

Development, Analysis and Evaluation of Arsenic (III) Sensor

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Abstract—Pollution or contamination plays a major part in dreadful health hazards. Water, the most essential entity for life is being consumed without the knowledge of contaminants. Arsenic (As III) above 10µg/L is regarded to be poisonous to life and causes lung or skin cancer and cardiovascular geotaxis, mutagenic & carcinogenic effects. Contamination of As may not only occurs in under or drinking water but also in sea water which may be a health threat to aquatic lives. Arsenic decontamination need to be strictly adopted upon any form of water purification. A special technique for the determination by express sensing of arsenic ionic level in water was examined on a Smooth or Glassy carbon electrode (GCE) with nano Gold transformation in 0.5 M H₂SO₄. The nano Gold was setup using a cycling potential continuously ranging from 0.619 to -0.481 V (vs. H_{AuCl}4.3H₂O) Chloro Auric Acid in 0.5 M H₂SO₄ solution. The Gold nano particles are electro- deposited on GCE. The electrode was examined by Differential Pulse Voltammetry (DPV), Anodic Stripping Voltammetry (ASV), Linear Sweep Voltammetry (LSV) and cyclic Voltammetry (CV). After optimization, a LOD (Level Of Detection) of 0.9 parts per billion (ppb) was achieved with a 60s transformation at -0.94 V in 0.5 M H₂SO₄ whereas the WHO's (World Health Organization) maximum allowable arsenic level is 10 ppb, in drinking water. Mercury Mercurious Sulphate (MMS) was used as standard reference electrode. The developed sensor was evaluated for copper ion interference and was found to have no interfering effect due to ionic copper which is the most potential obstructing ionic species in ionic arsenic determination.

Keywords—Electrodeposition, Gold nanoparticles, Arsenic, CV, DPV, LSV, ASV, MMS, LOD and Glassy Carbon Electrode (GCE).

I. INTRODUCTION

Earth is largely covered by water. A subject of great interest worldwide is the maintenance of environmental quality and if possible, the rehabilitation of affected areas. Rehabilitation is a must not only for drug addicts, environment but also very important for water quality control. Arsenite (As 3+) and Arsenate (As 5+) are formed as compounds in combination with Oxygen, Chloride and sulfur ions. Toxicity of Arsenite is higher than that of Arsenate. Various techniques for the detection of Arsenic contamination level in drinking water were developed, Graphite Furnace Atomic Absorption Spectrometry, ICMPS (Inductively Coupled Plasma Mass Spectrometry) and High-Performance liquid Chromatography with ICPMS. These techniques, though reliable do not have the feasibility of monitoring Arsenic in field where large number of sampling to be done. Also the above said methods could be performed only on suitable laboratory conditions and are time consuming efforts. The most common methodology (Hach Method) includes the procedure of transferring arsenic into arsine gas and gas liberated to react with the detector paper that was permeated with mercuric bromide. The procedure also involves production of Hydrochloric acid which is dreadful to handle. Arsenic pollutant is difficult to identify and required lab based sophisticated devices like Spectrometer. The process is complex and also time consuming which involves photo-detector and Light source. The principle

being Photometry based on Beer-Lambert's Law where the absorbance of light is measured by passing monochromatic light through the sample solution. The emerging light then falls on a photo diode to determine the light Absorbed by the solution and to arrive at the concentration of arsenic. This method besides being portable and low cost, allows analysis of data through computer. Ion Selective Electrodes (ISE) technique is the replacement of ancient Flame Photometry methodology of Electrolytes analysis. ISE is meant with different forms, Ion Sensing, Ion Selective or Ion Specific Electrode. The Ion Sensing Electrode is the poor grade sensor which can sense different ions at a time and is not specific to a particular ion. The Ion Selective Electrode is a Selective or better grade sensor for a particular ion. It senses more than one ion the relative selectivity against other ions in comparison to a specified ion makes it applicable for the measurement. The Ion Specific Electrode is the best grade and quality sensor which is regarded to be more specific to a particular ion. In this method, for calibration using logarithmic scale, non-linearity reduces the accuracy. However the above said methods are not those much sensitive and accurate in measurement when compared to the minute concentration of arsenic contamination in water (less than 10 $\mu\text{g/L}$). Hence, a sensor which could overcome the above difficulties of measurement needs to be fabricated.

