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Electrochemical Deposition of CdTe Nanowires as Sensor for DPV determination of Uric Acid

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ABSTRACT: *An electrochemical deposition of Cadmium telluride nanowires using-Super-short potential pulses to created adatoms on platinum electrode, then both cyclic and stripping voltammetry was applied to grow the nanowier. The modified electrode was used to differential pulse (DPV) voltammetry determination of Uric acid UA in human blood serum. Proposed method was easy with good results in determination of UA in the concentration range 0.10-22.00 µmol/mL (r^2 = 0.9991), the recoveries were within the range of 97.22-100.40%, with RSD from 0.66% to 2.80%, indicating a good accuracy with high precision of the method.*

Keywords : *CdTe nanowires, differential pulse voltammetry, electrochemical deposition, Uric acid, human blood serum.*

1. INTRODUCTION

In the past few years, one-dimensional nanostructural materials, such as nanotubes and nanowires, have much attention due to their fundamental importance and the wide-ranging potential of their application in nanodevices [1,2,3,4,5]. Many experimental approaches to fabricate nanowires have been reported, utilizing a variety of nanofabrication techniques [6,7,8,9] and crystal growth methods, including arc discharge [12], laser ablation [15, 16], catalytic CVD growth [10], and template synthesis [11], etc. In this field, it is still a challenge to synthesize aligned and well-distributed nanowire arrays as well as highly crystallized structures. However, electrochemical deposition of nanowires is an expanding subject in materials science [12]. Chemical sensing using nanowires almost always means semiconductor nanowires. Lieber and co-workers were the first to demonstrate in 2001[13], that chemically modified single crystalline silicon nanowires could function as Chem FETs [14, 15] transistors in which the gating of the channel conductivity is effected by the specific binding of ions to receptors (e.g., antibodies) present at the surface of the nanowire [16, 17, 18]. But uses of electrochemical sensors depended on the nanowires are still rare in the analytical field, and under exploration and development.

Uric Acid (UA) is an important final product of purine metabolism in the human body and it is produced from the cellular breakdown prod acts of the purine nucleosides, adenosine and guanosine [19]. The normal physiological level of UA in human body ranges from 0.3 to 0.5 mmol/L in serum and 1.4 to 4.4 mmol/L in urinary excretion [20]. Abnormal levels of UA are the symptoms of several diseases such as gout, hyperuricaemia and Lesch-Nyhan syndrome [21, 22]. Therefore, it is essential to develop simple and rapid method for the determination UA in biological fluids. Various methods have been developed for the determination of UA in biological samples, including colorimetry [23], spectrophotometry [24], chromatography [25], chemiluminescence [26], fluorescence [27], and electrochemistry [28]. However, some of these methods are very expensive or complicated and time-consuming and are unsuitable for field-monitoring. Generally, electroanalytical techniques having advantages of easy fabrication, more selective, fast response, less time-consuming and portable nature are more suitable for the detection of biomolecules. Fast growth of the nanotechnology calls for low cost preparation of nanomaterials. Such a method is the electrochemistry which allows us to deposit materials in many different forms from the solutions [29].

In this work, we deposited cadmium telluride nanowire at platinum surface electrode using the super short potential pulses, then applied cyclic and stripping technologies respectively for longitudinal grow up of adatoms to form nanowires . We used these nanowires as sensors to differential pulse voltammeter determine Uric acid in human blood serum where we studied electrochemical behavior, and control the Experimental condition. The aim of this study is combining of nanomaterials with analytical chemistry and contributing to the development this field.

2. MATERIAL AND METHODS

2.1. APPARATUS

The electrochemical measurements were operated on 746 VA Trace Analyzer with 747 VA stand (Metrohm CH-9101 Herisau-Switzerland). The traditional three-electrode system, depended on the experimental we used various electrode: Modified carbon as working electrode, calomel reference electrode (3 mol/L HCl, Merck, Z113093, Germany), and Glassy carbon rod (Metrom, 6.1241.120) as the auxiliary electrodes. The pH values of the buffer solution were measured with an inoLab pH Level 1 (WTW-Germany), the size of nanowire was Measure by Nanosizer (Zetasizer Nano ZS model, Malvern Panalytical, England), micropipettes (BIOHIT). All the experiments were carried out at 25±1°C, otherwise mentioned at text.

