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Influence of $NaBH_4$ on the sensitivity of As^{3+} and As^{5+} sensor using gold modified boron doped diamond electrodes

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ABSTRACT

Background: Arsenic is known as one of the carcinogenic metalloids and can cause various health issues when ingested or inhaled over prolonged periods of time. **Methods:** In this work, boron-doped diamond (BDD) electrode was altered with gold particles (Au) arranged by seeding continued with electrodeposition of $HAuCl_4$ solutions at the electrode surface, will be used as electrode to detect As^{3+} and As^{5+} in lake water. The deposited gold particles on the BDD surface were studied with scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS) and X-ray photoelectron spectroscopy (XPS). Detections of As^{3+}, As^{5+}, and mixture solutions of As^{3+} and As^{5+}, carried out with anodic stripping voltammetry (ASV). **Findings:** The, pre-treatment using $NaBH_4$ carried out for reduction from As^{5+} to As^{3+}, indicate an improvement at the sensitivity of As^{3+} and As^{5+} detection with a good linear responses for each solution in range concentrations of 0.02-0.2 ppm for As^{3+} and As^{5+}, with R^2=0.9759 and R^2= 0.9876, respectively. **Conclusion:** Furthermore, limit of detections of 0.0335 ppm and 0.0239 ppm can be attained for As^{3+} and As^{5+} displayed high linearity, revealing that detection of each species of As^{3+} and As^{5+} can be conducted in mixture of As^{3+} and As^{5+}. **Novelty/Originality of this Study:** This study involves the modification of BDD electrodes with gold (Au) using a combined seeding and electrodeposition technique, which enhances stability and sensitivity for detecting arsenic (As^{3+} and As^{5+}) at low concentrations. Additionally, the research introduces a pretreatment method using $NaBH_4$ to facilitate the detection of As^{5+} by reducing it to As^{3+}, thereby improving the detection limits with anodic stripping voltammetry (ASV).

KEYWORDS: As^{3+}; As^{5+}; Au; anodic stripping voltammetry; boron doped diamond.

1. Introduction

Arsenic is a very toxic compound and is widely distributed in nature. This existence in groundwater causes serious problems. Two categories of arsenic are found in the environment, an inorganic and organic arsenic, in which, inorganic arsenic is more toxic than organic arsenic. Inorganic arsenic contamination in groundwater, if it exceeds the threshold, can cause various diseases such as cancer and can also cause death. Based on the World Health Organization (WHO) and the US Environmental Protection Agency (US EPA), the highest threshold for arsenic in drinking water is 10 ppb (Fatoki et al., 2022).

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Thus, it is very important to make a sensitive sensing device to detect arsenic in a low level of concentration.

Meanwhile, boron-doped-diamond (BDD) electrodes are widely applied in electrochemical methods and can be used for sensor application, especially to detect arsenic compounds (Ivandini et al., 2010). BDD holds excellent electrochemical characteristics, namely wide potential window, low background current and high chemical and physical stability, that is very suitable for sensor application (Ivandini et al., 2017; Jiwanti et al., 2022). Numerous research publications on the application of BDD electrode for sensor application has been reported, such as for detecting glucose (Zheng et al., 2019), ciprofloxacin (Matsunaga et al., 2020), ofloxacin (Jiwanti, et al., 2023), zanamivir (Ivandini et al., 2017), and many more. To improve the catalytic activity as well as selectivity, metal nanoparticles modification on the surface of BDD electrodes is performed (Jiwanti, et al., 2023; Yamada et al., 2008). Previously, modifying the BDD electrode with gold (Au) particles (BDD-Au) to detect As^{3+} and As^{5+} has been carried out by several researchers (Hamid Kargari et al., 2023; Ivandini et al., 2006; Yamada et al., 2008). Detection of As^{3+} and As^{5+} by Yamada et al (Yamada et al., 2008) showed detection limit results of 5 ppb for As^{3+} and 100 ppb for As^{5+}. But the size and densities are uncontrolled. The limit for levels of arsenic compounds in drinking water sources is 10 ppb based on WHO and US EPA. Therefore, a pretreatment method with $NaBH_4$ is needed to facilitate the detection of As^{5+} using BDD-Au electrodes with the anodic stripping voltammetry (ASV) technique.