II. DESIGN SCHEME–METHODOLOGY



Fig. 1 Optimization for development of Sensor

Block Diagram

The proposed system is based on (Nanotechnology) Anodic Stripping Voltammetry (ASV). The design scheme is dealt with the fabrication of more sensitive Arsenic sensor using Electro-Chemical Stripping Voltammetry for determination. Being fast in determination and compact with portability Electro-Chemical methods are often chosen for this analysis. Volta metric Stripping methods enable a fast, low cost and reliable instrumentation capable of measurements at lower levels of concentration. Arsenic concentration in water using Nanotechnology – ASV is formed with differing terms of electro-deposition like time of deposition and concentration of nano particles. The deposition time is in proportion to the mean size of nano particles. Size of the particles is in direct proportion to the strength of the analyte under test. ASV is a special procedure which determines the strength of the analyte by electro-deposition of the analyte and then stripping it through sweeping anodic potential. Two types of electrodes will be employed. Conventional glassy carbon electrode and a screen-printed electrode will be employed in this paper work as sensing system for arsenic. Various films will be coated on these electrode surfaces and then used for arsenic sensing. Films, such as, mercury, bismuth, gold nano particle, etc. will be employed in this study. Standard As (III) solution will be used to calibrate the performance of the sensor. Varying concentrations of As (III) will be employed in the analysis using suitable background electrolyte. Pre concentration of As (III) will be carried out at suitable deposition potentials ranging between -0.3 and -0.7 Volt vs. Mercury Mercurious Sulphate (MMS). Then the electrode will be subjected to an anodic sweep from 0.619V to- 0.481 V Vs MMS. The peak of Arsenic stripping is expected at around 0.3 V Vs MMS. The peak current will be correlated with the ionic strength of Arsenic in the sample and calibration plot is drawn. From this calibration plot concentration of unknown quantity of As in the given solution can be estimated.

In Electro chemical procedures of Arsenic (III) determination either Anodic or Cathodic Stripping Voltammetry (ASV or CSV) is involved. CSV method of analysis has limitations in operation and toxic effects of mercury due to the electrode with hanging drop of mercury. Gold (Au) is proved to have higher sensitivity for oxidizing Arsenic than Platinum Mercury or Silver and best suits as the substrate for arsenic detection electrodes using ASV. Hua et al⁵ used gold ultra-micro electrode (25 μm) to achieve an LOD of 0.2 ppb in sea water. Kopnica and Novotny⁶ used Differential Pulse Anodic Stripping Voltammetry (DPASV) to get a level of detection as low as 0.2 ppb of As (III). Simmet⁷ investigated the Sono electro analytical determination of arsenic on Au electrode with an excellent detection limit of 0.75 ppb. Ellise and Hamilton⁸ realized an LOD as low as 0.38 ppb of Arsenic and Antimony using electrode with gold film in copper electrolytic solution. Bode wig e tal⁹ and Sun et al¹⁰ used a rotating gold electrode which could remove the bubbles of hydrogen while investigating arsenic with an LOD as low as 0.19 ppb. Feeney¹¹ and Kounaves¹² developed a fast technique to determine Arsenic using 564 gold ultra-micro electrode disk arrays (Au UMEA) and square wave's voltammetry (SWV). The present work consists of the development of GCE modified with gold nano-particles which could determine As (III) in water. The responses of the developed electrode sensor were compared using DPV, CV, LSV and SWV.

III. EXPERIMENTAL SECTION

Materials and Chemical Reagents:

Analytical grade Arsenite (NaAsO_2), Chloro Auric Acid ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$), Sulfuric Acid (H_2SO_4), Hydro Chloric Acid (HCl) and Mercury Mercurious Sulphate (MMS) were purchased from Sigma-Aldrich (New Delhi, India) which could be used without purifying. GCE and Platinum Electrode were bought from Ilion (Korea). All stock and subsequent dilution solutions were prepared using high purified and deionized water (Research Lab Central Electrochemical Research institute (CECRI)).

Instrumentation:

All the Electrochemical measurements were observed and recorded using an Auto Lab PGSTAT30 computer based potentiostat setup with GPES Version 4.96 Software and Standard 3 sensing element (EcoChemic). The MMS (Mercury Mercurious Sulphate) electrode with platinum wire was used as standard reference half-cell and Glassy carbon (GC) as working electrode. The GC electrode was polished using alumina in between each and every modification. After every polishing stage, the electrode was washed for 15 minutes using deionized water.

Experimental:

Sodium Arsenate (NaAsO_2) of analytical grade is used for preparing the stock solution. 70 $\mu\text{mole/L}$ of arsenic stock solution is prepared by dissolving 10mg of NaAsO_2 in 1 liter of deionized (extremely pure) water. Appropriate concentrations of Arsenic solutions are prepared by dilution principle using the formula,

$$V_i \times C_i = V_f \times C_f$$

Where V_f and V_i are the final and initial Volumes of solutions, C_f and C_i and are final and initial strengths of arsenic in the prepared solutions. Arsenic standard solutions of 0.9, 6, 9, 10, 12, 14, 17 and 20 $\mu\text{g/L}$ were prepared from the stock solution by dilution and used for the calibration of the developed sensor.

IV. PREPARATION OF ELECTRODE MODIFIED WITH GOLD NANO PARTICLE

The polished GC electrode was submerged into solutions of NaAsO_2 in 0.5 M H_2SO_4 under a potential step from 0.629 V (vs MMS) to a selected potentials -0.481 V was applied for a fixed time (5, 15, 30 s).

v. DEVELOPMENT OF ARSENIC SENSOR

GOLD nano particle deposition on GC electrode: Polished GC electrode with Alumina (Al_2O_3) was washed with deionised water. The Chemical setup consists of GC and platinum foil as working and counter electrode with MMS (Mercury Mercurious Sulphate) as standard reference standard reference electrodes. The current output was measured between working and the counter electrodes. Electrolyte is aqueous solution that consists of (4 ml) Chloro Auric Acid ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) + 0.5 mole of H_2SO_4 Chemical setup is connected with computer through Auto Lab Machine. Corresponding signal waveforms are generated using GPES (General Purpose Electrochemical System) software

