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Fabrication of mercury (Hg) sensor based on Tire Waste (TW) carbon electrode and voltammetry technique

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Abstract
Voltammetry is widely used to detect heavy metals such as mercury (Hg). The sensor material influences the results of the voltammetry method. Carbon-based sensors are commonly developed because of their wide potential range, low background current, inexpensive, inert, and suitable for various sensors. Recently, Tire Waste (TW) was chosen as a carbon source for the manufacture of electrodes because it is rich in carbon (88%). The material is easy to obtain, green technology and a carbon source that has not been properly utilized. Separation of carbon material from TW using the pyrolysis method produces 4.32 gr (2.6%) of soot (from 200 gr TW) as a material for making carbon-based Hg sensors. The XRD pattern of TW soot has amorphous phases. SEM topography shows that the surface of TW soot consists of particles that are almost uniform in shape. The estimated particle size is about 0.25 µm. The sensor was made with a mixture of TW soot and paraffin with a 2:1 ratio. Testing the specific value of sensor capacitance using the Cyclic Voltammetry (CV) method showed the presence of cathodic and anodic currents. The Hg deposit occurred at a peak cathodic current at a scan rate of 20 mV/s. The Limit of Detection (LoD) value is 0.0681 ppm and the Limit of Quantitation (LoQ) is 0.229 ppm. Measurement of Hg in natural water samples used river water from the Aceh Jaya sub-district at two points representing the upstream and downstream of the river. The Hg concentrations obtained were 0.000536 ppm and 0.00182 ppm, respectively and were compared with the inspection results using Atomic Absorbance Spectroscopy (AAS), 0.00058 ppm and 0.00186 ppm, respectively. The t-test results of the two Hg measurement methods at a significance level (α) of 5% obtained tcount > ttable (0.0208 <2.306), indicating that there is a significant difference between the two Hg concentration measurement methods for natural water.

Keywords:
Cyclic Voltammetry;
Hg sensors;
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INTRODUCTION

Mercury and its compounds are considered as very dangerous pollutants. Environmental contamination by mercury has become a global problem and mercury polluted areas have been identified worldwide. In most cases, release of mercury oxide (HgO) or mercury ions (Hg^{2+}) to the environment occurs due to industrial emissions, transportation, waste treatment or technological accidents [1].

Monitoring and analysis of mercury metal concentrations in the environment is necessary for pollution assessment and control. The development of reliable analytical methods that provide detection and quantification limits for the determination of mercury in environmental matrices is also increasingly needed [2].

Voltammetry is one of the electrochemical methods that are widely used to detect heavy metals such as mercury. Voltammetry is an electrochemical method that observes changes in current and potential [3][4]. Voltammetry techniques have been studied for use in the detection of several heavy metals such as Cd^{2+} [5], Pb^{2+} [6], Cu^{2+} [3] and Hg^{2+} [4][7]. Each of these studies discusses different sensor modifications. Several types of sensors such as platinum [8][9] and diamond-based sensors [10] show good performance, but both sensors are relatively expensive. Carbon-based sensors are currently being developed in voltammetry techniques because they have several advantages, namely a wide potential range, low background current, inexpensive, inert, and suitable for a variety of sensors [11].

Recently, Tires Waste (TW) was selected as a carbon source for making electrodes. In principle tires are very rich in elemental carbon (88%) and are considered as elastomeric compounds [12]. Tires Waste (TW) is a carbon source that has not been properly utilized because worldwide more than 1.2 billion TW is disposed of every year, and accumulates in landfills [13]. The Indonesian Tire Manufacturers Association (APBI) stated that 69 million motorcycle tires and 84 million car tires including commercial truck tires were sold in 2018. This causes serious disposal problems once the tires are used up. The same phenomenon also occurs throughout the world. Around 2.7 billion tires were produced globally in 2017 and only 1 billion were discarded [14]. Utilization of TW can be a promising green technology alternative.