2.2. REAGENTS AND CHEMICALS

Cadmium Sulfate $CdSO_4.H_2O$ (96 %, Sigma-Aldrich, for analysis), Dioxide Tellurium (99 %, Sigma-aldrich, Lab), Urea (98 %, PubChemi, for biochemistry), Anhydrous Sodium Sulfate (99 %, BDH, chemical pure) Tryptophan (98 %, Merck, GR), Ascorbic Acid (98 %, Merck, extra pure), Sulphuric Acid H_2SO_4 (95.0-98.0%, PRS Panreac) phosphoric acide H_3PO_4 (85-88%, Merck), Sodium Dihydrogen Orthophosphate $NaH_2PO_4.2H_2O$ (98.0%, HIMEDIA) Sodium Acetate CH_3COONa (99.5%, PRS Panreac), Sodium Hydroxide NaOH (LOBA chemie), Phosphate buffer (NaH_2PO_4/H_3PO_4) pH 7.0: 0.2 M solution dissolve 24 g of $NaH_2PO_4.H_2O$ in 800 mL of distilled water, add 85% H_3PO_4 until pH 7.0. Bring to volume with distilled water in a 1L volumetric flask. All solution prepared with distilled water.

2.3. CADMIUM TELLURIDE CdTe NANOSTRUCTURE ELECTRODEPOSITION BY COUPLED TWO TECHNIQUES PULSE AND CYCLIC/ STRIPPING

The electrochemical deposition of CdTe nanostructure was carried out in a 50 mL electrochemical glass cell with three electrodes, a saturated calomel reference electrode (SCE), a graphite counter electrode and a platinum worker electrode for deposition the nanostructure on its surface, we prepared three chemical precursors [30] as follows:

-Telluride ions chemical precursor:

0.1 mol/L Na_2SO_4 + 0.3 × 10^{-3} mol/L TeO_2, adjusting the pH at 2.0 ± 0.2 using a solution of 0.5 mol/L H_2SO_4 deposition temperature 65 ± 2 °C .

- Cadmium ions chemical precursor:

0.1 mol/L Na_2SO_4+ 0.5 × 10^{-3} mol/L $CdSO_4$, adjusting the pH at 2.0 ± 0.2 using a solution of 0.5 mol/L H_2SO_4 deposition temperature 65 ± 2 °C.

- Both tellurium and Cadmium ions chemical precursor:

0.1 mol/L Na_2SO_4 + 0.5 × 10^{-3} mol/L $CdSO_4$ + 0.3 × 10^{-3} mol/L TeO_2, adjusting the pH at 2.0 ± 0.2 using a solution of H_2SO_4 0.5 mol/L, deposition temperature 65 ± 2 °C.

Deposition steps

A. Step (I): The formation of the adatom (seeds) of tellurium on the surface of the platinum electrode:

We put 25 mL of the tellurium ions of chemical precursors in the electrochemical cell, with the three electrodes, apply a few following potential pulses (5-7 pulses) at fixed potential value 250 mV, so that the pulse time does not exceed 5 ms this process allows the formation of nucleation centers from tellurium on the surface of The platinum electrode as following: $TeO_2 + 4H^+ + 4e^- \rightarrow Te + 2H_2O$

But because of the short pulse time, these Nucleation centers grow up to form adatoms but it remains spaced and do not exceed the seed phase, and places from the surface of the platinum electrode remain bare without modification.

B. Step II: The formation of the adatoms (seeds) cadmium and cadmium telluride on the surface of the platinum electrode: We replace chemical precursor and put 25 mL of Cadmium ions chemical precursor with the three electrodes (the platinum electrode containing adatom tellurium on its surface as a working electrode). We repeat the application a few following potential pulses (5-7 pulses) at fixed potential value -655 mV, so that the pulse time does not exceed 5 ms. This process allows the formation of nucleation centers of cadmium on the space unmodified of the surface of the platinum electrode as in the following interaction: $Cd^{2+} + 2e^- \rightarrow Cd$

However, each two successive pulses must be separated by 3 s rest period, so that the reaction can occur between Cd^{2+} with Adatom tellurium, as shown by the following interaction: $Cd^{2+} + Te + 2e^- \rightarrow CdTe$.