In this current research, BDD electrodes were modified with gold (Au) using 2 steps, were seeding and electrodeposition techniques. It is expected to enhance the stability to determine arsenic (As^{3+} and As^{5+}). The seeding technique is planting a metal core or precursor on the electrode surface by physical adsorption (Zhang et al., 2021). This method has a binding metal precursor to the electrode surface using physical adsorption, then the chemical reduction used $NaBH_4$ (Gao et al., 2013). Through this technique, the embedded metal particles are expected to be distributed homogeneously on the matrix surface. In the final stage, an electrochemical electrodeposition process is carried out. In this process, metal will be deposited on a metal core that is embedded with a controlled particle size during electrodeposition. This process is then continued with chemical reduction of the As^{3+} and As^{5+} compounds using the $NaBH_4$ reagent. BH^{4-} ions will be adsorbed on the electrode surface to form nucleation for metal binding, making it easier to detect As^{3+} and As^{5+} in low concentrations. Analysis of As^{3+} and As^{5+} in original samples was carried out simultaneously in lake water.

2. Methods

2.1 Materials and instruments and preparation of gold modified BDD electrodes

The materials used for this research were $NaAsO_2$ (>90%), $Na_2HAsO_4.7H_2O$ (99%), HCl, $NaBH_4$ (95%), aquabidest, $HAuCl_4.4H_2O$ (99.9%), NaOH (97%) and 2-propanol (98%) purchased from Wako Pure Chemical Ind. All compounds were used without further purification. Meanwhile, BDD electrodes were synthesized using microwave plasma-assisted chemical vapor deposition (MPA-CVD) with B/C of 1:1000, Pressure of 85 Torr, and Plasma Power of 6000 W. The process of seeding Au particles on the BDD-H electrode surface was carried out by dropping 1 M $NaBH_4$ in 10 mL of 0.1 M NaOH then dropping 40 mL of 2.4 mM $HAuCl_4$ solution. This process is left for 24 hours until the solution dries. To remove excess NaBH4 and impurities, it was rinsed with aquabidest and dry with N_2 gas. The seeding process was carried out three times (Gao et al., 2013).

Furthermore, the deseeded BDD-Au was electrodeposited with 5 mL of 2.4 mM $HAuCl_4$ solution. Electrodeposition using a deposition potential at -0.2 V, with a deposition time of 1 to 5 minutes. The electrochemical cell equipment used is the BDD electrode as the working electrode, spiral-shaped Pt as the counter electrode, Ag/AgCl as the reference electrode. The table of deposition times carried out using multiple pulse amperometry (MPA) is shown in Table 1.

Table 1. Deposition time for electrodeposition Au with MPA

Deposition time (minutes)	Steps	Width
1	600	100
2	1000	120
3	1200	150
4	1600	150
5	2000	150

2.2 BDD-Au electrode as a As^{3+} an As^{5+} sensor and real sample application

Scan rate measurements were carried out for the detection of $As^{3+}$and As^{5+} by varying scan rates of 0.05–0.4 V/s using the ASV technique with deposition time for As^{3+} was 120 seconds, and As^{5+} was 180 seconds, with deposition potential of -0.5 V. Before being measured with ASV, the As^{3+}species were reduced with 0.05 M $NaBH_4$, and the As^{5+} species were reduced with 0.1 M $NaBH_4$.

Boron-doped diamond (BDD) electrodes that have been modified with gold (Au) were tested in each of 1 ppm $As^{3+}$and As^{5+}solutions nine times. Measurements were carried out at a deposition time of 120 seconds for As^{3+} and 180 seconds for As^{5+}, a deposition potential of -0.5 V, and a scan rate of 0.2 V/s. The As^{3+} species were reduced first with 0.05 M $NaBH_4$ and the As^{5+} with 0.1 M $NaBH_4$.

The solution mixture consisting of $As^{3+}$and As^{5+} species was measured by comparing the concentrations of the two species in a fixed volume (5 mL). The ratio of $As^{3+}$and As^{5+} solutions is 1:2, 1:3, 1:4, and 1:5 v/v. The concentrations used were 0.2 ppm - 1 ppm. Before measurements, As^{3+} and As^{5+} species were reduced with 0.1 M $NaBH_4$. Then, measurements were made using the ASV technique at a deposition time of 60 seconds, a deposition potential of -0.5 V, and a scan rate of 0.2 V/s.