Tire Waste (TW) can be classified as a low-cost source of renewable biomass energy, a source of sustainable and environmentally friendly materials [15]. Pyrolysis of TW at high temperatures in an oxygen-free (little oxygen) environment is considered a good technique for recovering up to 90% by weight of carbon residue, where the carbon residue can be further used for other purposes [12]. Incomplete combustion occurs due to a lack of oxygen needed to complete combustion. When fuel hydrocarbons react with oxygen from the air to produce CO_2 and H_2O as byproducts it is called stoichiometric combustion. Conversely, if more by-products are formed then the reaction is often referred to as non-stoichiometric combustion. Some of the carbon, during the chemical reaction of non-stoichiometric combustion, is converted to CO and ordinary carbon particles (soot). These soot particles form the basis for carbon nanostructures formed during the combustion process [16][17].

The main work in this research is to separate the carbon material (soot) from TW using the pyrolysis method [18], followed by making a carbon-based Hg sensor for the voltammetry method and measuring the sensor capacitance in the analysis of Hg in water. The research objectives were to obtain carbon material from TW, to characterize carbon material by XRD and SEM, to determine the electrochemical response to mercury in water by calculating LoD and LoQ and to test the Hg detection capability of the voltammetry method on natural water samples. This research is expected to be useful in providing information about one of the solutions in overcoming the problem of TW disposal, separating carbon from TW, producing a Hg sensor using the voltammetry method from TW carbon, knowing the detection limit of the sensor and the sensor's ability to detect Hg in natural water samples.

METHOD

Material

Samples of Tire Waste (TW) to obtain carbon material were obtained from a motorcycle tire replacement workshop in Lambaro Skep Village, Kuta Alam District, Banda Aceh City. The sample in this study was river water from the Aceh Jaya Regency, Aceh Province at sampling point I (River Water I) which is the upstream part of the river with coordinates N: 040 41' 16.1” E: 0950 41' 09.1” and at sampling point II (River Water II) which is the downstream part of the river with coordinates N: 040 36' 13.4” E: 0950 38', 32,5”.

Methods

This research will separate carbon material (soot) from TW using the pyrolysis method [19]. The pyrolysis reactor is a cylindrical aluminum container with a diameter of 15 cm and a height of 16 cm. The flame for pyrolysis comes from a portable can of Liquefied Petroleum Gas (LPG) (TOKAI GAS). The pyrolysis soot will be analyzed using X-Ray Diffraction (XRD) (SHIMADZU XRD-7000 model) to identify crystalline materials [20]. In the electrochemical process using Cyclic Voltammetry (CV), the surface structure of the substrate or electrode can affect the scanning rate [21][22], so that the soot resulting from pyrolysis will be analyzed with a Scanning Electron Microscope (SEM) (model JSM-6510LA). The resulting soot will be mixed with paraffin (Candle Wax) as a binder, then glued to copper wire (ETERNA) to make sensors. The sensor at treatment used HNO_3 0.1N (MERC) [23]. The sensor is then electrochemically tested to determine its specific capacitance value with CV using a potentiostat (ZIVE ppi) connected to a corder and equipped with Smart Manager (SM) software, using a three-electrode system (carbon electrode from TW as the sensor), Ag/AgCl electrode as reference electrode, and graphite as auxiliary electrode). Sensor response testing was carried out on standard Hg solutions and natural water samples, namely river water in Aceh Jaya Regency, Aceh Province using the CV method. In testing the sensor response also used a reagent solution of 0.1 M KCl as an electrolyte, acetate buffer pH 5.5 (a mixture of acetic acid (MERC) and Sodium Acetate (MERC)) as a pH buffer during the CV process and standard Hg solution (MERC) for to vary the concentration of the standard solution. Mercury detection results will be compared with measurements on the Atomic Adsorption Spectroscopy (AAS) instrument (SHIMAZU AA-7000). The parameters to be tested are sensitivity, Limit of Detection (LoD), Limit of Quantification (LoQ), water quality test and comparison of methods [3, 4, 24].

Pyrolysis of UTW

The TW was cleaned and air-dried, cut into many small fragments (1–3 cm) and weighed as much as 50 grams. Then it is burned using a flame from LPG gas in a closed furnace (to avoid contact with oxygen in the air) for 1 hours. The carbon material to be used is in the form of soot formed on the walls of the pyrolysis furnace. The soot was collected in a container covered with aluminum foil and weighed to determine the yield obtained. The soot was further characterized by XRD and SEM.