Arsenic nano particle deposition on Glassy Carbon (GC) electrode:

Arsenic detector is developed using working GCE and Platinum foil counter electrode with reference to MMS standard electrode. GC electrode is polished and cleaned by Alumina (Al_2O_3). The current output in between the working electrode. Potential was applied between working and 2^3 standard reference electrodes. The current output was measured between working and the counter electrodes. Electrolyte is aqueous solution that consists of (4 ml) Chloro Auric Acid ($\text{HAuCl}_4 \cdot 3\text{H}_2\text{O}$) + 0.5 mole of H_2SO_4 Chemical setup is connected with computer through Auto Lab Machine. Corresponding signal waveforms are generated using GPES (General Purpose Electrochemical System) software



Fig.2 Auto Lab Machine & GPES System



Fig.3 Sensor strip making machine

and reference half-cells were measured by applying Potential between them. Electrolyte is aqueous solution and consists of (2 m Mole) Sodium Arsenite (NaAsO_2) + 25 ml of H_2O . Chemical setup is connected with computer through Auto Lab Machine. Corresponding signal waveforms are generated using GPES (General Purpose Electrochemical System) software.

VI. RESULTS AND DISCUSSION

EVALUATION AND CALIBRATION OF DEVELOPED ARSENIC SENSOR

For Arsenic sensor fabrication the following analysis had been done using GPES software & Auto lab machine. The calibration curve in Fig.4 shows As (III) at 30 sec without Gold (Au) deposition (-0.6V to 0.5 V) conditioning potential -1 using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and $20\mu\text{g/L}$. The corresponding NaAsO_2 (Arsenite) concentration as 5, 10, 15, 20, 25, 30 and $35\mu\text{g/L}$ respectively.

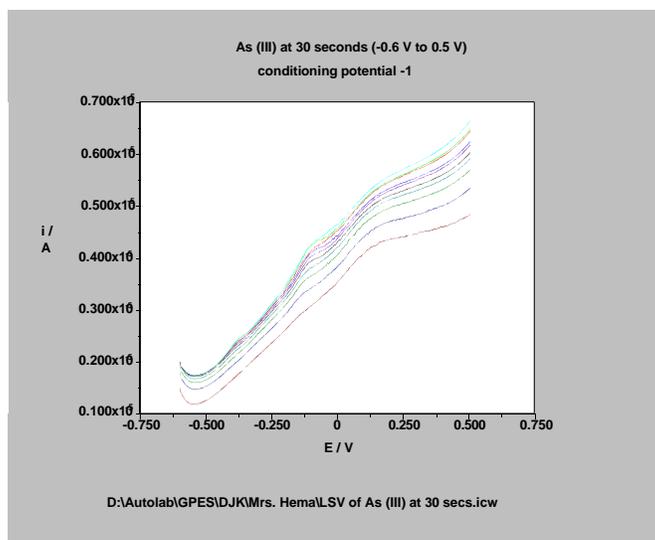


Fig. 4As (III) deposition at 30sec without Gold (Au) deposition (- 0.6V to 0.5 V) conditioning potential -1V.

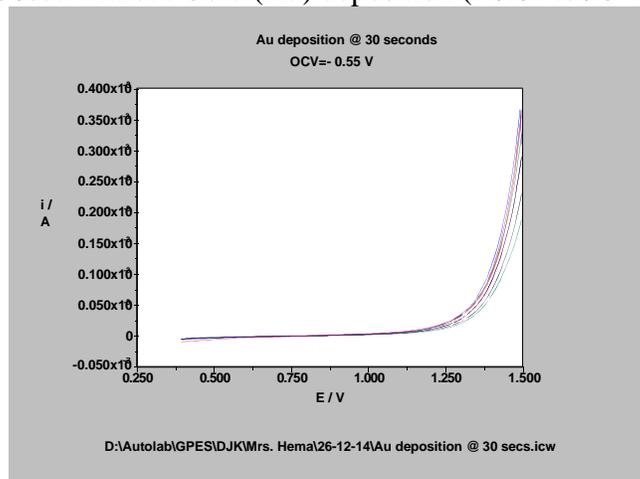


Fig. 5 LSV response of Au modified GC electrode. Au deposition @ 30 sec OCV = -0.55 V

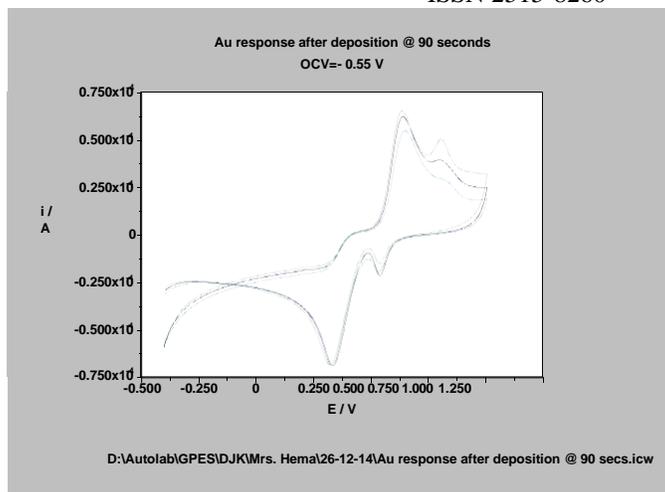


Fig.6 LSV response on Au modified GC electrode at different potentials. Au deposition @ 15 sec OCV = -0.55 V

The calibration curve in Fig.5 shows LSV response of Au modified GC electrode. Au deposition @ 30 sec OCV = -0.55 V using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 $\mu\text{g/L}$.