The real sample was taken from the lake water from Universitas Indonesia using a random sampling method. Then the lake water is filtered and the filtered solution is used as a test sample. The test was carried out by adding samples of As^{3+} and As^{5+} of 10 ppm to the test sample with a mixture volume of 5 mL. Before measurements, As^{3+} and As^{5+} species were reduced with 0.1 M $NaBH_4$. Then measurements were made using the ASV technique at a deposition time of 60 seconds, a deposition potential of -0.5 V, and a scan rate of 0.2 V/s.

3. Results and Discussion

3.1 BDD-Au electrode using the seeding technique

In this research, BDD electrodes were modified with gold (Au) using the seeding technique (Gao et al. 2013). The process of adding Au particles by electrochemistry was carried out at a constant potential at -0.2 V in a 2.4 mM $HAuCl_4$. A potential of -0.2 V was used after considering the hydrogen evolution reaction in the acid solution and the deposition rate of Au particles on the BDD electrode surface. The amount of deposited Au particles can be monitored by charge consumption during the electrochemical growth process. The reduction reaction that occurs between $HAuCl_4$ and $NaBH_4$ is as follows (Gao et al., 2013):

$$HAuCl_4 + NaBH_4 \rightarrow Au + B(OH)_3 + NaCl + 3Cl^- + 2H^+$$

Based on the reaction above, the chemical formation of Au particles on the surface of the BDD electrode is based on the $NaBH_4$ adsorption control process. The mechanism that occurs includes electrostatic interactions between Au0 and negative polarization of the BDD surface and specific adsorption of $NaBH_4$ at different orientations of the BDD surface.

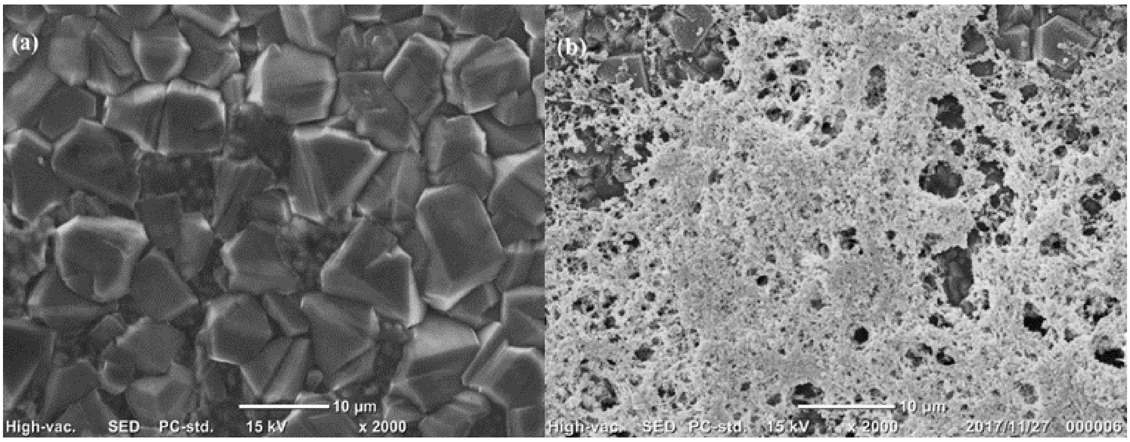

Fig. 1. BDD electrode morphology at 2000x magnification (a) without modified Au, and (b) modified Au