C. Step 3: The growth of nanoparticles from CdTe: We replace chemical precursor and put 25 mL of both tellurium and cadmium ions chemical precursor with the three electrodes (the platinum electrode containing adatom cadmium and

CdTe nuclei on its surface as a working electrode). We applied the cyclic scan from + 230 mv to + 700 mv. While scanning the positive direction we notice the formation of a dark layer on the surface of the electrode as a result of the deposition CdTe rich in tellurium Te [31], but during the scanning in the negative direction the layer turns to reddish brown as a result of the removal of excess tellurium within the field (650-700) mV.

By repeating the cyclic/stripping scan within this range, the deposition process is repeated allowing the growth of nanoparticles and the nanoparticles are expected to grow vertically to take the form of nanowires. We measured the size of the nano CdTe by nanosizer set and get two dimensions 11.22 and 32.15 nm this supports our proposal to form nanowires.

3. RESULTS AND DISCUSSION

3.1. ELECTROCHEMICAL RESPONSE OF UA ON CdTe NANOSTRUCTURE MODIFIED ELECTRODE:

The compared of electrochemical responses among the cyclic scan of 2.0 µmol/L UA (pH 7.0 PBS) at and at the CdTe bulk and CdTe nanostructure modified electrode, Fig 1,a, and bare electrode, Fig 1.b, show the good advance and ability to determine of UA.

