

Therefore, metal corrosion should be included as an essential factor for selecting the type of can for food and drink packaging. Several aspects affecting corrosion in the inner cans are the level of remaining oxygen in the food, the presence of corrosion accelerating agents such as nitrates and other sulfur compounds, drinks pH, temperature and storage time, and material components of cans presence of corrosion-resistant coating substances.

In addition, other factors can cause detrimental effects of consuming damaged packaging cans, such as incomplete beverage processing and storage time, which significantly affect the migration of lead metal from the can to the drink. In terms of storage time, it is known that the longer the storage time, the longer the contact time between the metal and the drinks (Irawan, 2013). Following this matter, orange juice with a relatively higher acidity level and contains oxidizing agents can cause corrosion in the cans.

Thus, the migration of lead (Pb) will occur (Yusrizal, 2015)

Heavy metals in the sample can be detected and measured using the wet digestion method by applying a mixture of HNO₃ and H₂O₂. It is then further identified by atomic absorption spectrophotometric instrument (AAS), Kharisma, 2006). AAS instrument has high sensitivity and selectivity levels, therefore, it is reliable to analyse metal with the ability to detect around 62 different metal elements (Suryati, 2011).

AAS can analyze quantitatively based on the absorbance measurement of sample solution. The amount of elemental content analyzed in the sample solution is calculated based on the standard graph or calibration curve. Based on Beer's law, there is a linear relationship between absorbance and the concentration of elements present in the solution (Skoog, 2004).

In this work, the main objective is to measure the influence of temperature (place of storage) and time (shelf life) of drink cans on the corrosion phenomenon, along with the possible migration of heavy metals (Pb) from the can to the drink.

2. MATERIALS AND METHODS:

This research was conducted in the laboratory of PT. Sucofindo Palembang Branch, Indonesia. This study used canned orange juice drink from the distributor warehouse of Palembang city, standard Pb(NO₃)₂ (Merck) powder, 0.5 mol/l concentrated HNO₃ solutions (Merck), concentrated HCl solution (Merck), demineralized water, buffer solution pH 4.7, and 10.

The sample was carried out at a temperature range of 5 °C to 40 °C with a length of time for testing 1-30 days. The sample at certain day intervals would be tested for the changes of pH and Pb ions levels using the AAS test.

Quantitative analysis of lead was carried out using an AAS analyzer with a graphite furnace. Around 20 µL of the concentrated sample, obtained from the acid process by reflux, is injected into the graphite cells. Previously, the optimal pyrolysis temperature (50 to 800 °C) and atomization (1000 to 1700 °C) were determined and programmed into the graphite furnace. Drying (100 °C) and cleaning (2500 °C) temperatures are programmed according to the instructions in the previous report. The technique used is a normal electrographic cell (Thermo Elemental Solaar) with argon flowing at a speed of 0.2 L / min and a

gap of 0.5 nm. Lead concentration was determined by constructing a calibration curve with lead concentrations ranging from 2.0 to 7.0 µg/L and reading the maximum absorbance at a wavelength of 216.9 nm. This dilution was prepared from a stock solution in 0.2% HNO₃ in a 5 mL volumetric flask. All samples were measured in triplicate, and the mean values were expressed as µgPb/Kg. (Rada-Mendoza *et al.*, 2018)

The wavelength used in the analysis was 283.3 nm. Lead has the energy of 7.0134.10⁻⁸ Joule. This can cause the Pb atom to be in the ground state (Pb o) and excited to a higher energy level (Pb *). The acetylene-air flow rates used as burner and oxide for Pb metal were 2.0 L / min and 10.0 L / min. Pb standard solution is made from Pb E-Merck 1000 ppm stock solution. This standard solution was diluted to 10 ppm. It was then separately diluted again into a series of standard solutions having a concentration of 0.5; 1; 2; 3, and 4 ppm. Dilution was carried out with 0.5M HNO₃ because the matrix in the standard solution must be the same as the matrix in the sample. The measurement of the absorbance of the standard solution was conducted using the AAS tool. The absorbance showed the sample's ability to absorb electromagnetic radiation at its maximum wavelength. The calibration curve of the Pb metal standard solution can be seen in the absorbance measurement of the standard solution using the AAS instrument. (Dewi, 2013).