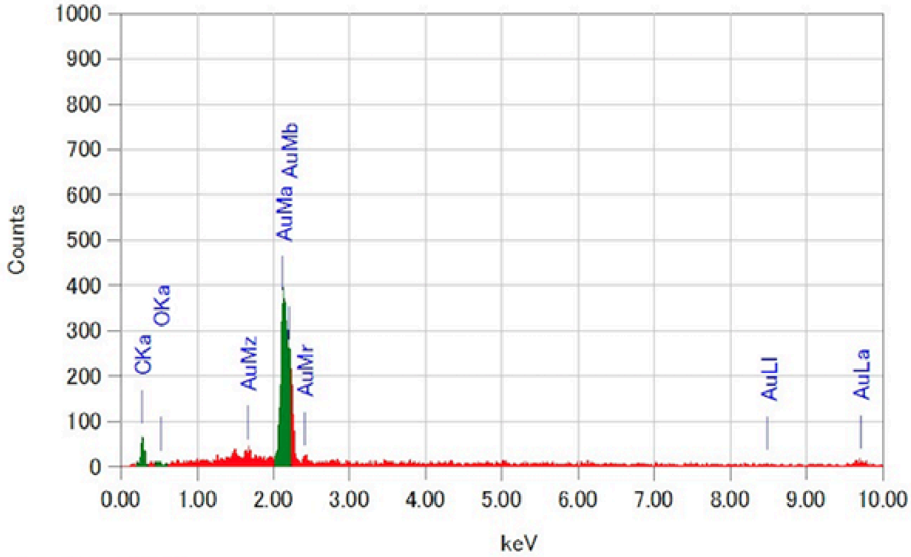

Fig. 2. Spectrum of elemental composition on the surface of the BDD-Au electrode

The prepared BDD-Au electrodes were characterized using scanning electron microscopy-energy dispersive spectroscopy (SEM-EDS). Figure 1 displays the morphology of BDD electrodes without Au modification and with Au modification using SEM. SEM characterization shows that there are Au particles that can be deposited and distributed on the BDD surface. The size of the Au particles deposited on the BDD surface is influenced by the repetition of the seeding process resulting in a buildup of Au particles. This is possibly due to the large concentration of $HAuCl_4$used so that an Au film forms which coats the surface of the BDD electrode, as proven by EDS characterization which shows an Au/C weight ratio of 9:2. Figure 2 shows the overlapping energy peaks of the Au element in the M shell with 2.121 keV, C in the K shell with 0.277 keV, and O in the K shell with 0.525 keV. The deposited Au is confirmed by EDS characterization.

After that, the BDD-Au electrode was studied using X-ray photoelectron spectroscopy (XPS). In Figure 3, showing three bond energy peaks identified on the BDD-Au electrode. At binding energies of 84 and 87.2 eV, it shows the presence of Au metal (4f7/2 and 4f5/2), at 285 eV it shows the C-C bond on the BDD electrode, and at 531 eV it shows the C-O bond resulting from the oxidation of the C-H bond on the BDD surface. Common C-H bonds are common on the surface of BDD because BDD is synthesized in a hydrogen atmosphere.

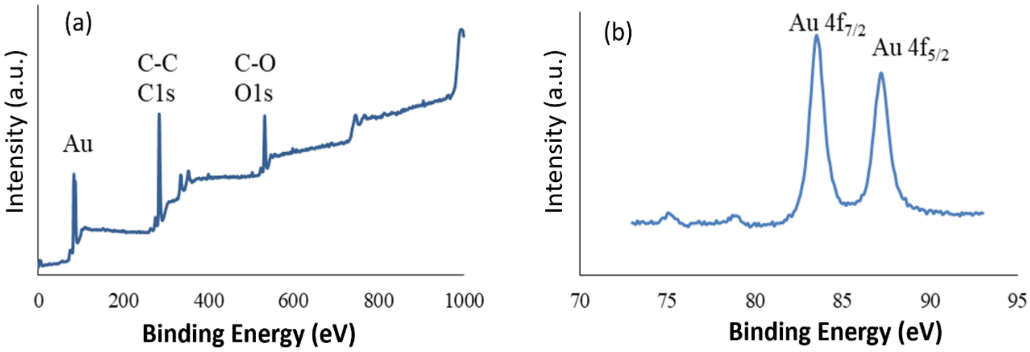

Fig. 3. XPS spectra of BDD-Au electrode (a) wide scan (b) narrow scan of Au

3.2 Pretreatment As^{3+} and As^{5+} with $NaBH_4$

The arsenic detection process on the BDD-Au electrode involves an oxidation reaction. Metal nanoparticles, such as Au, have the ability to oxidize arsenic which produces changes in electrical signals which are then measured as changes in current or potential. However, detection of As^{5+} tends to be difficult at low concentrations, therefore pretreatment must be performed, namely reducing As^{3+} and As^{5+} with the reducing agent $NaBH_4$. This process is carried out to facilitate measurements at low deposition potential. Figure 4 shows the stripping voltammogram of variations in concentrations of 0.2 to 1 ppm As^{3+} and As^{5+} in 0.1 M HCl with and without pretreatment with $NaBH_4$. From the stripping voltammogram, the As^{5+} oxidation peak can be seen at a potential of around 0.4 V. The intensity of the oxidation peak shows differences when pretreatment is performed and when pretreatment is not performed. The current oxidation peak in As^{3+} measurements became higher after pretreatment and the oxidation peak of As^{5+} which was reduced to As^{3+} was identified at a potential of around 0.22 V.