Making working electrodes (sensors) from UTW soot

A single copper cable 10 cm long was stripped at the end by 0.3 cm. The soot and paraffin were mixed in a 2:1 ratio in a mortar and heated to form a paste. The paste is printed with an insulating pipe (plastic) of the same diameter as 0.5 cm long copper wire. The exposed end of the copper wire is contacted with the printed carbon paste. The other end is peeled off to be connected to the instrument potentiostat. Prior to the experiment, the electrode surface was smoothed with a tissue paper until a shiny and clean electrode surface was obtained.

Sensor’s capacitance specific value testing

25 mL of 0.1M KCl solution was put into the sample container. The three electrodes (electrodes made of TW carbon as working electrodes (sensors), Ag/AgCl electrodes as reference electrodes, and graphite electrodes as supporting electrodes) are placed in the sample holder and immersed in 0.1 M KCl solution as presented in Figure 1. The potentiostat is operated with cyclic voltammetry parameters (Cyclic Voltammetry, CV), at a scan-rate of 100 mV/s in the range -1 – 1 Volt for 1 cycle to obtain a cyclic voltammogram image of a standard carbon electrode in 0.1 M KCl solution.

Sensor’s preparation

The sensor, reference electrode and auxiliary electrode are connected to a potentiostat device and CV is carried out in the area of -1.6 – (-0.6) V in 0.1 M HNO3 solution, using a scan rate of 50 mV/s for 60 cycles. After the CV process, all three were taken out, rinsed with aquabides and then dried with tissue paper. Formation of Hg(II) deposits by immersing the three electrodes in a solution of 0.002 ppm Hg(II) at pH 5.5 (0.1M acetate buffer), then carrying out a CV process with a scan-rate variation from 10 mV/s – 100mV/s , in the range -1.6 to -1.6 V with a midle of -1.1, each of 1 cycle. The scan rate with increasing cathodic current is the optimum scanrate.

Electrochemical Response of the Sensor to Hg

Standard Hg solution with a concentration of 0.001; 0.005; 0.01; 0.05; 0.1; 0.5 and 1 ppm in 0.1 M acetate buffer at pH 5.5 of 100 mL each was put into the sample container. The sensor is placed on the sample holder. CV measurements were carried out for each standard using a sensor at the peak oxidation voltage.

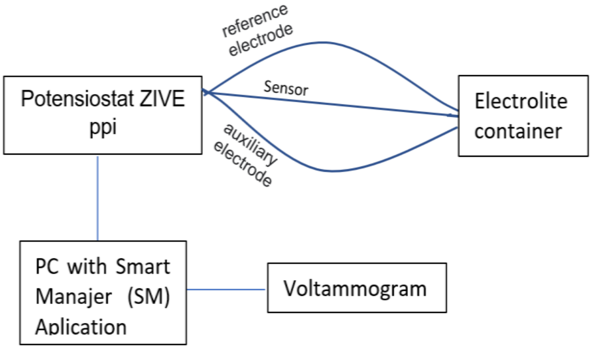

Figure 1. Diagram of the configuration of the three electrodes integrated into the potentiost

Mercury Sensor Testing on River Water Samples

The water sample is filtered using Whatman No.1 filter paper, the pH of the water is adjusted to pH 3 by adding 0.05 M acetic acid or 0.05 M sodium acetate. The water sample is used for dilution of Standard Hg from a concentration of 1 ppm to 0.05 ppm. Measurements were made using the CV method at the peak oxidation voltage of half a cycle.

RESULTS AND DISCUSSION

Pyrolysis of UTW

Pyrolysis of 200 grams of TW using LPG at a temperature of ±400 °C for 1 hour produced 4.32 grams of soot (2.6%) and 78.24 grams of charcoal (39.12%). The total percentage of soot and charcoal carbon material obtained in this study was 41.72%, more than previous studies which stated that there was 29% - 31% black carbon in TW [22].