The calibration curve in Fig.6 shows LSV response on Au modified GC electrode at different potentials. Au deposition @ 15 sec OCV = -0.55 V using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 $\mu\text{g/L}$.

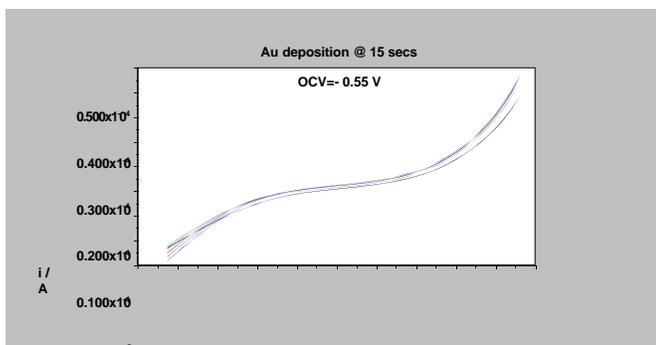


Fig.7 CV Response of the developed Au modified GC electrode towards Arsenic ions. (Au response after deposition @ 30 sec OCV = -0.55 V).

The calibration curve in Fig.7 shows CV (Cyclic Voltammetry) response of the developed Au modified GC electrode As (III). Au response after deposition @ 30 sec OCV = -0.55 V, using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 $\mu\text{g/L}$ of As (III) on Au @ 120 sec.

The calibration curve in Fig.8 shows CV (Cyclic Voltammetry) response of As (III) modified GC electrode. 1 μg NaAsO₂ response on Au @ 120 sec OCV = -0.48 V using standard solutions of As (Arsenic)

concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 $\mu\text{g/L}$.

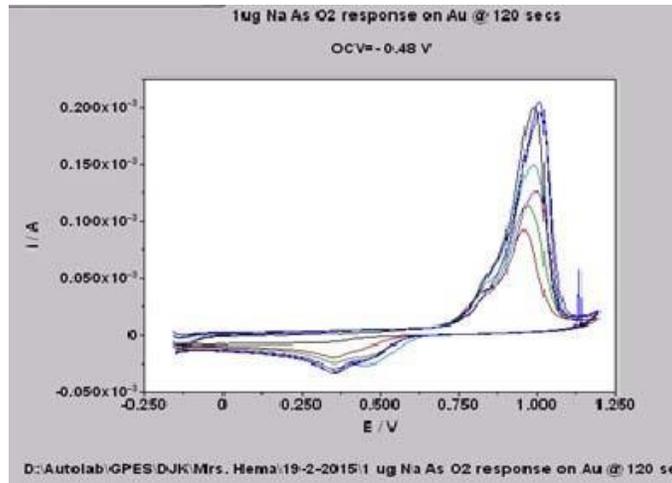


Fig.8 Cyclic Voltammetry (CV) = -10 μg NaAsO-0.48 V response

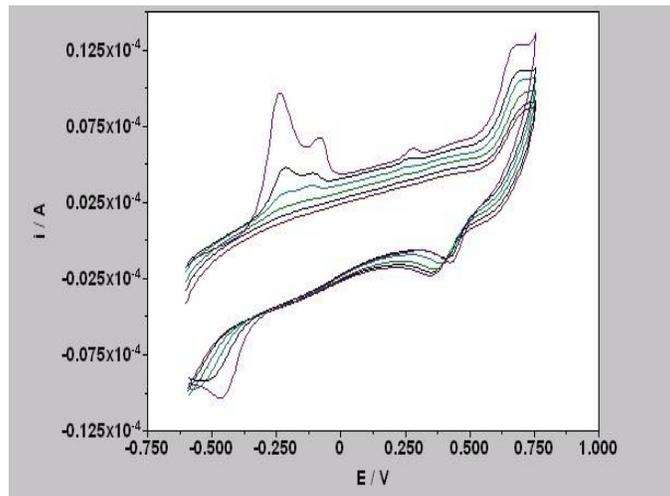


Fig.9 500 μl NaAsO₂ response w.r.t different deposition times (15, 30, 60, 120, 240, 480 secs) on Gold(Au) film deposited on GC @ 15 sec in 0.5 molH₂SO₄solution.