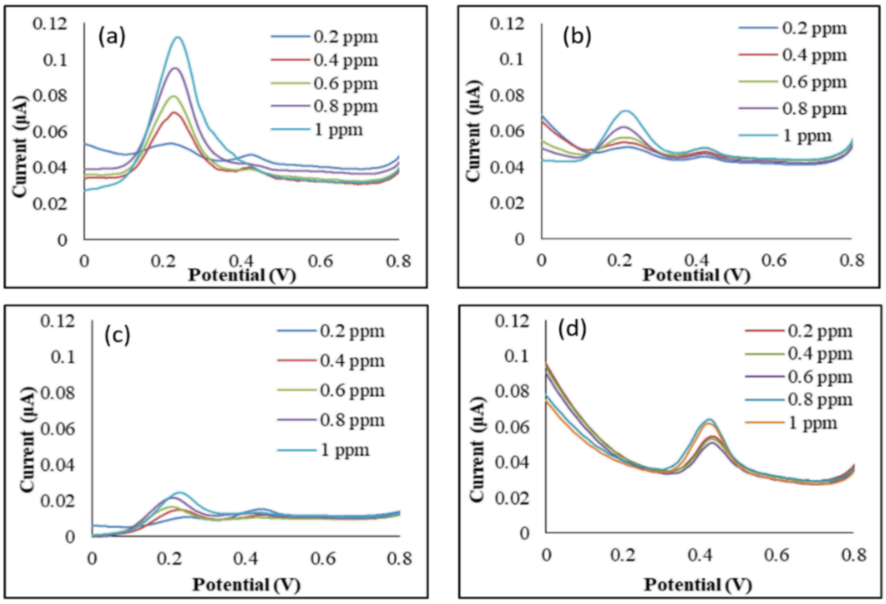

Fig. 4. Comparison of stripping voltammograms of (a) As^{3+} with $NaBH_4$, (b) As^{3+} without $NaBH_4$, (c) As^{5+} with $NaBH_4$, and (d) As^{5+} without $NaBH_4$

The concentration of $NaBH_4$ used to reduce As^{3+} and As^{5+} was optimized to ensure that all As^{3+} and As^{5+} species were reduced. The variations in $NaBH_4$ concentration used were 0.01, 0.05, and 0.1 M. The concentration of As^{3+} and As^{5+} used was 1 ppm. The result obtained indicates that the optimum concentration for reducing As^{3+} is 0.05 M while for reducing overall As^{5+} is 0.1 M. This may occur because the $NaBH_4$ used reduces As^{3+} to As^0.

A further reduction to AsH_3 is estimated to occur slowly so that 0.05 M is sufficient to reduce all As^{3+}. On the other hand, As^{5+} is reduced to As^{3+} which can then continue to be reduced to As0. The reduction of As^{3+} to As^0 requires more $NaBH_4$ so that the current response continues to increase with increasing concentrations of $NaBH_4$.

3.3 Application of BDD-Au electrode as a As^{3+} and As^{5+} sensor

Scan rate measurements are carried out with various scan rate variations, this was performed due to the greater the scan rate used, the greater the current produced. Determination of the optimum scan rate was carried out at various scan rates of 0.05, 0.1, 0.2, 0.25, and 0.4 V/s with measurement conditions in the potential range of 0 to 0.8V, deposition time of 120 seconds and deposition potential of -0.5 V. Figures 5 (a – b) shows that the As^{3+} and As^{5+} peaks current increase as the scan rate increases and shift toward a more positive potential. Both the measurements, at a scan rate of 400 mV/s, give the highest current. In Figure 5 (c – d), the R2 values obtained 0.998 and 0.968 for As^{3+} and As^{5+}, respectively.