Furthermore, the analytical validation for the lead standard curve can be stated as follows:

- The linearity of the lead standard curve (Pb) is 0.9999, meaning that ± 99% change in absorbance is influenced by changes in lead concentration (Pb), while other factors are ± 1%.
- The standard Pb curve can determine the smallest limit of an analyte based on the calculations in the appendix is LOD = 0.028 ppm and LOQ = 0.0933 ppm
- The sensitivity of the standard Pb curve is 0.00757. This value indicates that every unit change in concentration will result in a change in absorbance of 0.00757
- The accuracy values of the standard lead curve expressed in % recovery are 91.4% for 0.5 ppm; 99.9% for 1 ppm; 98% for 2 ppm; 101.3% for 3 ppm and 99.75% for 4 ppm. Priyambodo (2011) stated that the requirement for accuracy is the percent recovery within the range of 98-102%.
- This work obtained 2.74% of precision. As the coefficient of variation for the standard lead curve (Pb) is below 5%, it can be stated that the

standard curve precision has been reached as it is sensitive for analyzing lead metal (Pb). (Dewi, 2013)

The column method used in analyzing lead (Pb) was generally carried out by utilizing a 500 mL lead solution with a concentration range of 0.5-100 mg of lead used as an aliquot. Adjusting the pH of the aliquot to 9.6 was done by adding a buffer solution and then diluting it to 20 mL with distillate water. The column used was filled with PAN-naphthalene as the adsorbent, which then adjusted the acidity level to pH 9.5 by adding a 2 to 3 mL buffer solution. Aliquots were then streamed through the column at a rate of 1 mL/min. The solid metal adsorbed into the naphthalene was then dissolved with 5 mL of dimethylformamide (DMF). The analyzed sample was flown into the fire with acetylene-air fuel with a wavelength of 217.0 nm. The same treatment was carried out for reagent blanks in order to obtain a calibration chart (Taher, 2003)

Measurement of Pb level was started by measuring the standard solution. Measurements were initiated by the standard solution with the smallest concentration, then continued until the highest concentration, followed by measuring sample absorption. The sample absorption obtained was entered into the calibration curve equation in order to obtain the sample content. Measurements were made using the AAS method with the provisions of the wavelength of 283.3 nm, gas speed (acetylene) 2 L / minute, oxidant speed (air) of 15 L / minute, and burner height of 7mm. This wavelength is the most powerful wavelength absorbing the line for the electronic transition from the ground level to the excitation level. When atoms in the ground state are given the appropriate energy, then would be absorbed, and the atoms would be excited to a higher energy level (excited state).

In contrast, unstable atoms would return to the basic energy level by releasing a certain amount of energy. The optimum wavelength of energy in the form of light for Lead (Pb) is 283.3 nm (Shimadzu, 2007; Perdana, 2019). The research flowchart of this work is displayed in figure 6.

3. RESULTS AND DISCUSSION:

3.1. Results

This study used a sample of orange juice with different treatments, and each treatment has a differentiated analysis code consisting of S-SBJ-1-1 (Distributor warehouse) for one day storage

time, for a storage time of 10 days with the code S-SBJ-10-1 (Freezer, temperature 5 °C), S-SJB-10-2 (Refrigerator, temperature 20°C), S-SJB-10-3 (Homeroom, temperature 28 °C), S-SJB-10-4 (Oven, temperature 40 °C), S-SJB-10-5 (Open space, temperature 22°C to 38 °C). For sample storage for 30 days, the codes are S-SBJ-30-1 (Freezer, temperature 5 °C), S-SJB-30-2 (Refrigerator, temperature 20 °C), S-SJB-30-3 (Room house, temperature 28 °C, S-SJB-30-4 (Oven, temperature 40 °C), S-SJB-30-5 (Open space, temperature 22°C to 38 °C). The analysis results of orange juice samples cans can be seen as follows:

For sample 1, 2, 3, and 4, the ABS values were 0.00074; 0.000725 , 0,000763 ,0,000801 , respectively. Then 0,001528 for sample 5; 0,000897 for sample 6; 0,000782 for sample 7; 0,000801 for sample 8; 0,000897 for sample 9; 0,002751 for sample 10; and for ABS sample 11 of 0,002751. Based on Indonesia government regulation, the maximum limit of Pb metal in 1 kg of fruit drink is 0.1 mg.