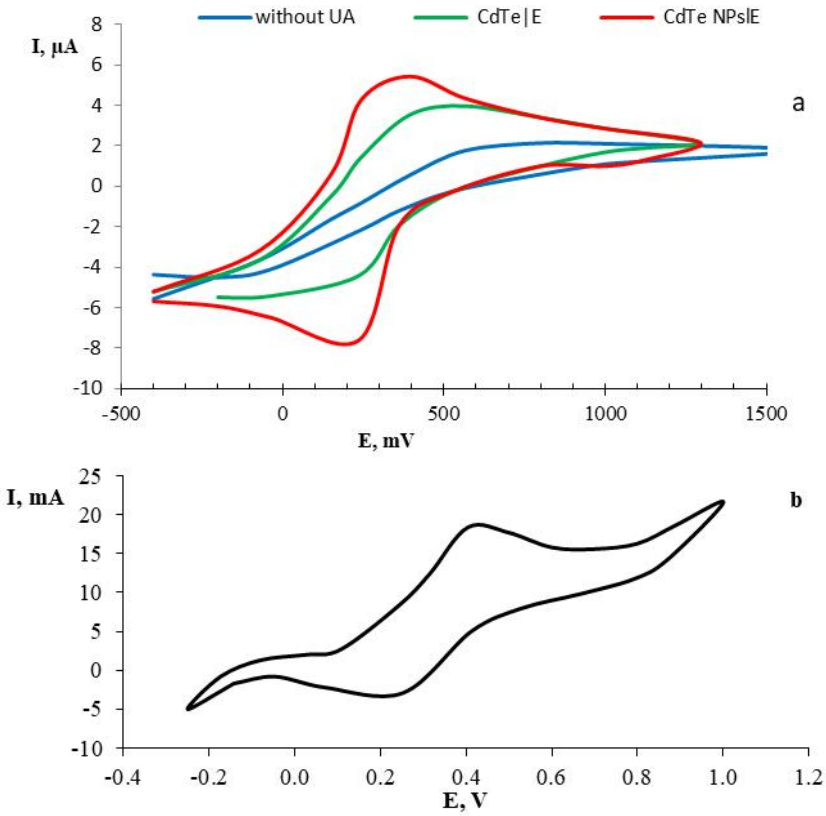

Fig 1: Typical CVg in the following cases

(a): (—) Cyclic scan of 2.0 µmol/L UA measured at CdTe nanowire modified electrode vs. reference electrode
(—): Cyclic scan of 2.0 µmol/L UA measured at bulk CdTe modified electrode vs. reference electrode
(—): Cyclic scan of supporting electrolyte (without UA) at measured at CdTe nanowire modified electrode vs. reference electrode
(b): Cyclic scan of 2.0 µmol/L UA measured at unmodified Pt electrode vs. reference electrode
where: Calomel 3 mol/L KCl as reference electrode, phosphate buffer solution (pH 7) as the supporting electrolyte. CV parameters were as follows: initial and end potentials were -400 mV, and +1200 mV, respectively; at fixed scan rate 100 mV/s, the experiment were carried out at room temperature 23-25 °C. The solution was unstirred

We studied the effect of the number of cycles performed in the cyclic scan during the process of electrochemical deposition of nanoparticles on the platinum electrode at the electrochemical response of the Uric acid using the CdTe nanowires, where we deposited CdTe NPs by applying a different number of cycles: 3, 5, 7, 1 0, 12 cycle, and then we

studied the electrochemical response of the urine acid in each case, applying the CV between the potential - 400 mV and 1200 mV, at PH 7.0 using a phosphate buffer solution with a 2.0 µmol/L of UA, and 100 mV/s rate scan. Results show, Fig. 2, when the number of cycles increased from 3 cycle to 7 cycle, the shape of the oxidation and reduction peaks becomes sharper and clearer, with increases the currents intensity of the peak, which lead to increase the sensitivity of the proposed analytical method. While the values of the current intensity decreases when the increase the number of Cycle in the cyclic scan due to the increase in the size of the electrochemical deposited nanoparticles, we have chosen seven cycles for the CdTe NPs deposition.

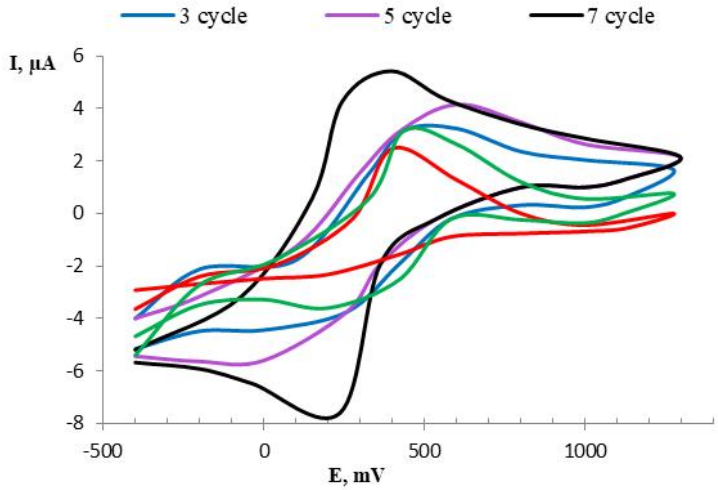

Fig. 2: Cyclic scan of 2.0 µmol/L UA measured at CdTe nanowire modified electrode vs. Calomel 3 mol/L KCl at different number of electrochemical deposition cyclic of CdTe nanowires>
Where: Phosphate buffer solution (pH 7) as the supporting electrolyte. CV parameters were as follows: initial and end potentials were -400 mV, and +1200 mV, respectively; at fixed scan rate 100 mV/s, the experiment were carried out at room temperature 23-25 °C. The solution was unstirred

3.2. OPTIMIZATION

We used DPV technology to determine the uric acid in human blood serum samples, we conducted the necessary analytical study as follows:

3.2.1. INFLUENCE OF pH

We studied effect of pH values on peak current, the study done within pH 4.00-10.00. The results show, Fig. 3, which the peak current enhanced with the increase of pH from 4.00 to 9.00, after that the peak current begins to decrease manifestly, so the value of pH 9 is chosen for performance of the experiments.