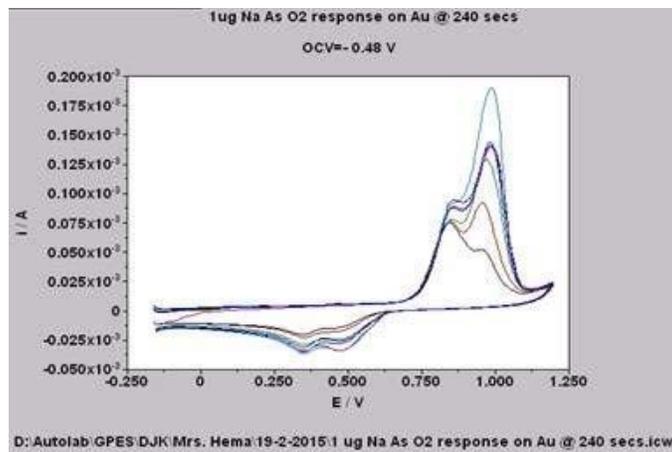


Fig.10 500 μ l NaAsO₂ response w.r.t diff deposition times (15, 30, 60, 120, 240, 480 sec) on Gold(Au) film deposited on GC @ 30 secs in 0.5 mol H₂SO₄ solution

The calibration curve in Fig.9 shows 500 μ l NaAsO₂ response w.r.t diff deposition times (15, 30, 60, 120, 240, 480 secs) on Gold(Au) film deposited on GC @ 15 sec in 0.5 mol H₂SO₄ solution.using different deposition times (15, 30, 60, 120, 240, 480 secs) of As(III) (Arsenic)deposition

The calibration curve in Fig.10 shows 500 μ l NaAsO₂ response w.r.t different deposition times (15, 30, 60, 120, 240, 480 secs) on Gold (Au) film deposited on GC @ 30 secs in 0.5 mol H₂SO₄ solution using standard solutions of As(Arsenic) concentration of 0.9, 6, 9,10,12, 14,17 and 20 μ g/L.

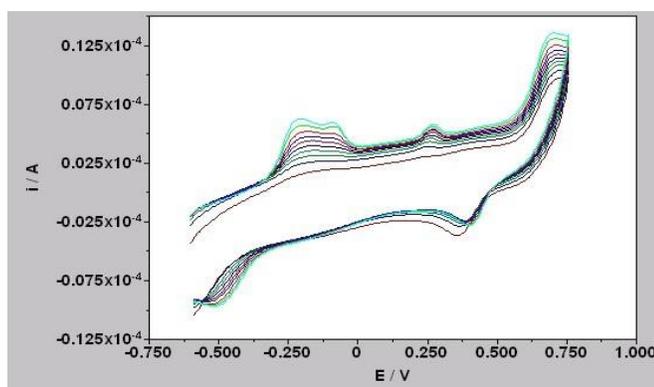


Fig.11 Cyclic Voltammetry-10 μ g NaAsO₂ response of As(III) on Au @ 240 sec OCV = -0.48 V

The calibration curve in Fig.11 shows CV (Cyclic Voltammetry) response of as (III) modified GC electrode. 1 μ g NaAsO₂ response on Au @ 240 sec OCV = -0.48 V using standard solutions of as (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 μ g/L

The calibration curve in Fig.12 shows the response of developed Arsenic sensor in DPV (Differential pulse voltammetry) of 10 μ g NaAsO₂ response on Au @ 15 sec using standard solutions of as (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 μ g/L.

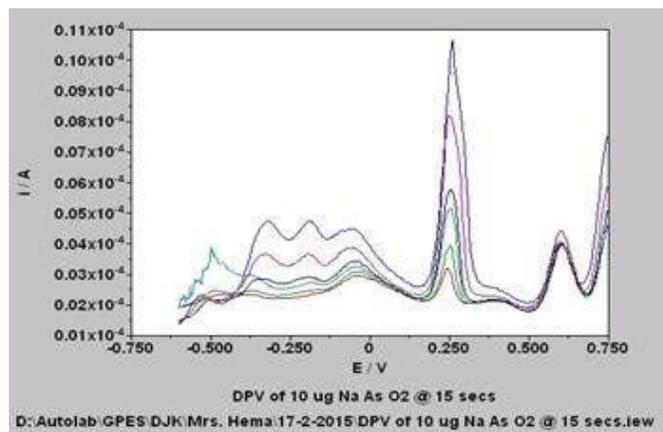


Fig.12DPV (Differential pulse voltammetry) of 10 μ g NaAsO₂ response of As (III) on Au @ 15 sec

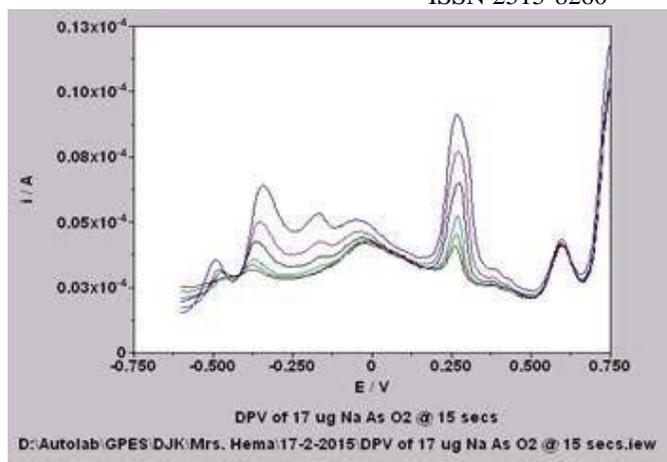


Fig.13 DPV (Differential pulse voltammetry) of 17 µgNaAsO₂ response of As (III) on Au @ 15 sec

The time slot of 15 sec was chosen for Au modification of GC electrode by deposition of nanoparticles before to recording the curves in Fig.12, Fig.13, Fig.14, Fig.15, Fig.16 and Fig.17. The NaAsO₂ concentrations used are 10, 17 and 24 µg/L respectively with Differential Pulse Voltammetry (DPV). DPV finds the impurities.