3.2. Discussion

Based on the measurement, it is known that the temperature can cause the migration of lead metal from the can to the drink. The release of lead metal can also be affected by the duration of storage. The longer the food is stored, the longer the contact time between the food and the tin packaging leading to higher possibility of metal migration from the can to the drink (Perdana, 2019)

In Figure 8, it is known that the treatment of the sample influences the pH value in the sample. The initial sample with code S-SBJ-1-1 has a pH value of 2.930; the sample with code S-SBJ-10-1 is 2.934. In this case, the increase in pH is 0.004. Furthermore, the pH of the orange juice sample S-SBJ-10- 2 is 2,932 and represents a 0.002 increase from the baseline sample. Sample S-SBJ-10-3 is a 2,935. Comparing the pH value of the sample S-SBJ-10-3 with the initial sample, there is an increase of 0.005. Sample S-SBJ-10-5 is 2,932; comparing its pH value with the initial sample, there is an increase of 0.002. Sample S-SBJ-30-1 is 2,946. Compared to the initial sample, the increase is 0.016. The sample S-SBJ-30-2 is 2.953, the increase from the initial sample is 0.023. Moreover, sample pH with code S-SBJ-30-3 is 2.965. sample S-SBJ-30-4 is 2.974.

In comparison with the initial sample, there is an increase of 0.044. Finally, the sample S-SBJ-

30-5 is 2,982. Comparing the pH value of the initial sample, there is an increase of 0.052. Therefore, the difference in the pH value per sample depends on the treatment of the sample, which is influenced by temperature and time.

4. CONCLUSIONS:

Based on the experimental data, parameters such as temperature and storage time influence the heavy metal lead (Pb) migration from the can container to contained orange juice drinks. In addition, the proper storage area for canned drinks is below 28 °C. It also should avoid direct sunlight and high humidity. In the storage, canned drinks should not be stored at temperatures above 38°C with continuous exposure due to the high migration of lead metal (Pb) as it is harmful to the drink quality. Overall, there is an insignificant change in the pH value among the measured samples.

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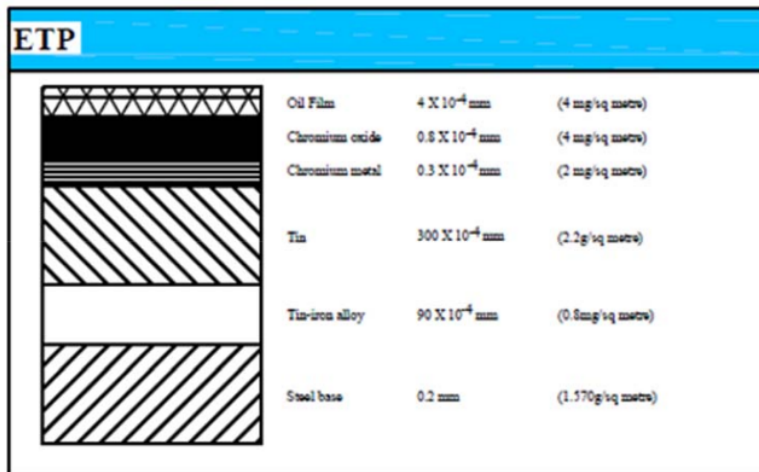