Although the best sensitivity is at pH 9.0, but at this value, any change resulting in increased pH will result in a sudden decrease in peak current intensity, so we chose pH 7.0 to perform all following experiments, because it is within the semi-constant range of the peak current values, as well as this pH value is portable for analysis at bio-samples medium (serum and intercellular fluid), as well as to prolong the life of the modified electrode

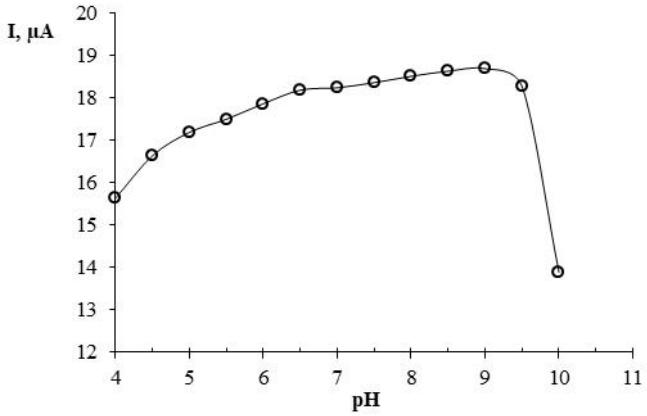

Fig. 3: Effect of pH on the electrochemical peak

3.2.2. INFLUENCE OF PULSE AMPLITUDE

We studied the effect of pulse amplitude on the current intensity of the UA oxidation peak, within the range 60 - 260 mV. Results are shown, Fig. 4, increasing the current intensity of the UA oxidation peak with the increase of the pulse amplitude in a semi-linear manner, but it begins to decrease after the value of 240 mV, so we chose 240 mV pulse amplitude as the optimal value in all experiments.

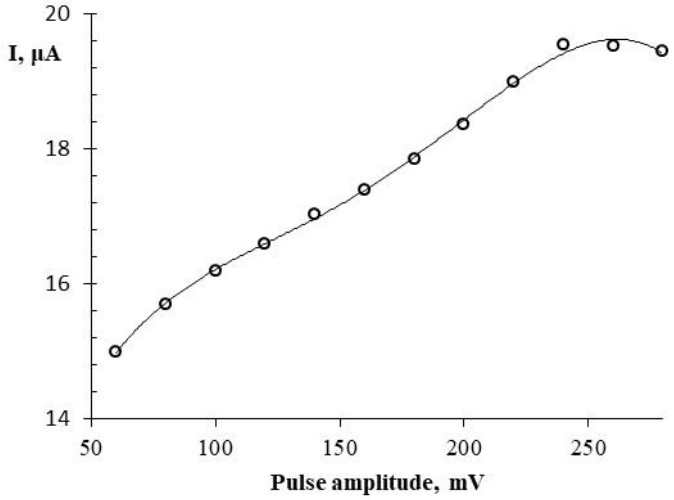

Fig. 4: Effect of pulse amplitude on the electrochemical peak

3.2.3. STABILITY OF CdTe NANOWIRE MODIFIED ELECTRODE

The already prepared CdTe nanowire modified electrodes were stored at room temperature, respectively. the measurements were made every 24 h in the same solution with electrodes stored at room temperature in three different solutions from distilled water, weak acidity solution from H_2SO_4 (pH 6), and weak basicity solution from NaOH (pH 8). Experimental data are listed in Table 1, indicates that the modified electrode that stored in distilled water were stable for ten days, After the modified electrode had been stored in distilled water at room temperature for ten days, the stripping signal decreased by 26 %, while that the modified electrode which stored in weak acidity solution were stable for sex days, After the this period, the stripping signal decreased by 16 %, while that the modified electrode which stored in weak basicity solution were stable for three days , where the stripping signal decreased by 37 % after this period**.**

Table 1: Stability of CdTe Nanowires modified electrodes at room temperature

torage time (day)	1	2	3	4	5	6	7	8			
	R %										
In distilled water	99.87	99.85	99.39	99.34	98.96	98.61	98.39	98.27	98.12	97.34	74.50
In acidity solution (H_2SO_4 pH 6)	98.89	97.46	97.23	96.72	96.12	95.33	85.04	-	-	-	-
In basicity solution (NaOH pH 8)	96.5	95.91	94.85	63.14	-	-	-	-	-	-	-

DPV experimental condition: pH 7.0 PBS, start potential 0.0 mV, end potential 700 mV, amplitude 240 mV, scan rate 100 mV/s.