The calibration curve in Fig.13 shows the response of developed Arsenic sensor in DPV (Differential pulse voltammetry) of 17 µg NaAsO₂ response on Au @ 15 sec using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 µg/L.

The calibration curve in Fig.14 shows the response of developed Arsenic sensor in DPV (Differential pulse voltammetry) of 24 µg NaAsO₂ response on Au @ 15 sec using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20 µg/L.

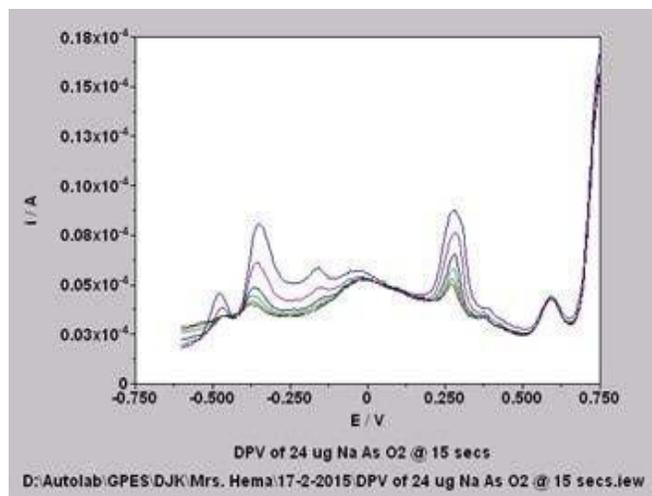


Fig.14 DPV (Differential pulse voltammetry) of 24 µg NaAsO₂ response of as (III) on Au @ 15 sec

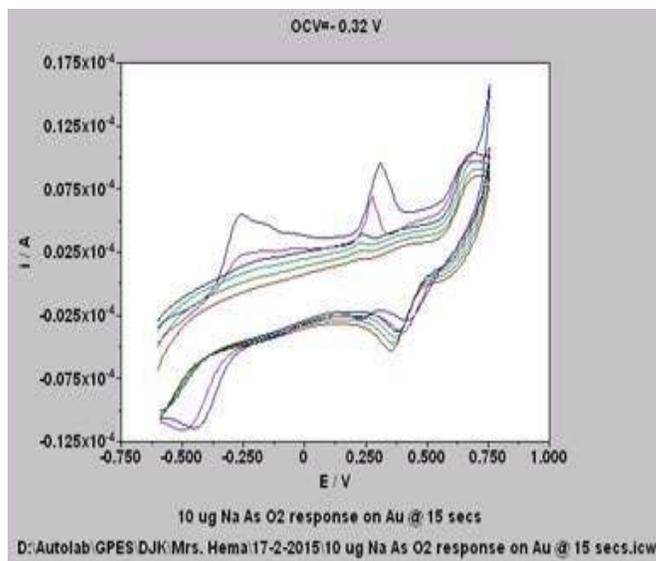


Fig.15 CV (Cyclic Voltammetry) 10µg NaAsO₂ response of As (III) on Au @ 15 sec OCV = -0.32 V with different deposition times.

The calibration curve in Fig.15 shows the response of developed Arsenic sensor in CV (Cyclic Voltammetry) 10µg NaAsO₂ response on Au @ 15 sec OCV = -0.32 V using standard solutions of As (Arsenic) concentration of 10 µg/L.

The calibration curve in Fig.16 shows the response of developed Arsenic sensor in CV (Cyclic Voltammetry) 17µg NaAsO₂ response on Au @ 15 sec OCV = -0.32 V using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20µg/L.

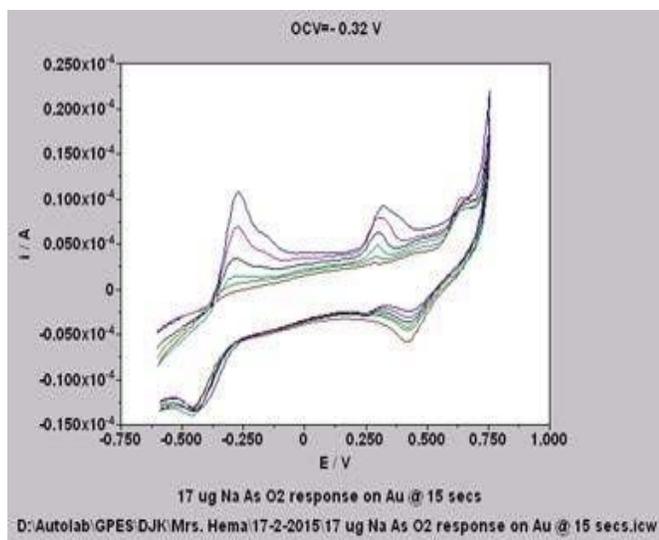


Fig.16 CV (Cyclic Voltammetry) 17µg NaAsO₂ response of As (III) on Au @ 15 sec OCV = -0.32 V