Figure 1. the layer arrangement of the electrolytic tin plate type packaging

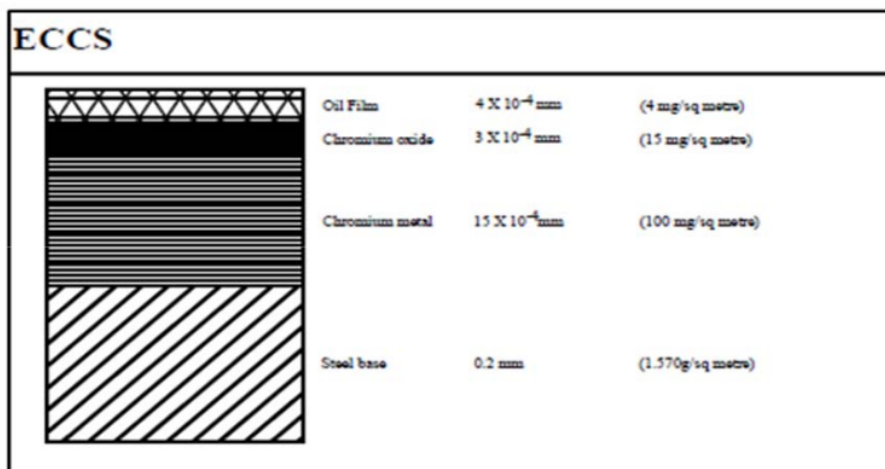


Figure 2. The layer arrangement of the electrolytic chromium-coated steel cans

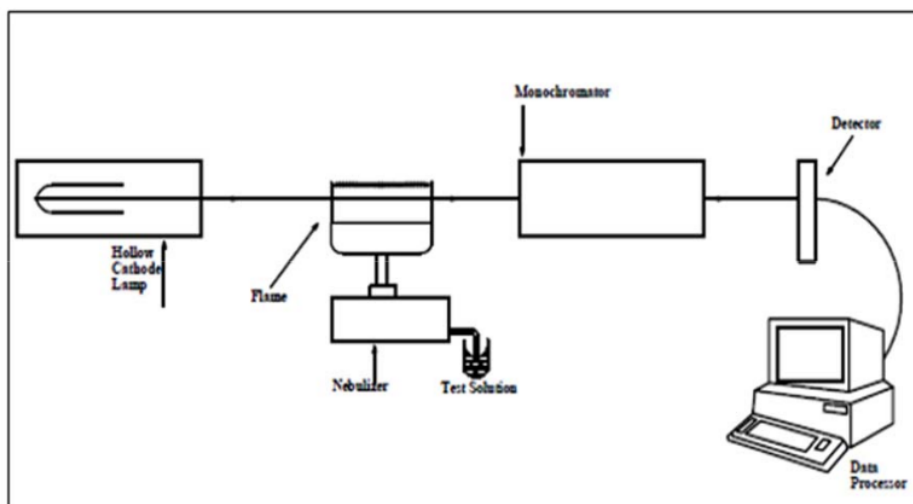


Figure 3. Schematic Diagram Atomic Absorption Spectrophotometry

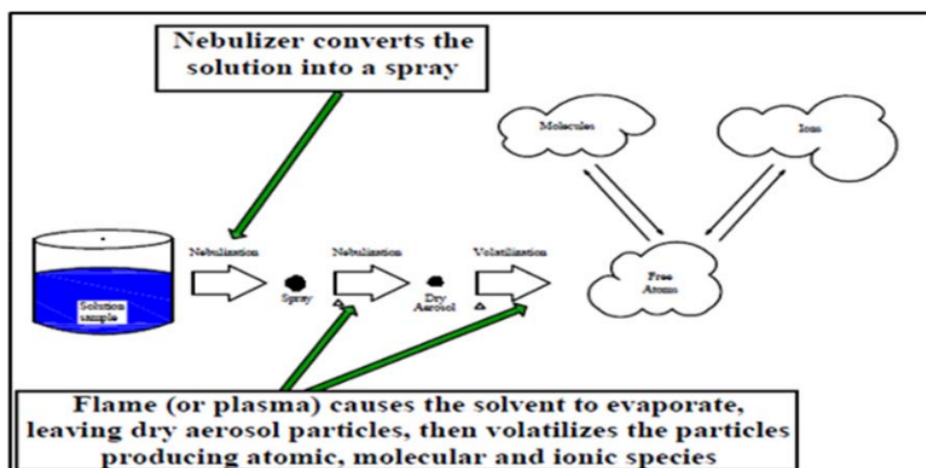