3.3. METHOD VALIDATION

The calibration graph is described by the equation: Y=1.952 X + 1.227, (where Y = peak current and X= concentration in µmol/L), obtained by the method of least squares using Excel program. The peak current increased linearly with the concentration of standard UA in the range of 0.1-22 µmol/L (R^2 = 0.9991), as shown at the Fig. 5. The LOD = 0.015 µmol/L and LOQ = 0.048 µmol/L were calculated according to the same guidelines using the formulae: (LOD = 3.3 SD/b) and (LOQ= 10 SD/b), where SD is the standard deviation of five reagent blank determinations and b is the slope of the calibration curve [32]. The precision and accuracy were assessed according to the IUPAC recommendations by analyzing 0.1, 0.2, 0.4, 0.6, 0.8, 1.0, 2.0, 4.0, 6.0, 8.0, 10.0, 12.0, 14.0, 16.0, 18.0, 20.0, 22.0 µM of

UA in aqueous solutions, as shown at the Table 2. In addition of calculation of Analytical standard error S_m, and Confidence limit CL

The relative accuracy R % was within the range of 97.22-100.40 %, with RSD from 0.66 % to 2.80 %, indicating a good accuracy with high precision of the method.

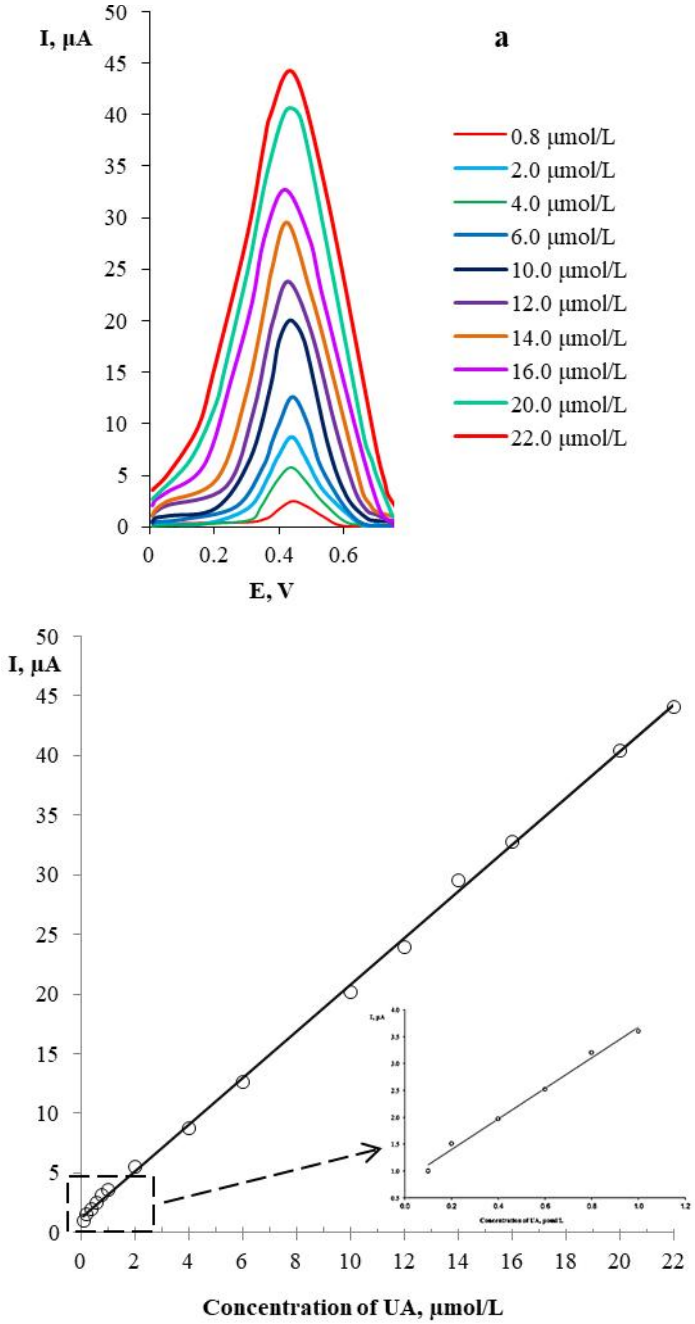

Fig. 5: (a) Calibration curves of the UA concentrations (b) DPV detection of the different concentrations of the UA, pH 7.0 PBS, start potential 0.0 mV, end potential 700 mV, amplitude 240 mV, scan rate 100 mV/s