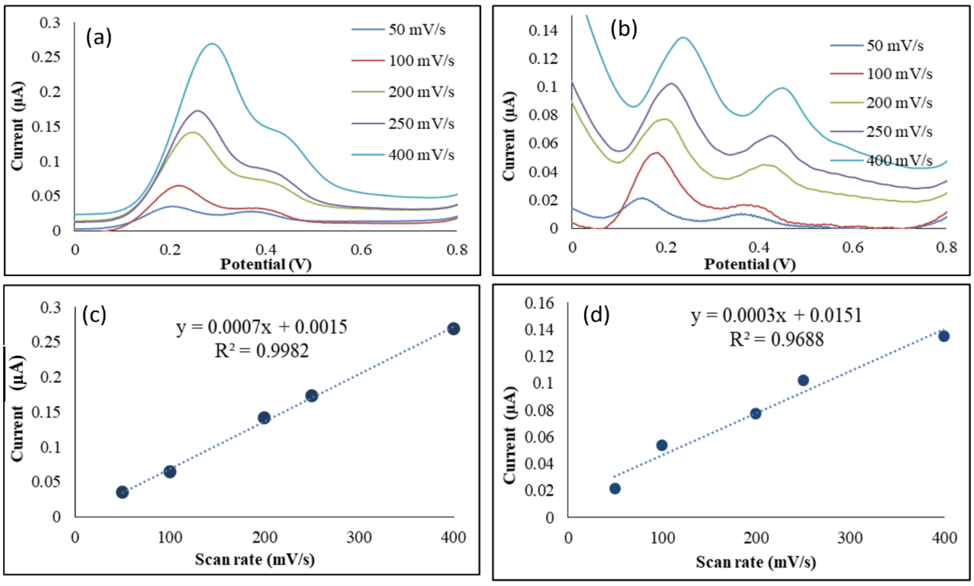

Fig. 5. Stripping voltammograms of (a) As^{3+} species and (b) As^{5+}at various scan rates. Plot of current response vs scan rate of (c) As^{3+} and (d) As^{5+}

3.4 Performance of prepared electrode to determine the concentration of As^{3+} and As^{5+}

Determination of the reproducibility of the As^{3+} and As^{5+} species was performed nine times in the potential range of 0 to 0.8 V (vs. Ag/AgCl) with optimum measurement conditions at deposition time of 120 s, deposition potential of -0.5 V, and scan rate of 0.2 V/s. Figure 6a, c shows the stripping voltammogram of the As^{3+} species and the current response to the number of measurements with BDD-Au. The measurement results obtained a relative standard deviation (RSD) value for As^{3+} of 2.7501%, which shows that the repeatability value of the oxidation current measured with the BDD-Au electrode is quite precise. Figure 6b, d show the stripping voltammogram of the As^{5+} species and the current response to the number of measurements with BDD-Au. The RSD value for As^{5+} is 3.7313%, which shows that the precision value is sufficient for the repeatability of the oxidation current measured with the BDD-Au electrode.