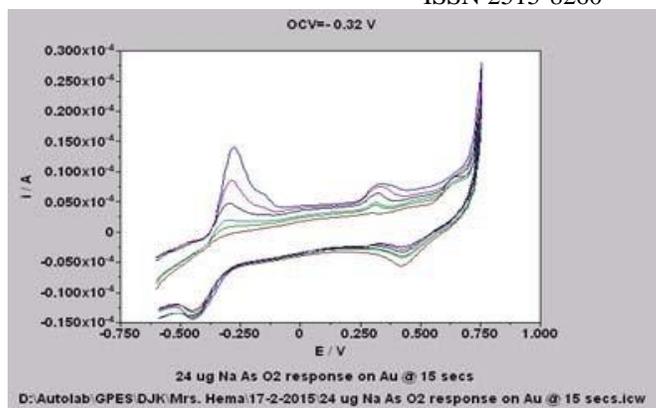


Fig.17 CV (Cyclic Voltammetry) 24µg NaAsO₂ response of As (III) on Au @ 15 sec OCV = -0.32 V

The calibration curve in Fig.17 shows the response of developed Arsenic sensor in CV (Cyclic Voltammetry) 24µg NaAsO₂ response on Au @ 15 sec deposition time. OCV =0.32 V using standard solutions of As (Arsenic) concentration of 14 µg/L.

The calibration curve in Fig.18 shows CV (Cyclic Voltammetry) response of As (III) modified GC electrode. 5µg NaAsO₂ response on Au @ 120 sec OCV = -0.48 V using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20µg/L.

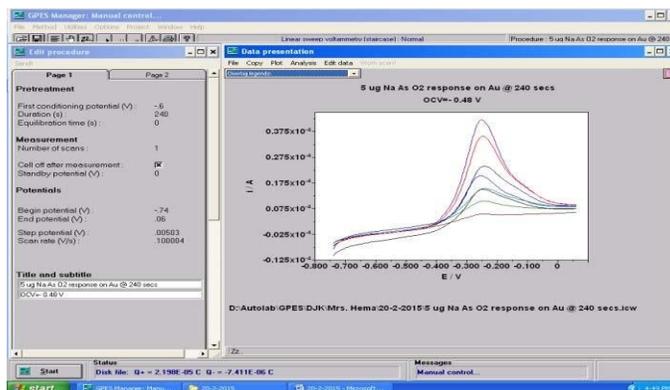


Fig.18 Linear Sweep Voltammetry (LSV) 5µgNaAsO₂ response on Au @ 120 sec OCV = -0.48 V

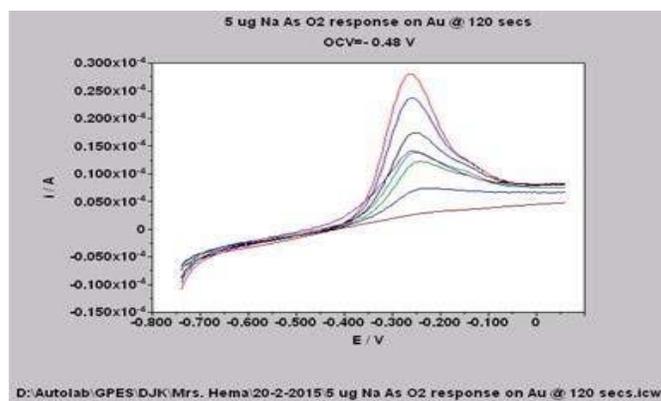


Fig.19LSV - 5 μ g NaAsO₂ response of the developed electrode on Au @ 240 sec OCV = -0.48 V.

The calibration curve in Fig.19 shows LSV response of As (III) on the developed GC electrode. 5µg NaAsO₂ response on (5sec deposited) Gold (Au) @ 240 sec deposition time of As (III), OCV= -0.48 V using standard solutions of As (Arsenic) concentration of 0.9, 6, 9, 10, 12, 14, 17 and 20µg/L.

VII. TABLE 1: CONCENTRATION OF ARSENIC (AS III) AND CORRESPONDING CURRENT (ARSENIC CONTAMINATION LEVEL)

Concentration of Sodium Arsenite (NaAsO ₂) µg/L	Concentration of Arsenic (AS III) µg/L	Current[µA] corresponding to Arsenic contamination @ deposition time of	
		240 Sec	120 Sec
5	0.9	6.5	2.5
10	5.8	11	7.5
15	8.7	15	12.5
17	9.8	16.5	13.5
20	11.5	20	14
25	14.4	24.5	17.5
30	17.3	35	23.5
35	20.2	42	27.5

Data are tabulated in Table 1 by using Fig.18 & 19 curves. Table 1 shows Concentration of Arsenic (AS III) and corresponding current (Arsenic contamination level) with two different deposition time of 120 and 240 sec

Table 2: Comparative result of Arsenic Ionic detections with Photometric and developed system of measurement.