In this study, soot was chosen as a material for making sensors because the nano size of soot causes the electrolyte to easily penetrate to the deepest part of the electrode, so that the total active ingredient in the electrode can be utilized optimally. Carbon soot is an effective, cheap and potential electrode material to replace commercial electrodes [23]. TW soot XRD diffractogram as shown in Figure 2, shows 3 peaks with intensities at angles around 36.04, 35.87 and 26.81 at 2θ. Based on a literature search, in the oil lamp soot XRD diffractogram there is a peak with intensity at an angle of about 26.24 [24] and in the candle soot XRD diffractogram there is a peak with intensity at an angle of 23.24 [25]. Oil lamp soot and candle soot are stated to have an amorphous phase. TW soot also has a peak with intensity at an angle of around 26.81 which is similar to the two soots. Based on these conditions, it can be estimated that TW soot has an amorphous phase such as oil lamp soot and candle

SEM analysis of TW soot showed almost uniform particle shape at 20,000x magnification, as presented in Figure 3).The approximate particle size compared to the 1 µm scale line is about 0.25 µm (250 nm). There is no research on the topography of TW soot, so it cannot be compared with previous studies.

Making working electrodes (sensors) from UTW soot and Sensor's capacitance specific value testing

Sensors with a ratio of soot: paraffin 2:1, as depicted in Figure 4, the consistency of the mixture is denser but still suitable for printing and connected with copper wire. Next, testing the specific value of the sensor capacitance, as shown in Figure 5.

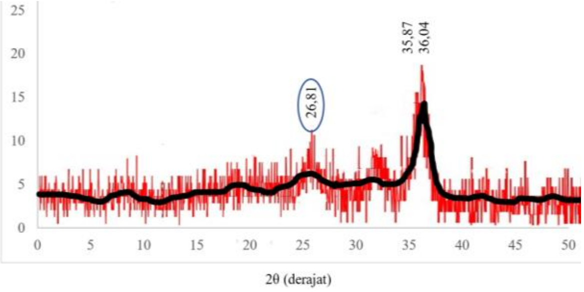

Figure 2. XRD diffractogram of Tires Waste (TW) soot at 2θ, the circled angles (26.81) are similar to those found for oil lamp soot (26.24) ang candle soot (23.24) at 2θ

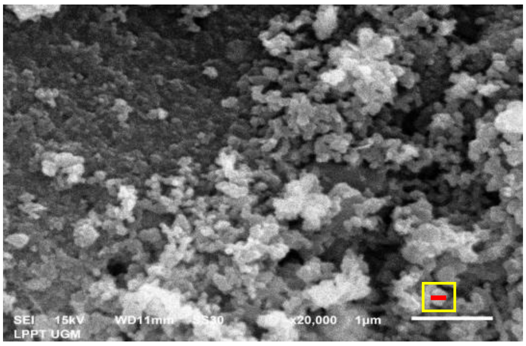

Figure 3. Tire Waste (TW) soot surface topography the estimated particle size marked with a yellow square is given the diameter of the red line which is approx. 0.25 µm (250 nm)

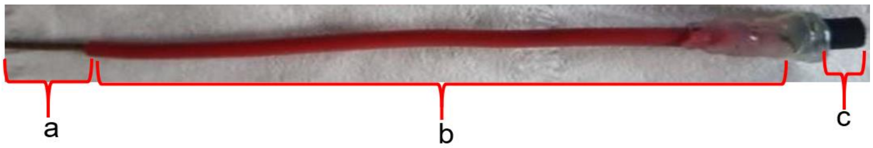

Figure 4. Mercury (Hg) sensor based on Tires Waste (TW): a. Stripped end of cable (0.3 cm) to connect potentiostat instrument, b. The length of the copper cable is 9.2 cm, c. Paste of a mixture of TW soot and paraffin (2:1) 0.5 cm