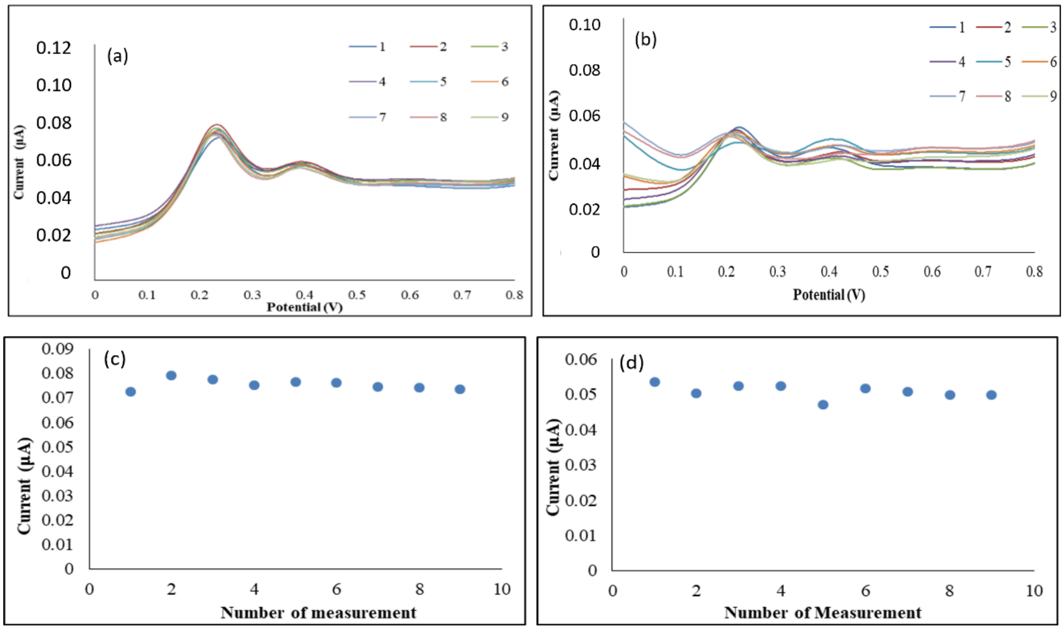

Fig. 6. (a) Stripping voltammogram of As^{3+} for nine measurements with a BDD-Au electrode (b) Stripping voltammogram of As^{5+}(c) Plot of current response of As^{3+} and (d) Plot of current response of As^{5+} to the number of measurements on the BDD-Au electrode

3.5 Analysis of As^{3+} and As^{5+} mixed solution

Measurements of mixed solutions containing As^{3+} and As^{5+} were carried out at various concentrations in fixed volumes. Measurements were carried out in the potential range of 0 to 0.8 V (vs Ag/AgCl) with conditions of 60 seconds deposition time, deposition potential -0.5 V, and scan rate 0.2 V/s and $NaBH_4$. The concentration used is 0.1 M with As^{3+} remaining at 0.2 ppm and variations in As^{5+}, and vice versa (Figure 7).

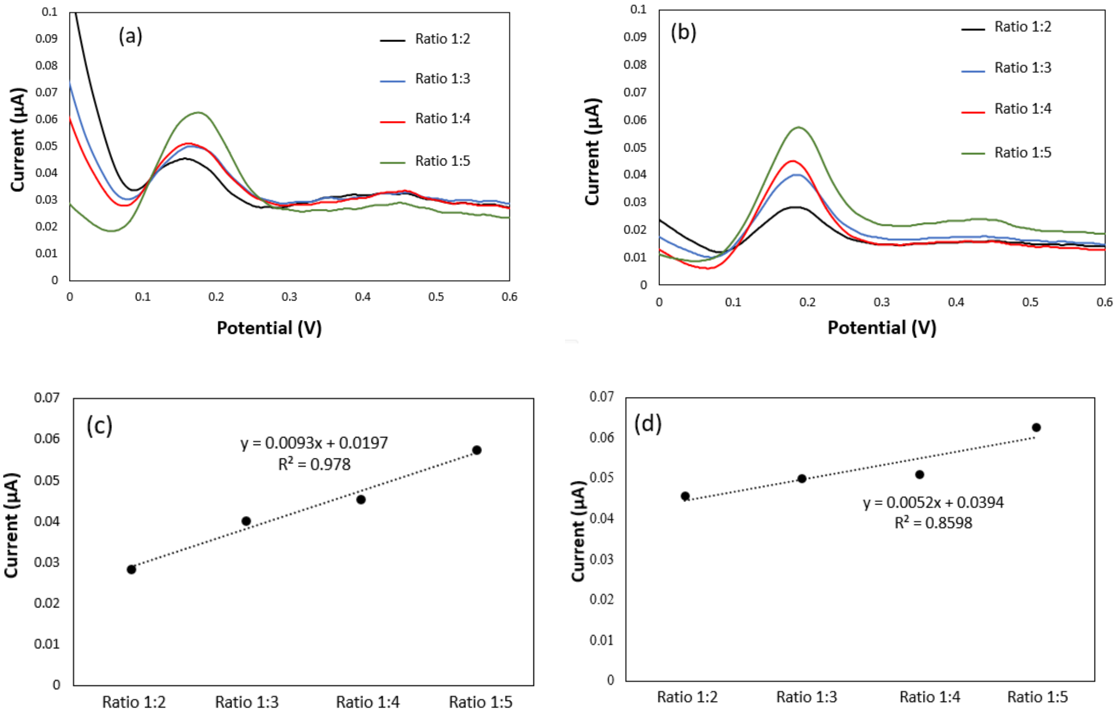

Fig. 7. (a) Stripping voltammogram to the comparison of As^{3+}:As^{5+}(b) Stripping voltammogram to the comparison of As^{5+}:As^{3+}, (c) Current response plot of current response to the comparison of As^{3+}:As^{5+} and (d) Current response plot of current response to the comparison of As^{5+}:As^{3+}