Ionic Arsenic Level in µg/L		
S. No	Photometry	Developed System
1	1.72	1.63
2	2.95	3.11
3	10.96	9.98
4	14.01	14.14
5	5.84	6.01
6	12.11	12.13
7	3.32	3.41
8	0.75	0.76
9	10.02	10.03
10	1.68	1.68
11	0.09	0.07
12	2.29	2.25

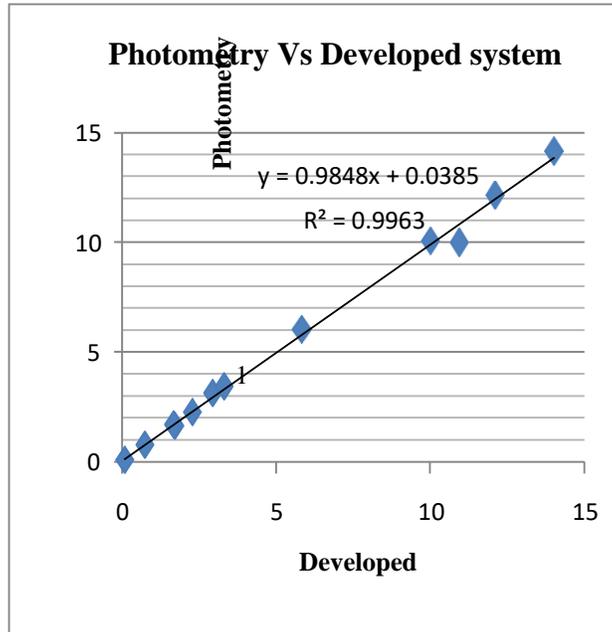


Fig.20: Concentration of As (III) Vs Current @ 240 and 120 sec deposition time

Fig.20 shows the concentration of As (III) Vs Current @ 240 and 120 sec deposition time curves. It was noted that the concentration increases the stripping current which was corresponding to Arsenic contamination level also increases. It is observed that the deposition time of 2 min (120 sec) is sufficient for the determination of As (III) in water.

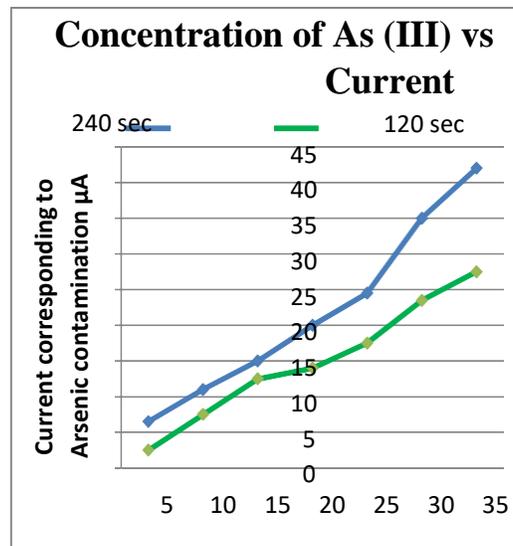


Fig.21 Shows the correlation curve for comparative results of Arsenic ionic concentrations using conventional Photometric method and developed system

The curve in Fig.2 shows a correlation of 0.996 which means a good correlation to conventional system.

Characterization of Au modification, on the electrode under development.

CV, profile for each developed electrode were recorded on 0.05 molar H₂SO₄ between the potential gradient of 0.629 V to -0.481 V with MMS as counter half-cell and at the rate of 100 mV S⁻¹ scanning speed. The responsive curves were as shown in Fig.4. It was observed that there were no peaks for a bare Gc electrode on either of the scan directions. The developed sensor is calibrated using standard solutions of 0.9, 6, 9, 10, 12, 14, 17 and 20µg/L

VIII. ELECTROCHEMICAL RESPONSE OF THE DEVELOPED AS ELECTRODE TOWARDS CV, DPV AND LSV.

An Instrumentation setup is developed with GPS tracking along with the Arsenic ionic sensor for determining the contamination levels of Arsenic in water samples, collected at various places. Arsenic concentration results obtained with the developed system are compared with the conventional system of measurement (Photometric method). Table 2 shows the comparative results obtained using conventional and developed system of arsenic level determination.

IX. CLINICAL SIGNIFICANCE

The clinically allowable Arsenic contamination level in drinking water is 10µg/L above which it is considered to be toxic to health. Arsenic poisoning could be observed with severe dehydration, vomiting and / or diarrhea, headache and drowsiness. Prolonged poisoning with arsenic in water may lead to irrecoverable neuro-physiologic and respiratory defects leading to skin and lung cancer.

X. CONCLUSION

Adopting Anodic Stripping Voltammetry (ASV), Arsenic level detector in water is developed. The developed sensor is evaluated for its detection limits and found to be better sensitive than conventional methodologies. An Instrument for monitoring Arsenic level contamination in water could be developed using ARM Processor and GPS tracking system implementing the developed arsenic sensor. The ionic arsenic levels were detected in water collected at various places and the results were compared with conventional photometric methods. The comparison showed a correlation of 0.996 which seems to be a best correlation and sensitive than the conventional method. It is also noted that the increasing of deposition time also increases the output peak current. Thus the deposition time holds the main role in this sensor. It is also noted that the As concentration increases the peak current at the output also increases accordingly. The amplitude is directly proportional to the contamination level of the sample water under test. LSV response is superior to CV. The study determines a fast, user friendly, low cost, portable and easy way. Since Arsenic poisoning in water is more hazardous to health, the developed system will be of immense important in providing better health to humanity. The system may also be implemented in rehabilitation efforts of purification of

Drinking water. This sensor strip is also tested for the detection of bulk metals like lead, bismuth, Copper, Gold, etc. pollutants in water.

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