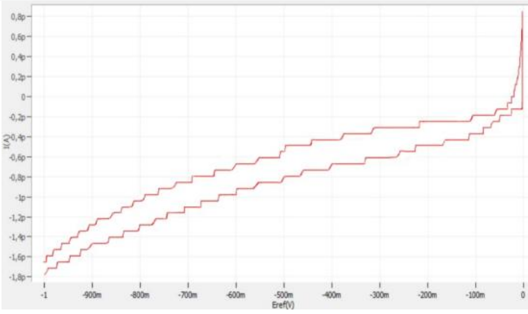

Figure 5. Cyclic voltammogram sensor with a ratio of soot: paraffin 2:1 in 0.1 M KCl solution, at a scan-rate of 100 mV/s in the range -1 – 1 Volt for 1 cycle

Sensor’s preparation

The results of the potential optimization test obtained an increase in cathodic current at a scant-rate of 20 mV/s. Comparison of voltammogram scan rates of 10, 20 and 30 mV/s which can be seen in Figure 6.

At a scant-rate of 20 mV/s there is an increase in cathodic current, thus a scant-rate of 20 mV/s is chosen as the optimum scant-rate for Hg measurement.

Electrochemical Response of the Sensor to Hg

The electrochemical response of the sensor to Hg in water CV method uses a standard Hg concentration of 0.001; 0.005; 0.01; 0.05; 0.1; 0.5 and 1 ppm in 0.1 M acetate buffer at pH 5.5. The electrochemical response voltammogram can be seen in Figure 7 with the cathodic peak current (Ipc) shown in Table 1.

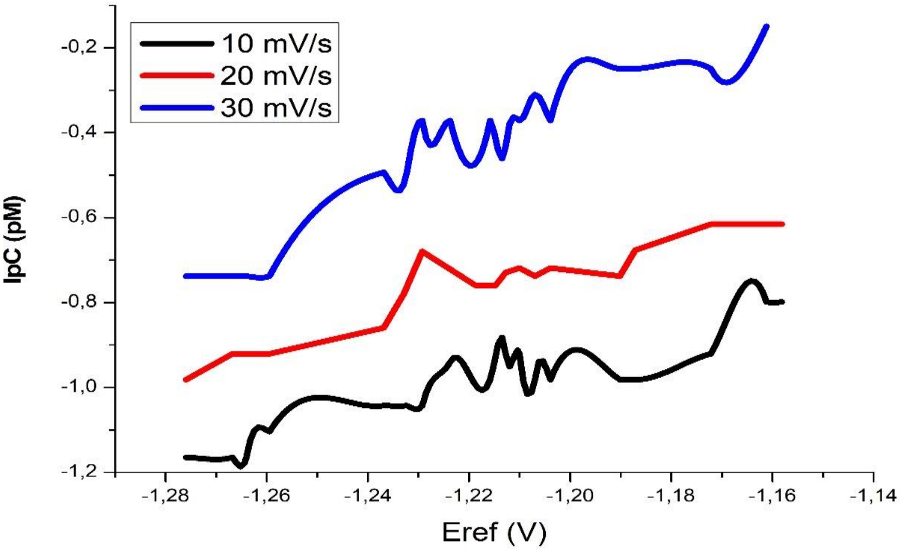

Figure 6. Cycli voltammogram optimization of the scant-rate of the UTW carbon sensor, scan-rates of 10, 20 and 30 mV/s.

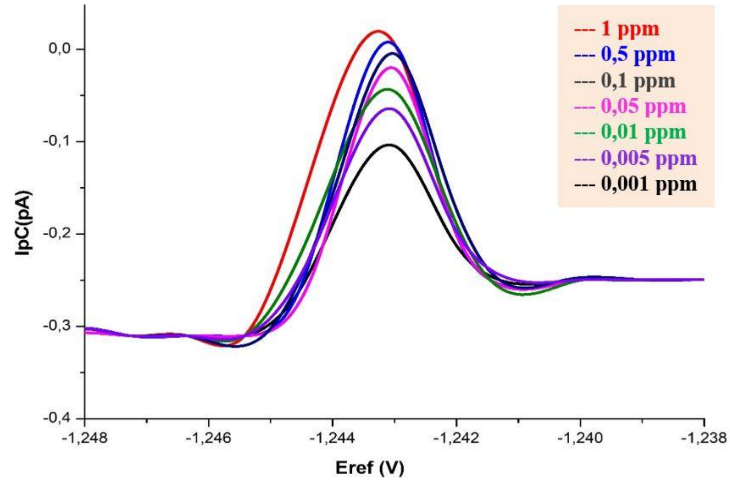

Figure 7. Voltammogram Standard Hg concentration 0.001 - 1 ppm in 0.1 M acetate buffer at pH 5,5