Table 2 Evaluation of precision and accuracy for UA determination

Taken µM	Found ±SD µM	SD µM	R %	RSD %	CL µM	Sm
0.1	0.097	0.003	97.22	2.80	0.003±0.097	0.001143
0.2	0.195	0.005	97.48	2.31	0.005±0.195	0.001886
0.4	0.391	0.009	97.85	2.25	0.009±0.391	0.003674
0.6	0.591	0.013	98.43	2.24	0.014±0.591	0.005487

0.8	0.789	0.017	98.65	2.12	0.018±0.789	0.006924
1.0	0.995	0.020	99.50	2.04	0.021±0.995	0.008328
2.0	2.008	0.040	100.40	1.99	0.042±2.008	0.016248
4.0	3.990	0.071	99.75	1.77	0.074±3.990	0.028904
6.0	5.921	0.095	98.69	1.58	0.010±5.921	0.038702
10.0	9.847	0.132	98.47	1.32	0.138±9.847	0.053889
12.0	11.780	0.152	98.17	1.27	0.160±11.780	0.062217
14.0	13.726	0.151	98.04	1.08	0.159±13.726	0.061727
16.0	15.600	0.144	97.50	0.90	0.151±15.600	0.058788
20.0	19.458	0.146	97.29	0.73	0.153±19.458	0.059604
22.0	21.396	0.145	97.25	0.66	0.152±21.396	0.059277

n=6, t=2.571

3.4. INTERFERENCE STUDY

The major interference in the determination of UA is L-Dopa, Tryptophan, Urea and Ascorbic Acid as they coexist together in living systems. The concentrations of the interfering substances were examined within the concentration range from 1.0-fold until 100- fold. A relative error of ±5 % on the concentration of Uric acid was considered tolerable. The determination of 10 µmol/L Uric acid is not affected at all by the presence of L-Dopa, because the oxidant potential of UA is +413 mV and the potential response of L-Dopa is +250 mV, the difference between the two potential values is sufficient for determine each separately. Other impediments was study have interfere at concentration much bigger than concentration of Uric acid, which Urea interferes at 24-fold, Ascorbic acid at 5-fold, Tryptophan at 14-fold, so we can see that this proposed method is selective.

3.5. ANALYTICAL APPLICATION

We applied the proposed method of determining Uric acid in the serum human blood from five different samples, obtained from the Martini Hospital in Aleppo. The study was conducted without any treatment for the serum sample. We take 10.0 mL of the serum by a micro pipette within a 25 ml standard volumetric flask and complete the volume with 0.1 mol /L phosphate solution, and then apply the suggested method .

The results show, Fig. 6, the appearance of an oxidation peak at E = +413 mV, similar to the peak obtained during the quantitative and statistical analysis of Uric acid, Fig. 5, In order to verify the validity of the results obtained by applying the electrochemical method proposed in this study, compared with the results of the enzymatic reference method with the presence of urease and spectroscopy [33], and obtained a good consensus between the results of the two methods.

Table 3: Determination of serum Uric acid by application of the proposed method and enzymatic reference method

Samples No.	Suggested method mg/dL	Enzymatic reference method mg/dL	Result
Sample (1)	5.86	5.90	Normal
Sample (2)	7.04	6.90	*Normal
Sample (3)	3.87	3.80	Normal
Sample (4)	6.33	6.40	Normal
Sample (5)	8.54	8.49	Morbidity

* This sample is normal for men blood sample but it morbidity sample if it is woman blood sample.

CONCLUSION

The new method depended on electrochemical deposition CdTe nanowire modified electrode for determination of Uric acid by different pulse voltammetry, this proposed method is quick and have good accuracy with high precision and selective, so it suitable for routine analysis of Uric acid

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