Figure 4. Atomic Spectroscopy

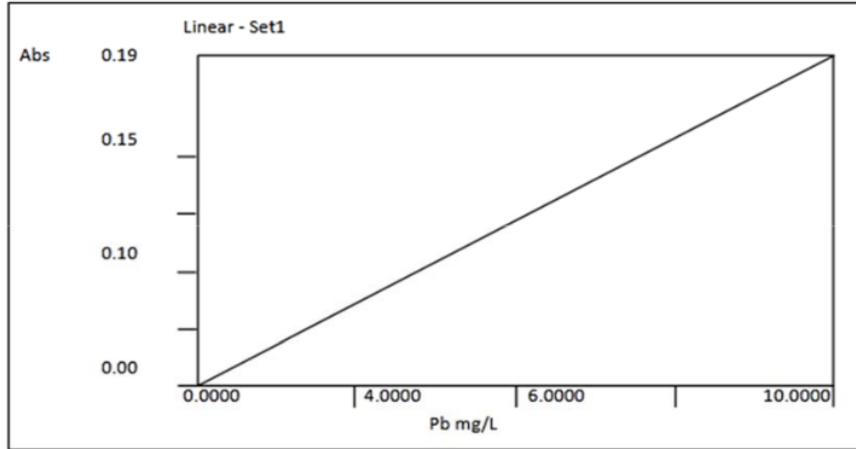


Figure 5. Lead (Pb) Validation standard curve graph

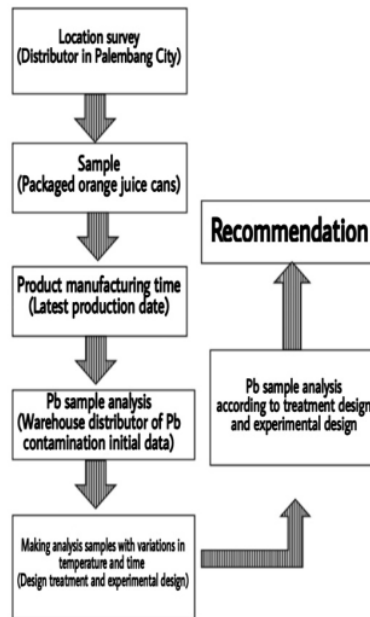


Figure 6. Research Flowchart

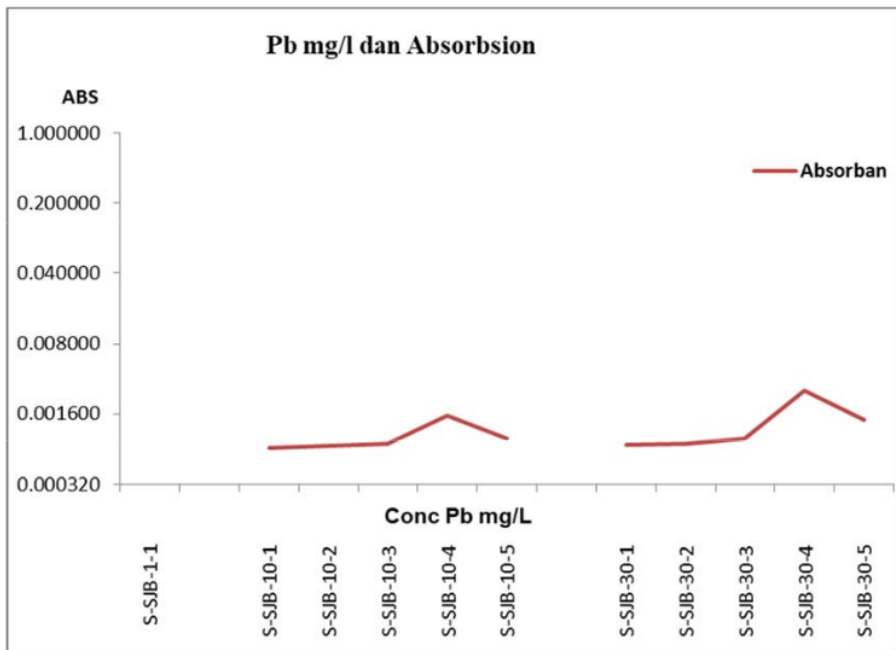


Figure 7. The concentration of Pb to Absorption

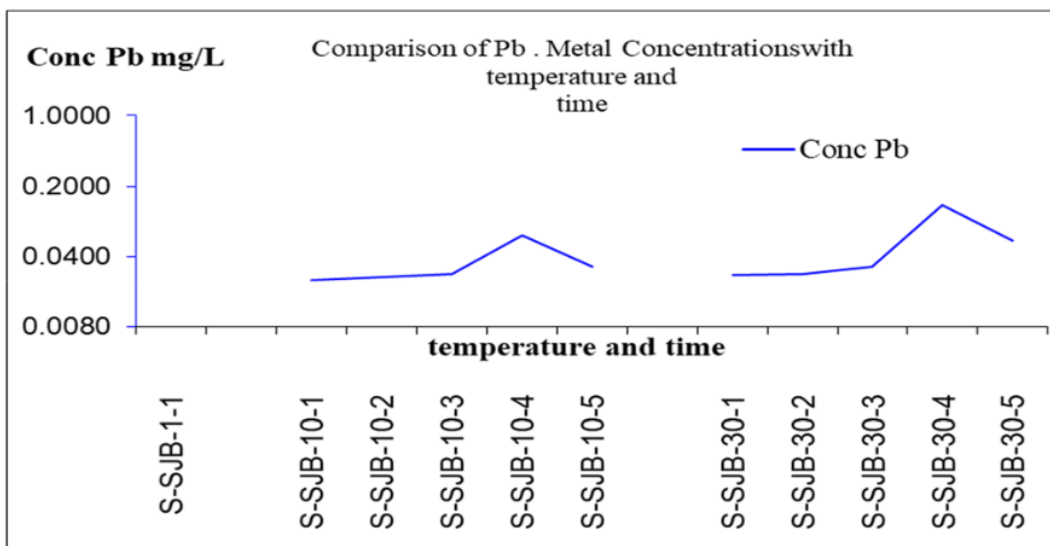