Table 1. Cathodic peak current (Ipc) for standard Hg Concentration 0.001; 0.005; 0.01; 0.05 and 0.1ppm

Concentration Hg (ppm)	Ipc (pA)
0.1	-0.898473
0.05	-0.964535
0.01	-1.22591
0.005	-1.28675
0.001	-1.30887

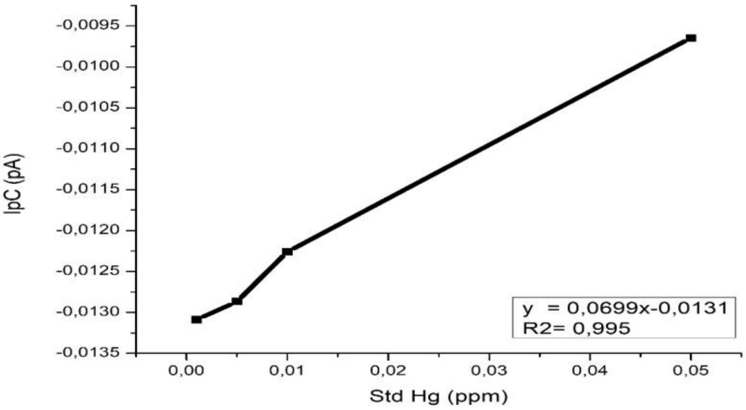

Figure 8. Linear regression curve of Standard Hg concentration (0.001-0.05 ppm) vs IpC (pA)

Based the linear regression curve, a linear regression equation is obtained y = 0.069x-0.013 with a value of R2 = 0.995 (r = 0.997), the value of Standard Deviation (SD) is 1.61 x10-12 and the percentage of Relative Standard Deviation (RSD) is 13.5%. LoD is calculated using the equation LoD = 3 s/m and LoQ is calculated using the equation LoQ = 10 s/m, where 's' is the average standard deviation and 'm' is the slope of the calibration curve [3][4]. As a result, the LoD and LoQ values of the carbon-based Hg sensor from Tire Waste (TW) are estimated to be 0.068 ppm and 0.229 ppm.

Mercury Sensor Testing on River Water Samples

The water sample containing 0.05 ppm standard Hg was then measured for Hg concentration using the UTW carbon-based Hg sensor with the CV method at a peak oxidation voltage of 20 mV/s for half a cycle. The concentration of Hg in the sample using the linear regression equation obtained from River I and II were 0.000536 ppm and 0.00182 ppm respectively. Furthermore, as a comparison, the water samples were also tested with AAS and the concentration of Hg in River Water I and River II samples were 0.00058 ppm, 0.00186 ppm, respectively. Then the t test between the two methods was carried out, ttable was obtained by determining the confidence interval at 95%, the significance level (α) was 5% (0.05). The results of the t test at a significance level (α) of 5% obtained tcount > ttable (6.037 <2.262), which shows that there is no significant difference between the two methods of measuring Hg concentrations for river water.

CONCLUSION

The carbon material (soot) that was successfully separated from 200 grams of TW was 4.32 grams (2.6%). Based on the XRD diffractogram, TW soot is estimated to have an amorphous phase. Based on SEM topography the estimated particle size of TW soot is about 250 nm. The suitable ratio of soot: paraffin for the manufacture of TW carbon-based Hg sensors is 2:1. The electrochemical response of the UTW carbon electrode-based voltammetry sensor to mercury in water gave LoD and LoQ values of 0.068 ppm and 0.229 ppm, respectively. The t-test results of CV with TW carbo-based sensors and AAS for measuring Hg in river water at a significance level (α) of 5% obtained $t_{count} > t_{table}$ (6.037 <2.262), which shows no significant difference between the two-concentration measurement methods Hg for river water.

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