3.6 Analysis of UI lake water samples

The UI lake water samples used came from UI lake water in front of FMIPA UI. The sampling technique used is simple random sampling, namely a simple sampling technique carried out randomly. It is simple because sample selection is done without having to pay attention to the strata in the population. This technique can be considered representative only if it is carried out on members of a population that is assumed to be homogeneous (Elfil et al., 2019; Kalton, 2011). Figure 8 shows the stripping voltammogram resulting from measuring UI lake water samples with the addition of 10 ppm As^{3+} and 10 ppm As^{5+}.

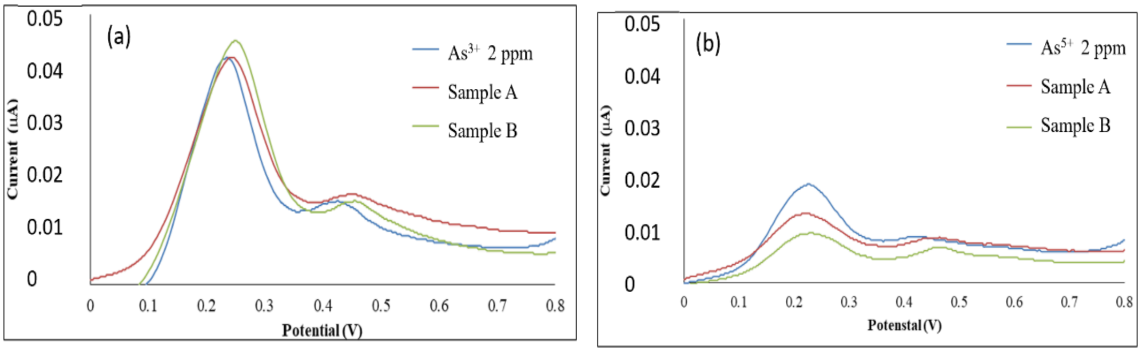

Fig. 8. Stripping voltammogram of measurement UI lake water with the addition of 10 ppm of a) As^{3+} and b) As^{5+}

Sample measurement is carried out by measuring the recovery using a simulation method, a pure amount of analyte is added to the placebo, then the mixture is analyzed and the results are compared with the added standard levels (Riyanto, 2014). The actual concentration is 2 ppm, while the concentration of the analyte added to the 4 mL sample is 10 ppm for 1 mL, so the total volume of the analyte spiked and the sample is 5 mL. From Figure 8, the recovery of As^{3+} in the UI A lake water sample is 99.7% and sample B is 107.6%. Meanwhile, the recovery of As^{5+} in the UI A lake water sample was 70.2% and sample B was 49.5%. Overall, the measurement results show that the simulation method used is effective to quantify the recovery by considering the presence of pure analyte and placebo. These findings provide important insights into the quantitative analysis of arsenic concentrations in UI Lake water samples.

4. Conclusions

Modification of BDD electrodes with Au using the seeding method followed by electrodeposition was successfully carried out for As^{3+} and As^{5+} application studies. Characterization of the Au film on the BDD surface using SEM, and XPS, shows the existence of Au on the surface of the BDD electrode. The influence of $NaBH_4$ for As^{3+} and As^{5+} sensors has increased the sensitivity and stability of the electrode. Pretreatment using $NaBH_4$ can facilitate the measurement of As^{5+} species with low deposition potential, because As^{5+} has been reduced to As^{3+} before measurement with ASV. The recovery of As^{3+} and As^{5+} in the UI A lake water sample were 99.7% and 70.2%, respectively. The recovery values of as^{3+} and As^{5+} in sample B were 107.6% and 49.5%, respectively.

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Author Contribution

Conceptualization, M.K., P.K.J.; Methodology, P.Y., D.T.L., Y.E.; Formal Analysis, P.Y., D.T.L.; Investigation, P.Y., D.T.L., M.K.; Writing – Original Draft Preparation, P.Y., P.K.J.; Writing – Review & Editing, P.Y., P.K.J.

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Conflicts of Interest

The authors declare no conflict of interest.

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