Figure 8. Effect of temperature and time on Pb concentration in the sample

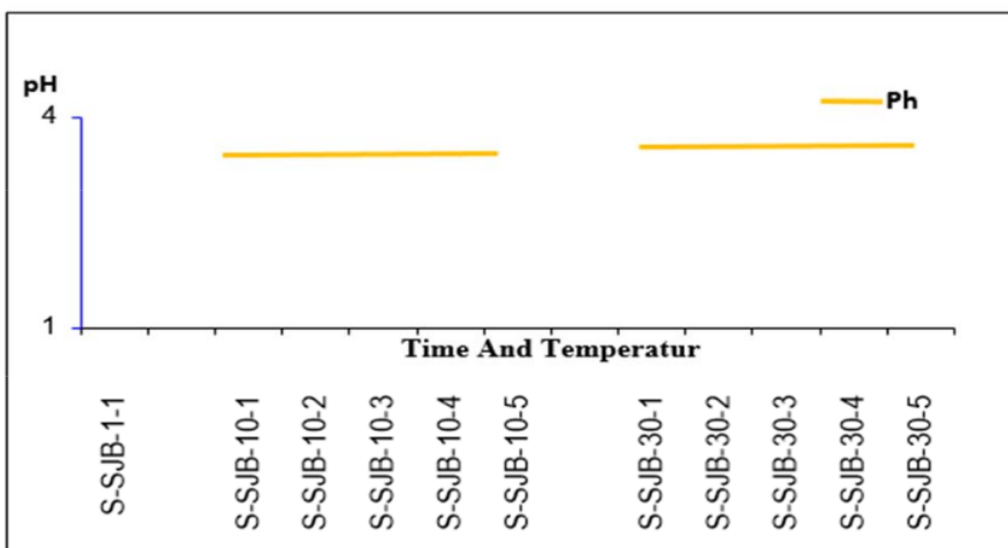


Figure 9. Sample pH Analysis Results

THE EFFECT OF TEMPERATURE AND TIME ON THE MIGRATION OF LEAD METAL (PB) IONS IN CANNED ORANGE JUICE DRINK USING ATOMIC ABSORPTION SPECTROPHOTOMETRY ANALYSIS

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