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Electrodeposition of 3D hierarchical porous flower–like Cobalt-MWCNT nanocomposites electrode for non-enzymatic glucose sensing

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In this work, pure Cobalt and Cobalt-MWCNT nanocomposites were electrodeposited on mild steel substrate using acetate bath. The surface morphology, elemental composition and the preferred orientation of pure Cobalt and Cobalt-MWCNT nanocomposites were characterized using SEM, EDAX and XRD analysis. The SEM images of Co-MWCNT composite exhibited as the nano flakes which are interconnected to form a 3D hierarchical porous flower like morphology. MWCNTs are well incorporated into the matrix. XRD also confirmed the composite formation and the presence of CNT. Cyclic voltammetry and chronoamperometric techniques were employed to characterize the electro catalytic activity of the modified electrode towards the glucose detection. The results revealed that the proposed sensing material synergistically acts as an electro catalyst for the electro-oxidation of glucose with enhanced sensitivity and lower detection limit. The fast current response signals, wide linear range and higher selectivity make Cobalt-MWCNT nanocomposite electrode a favorable candidate for practical glucose detection application.

Introduction

Diabetes mellitus, the disease known for insulin deficiency is always being a public health problem. The tremendous increase in the diabetic population still attracts much attention on glucose sensing and the periodic monitoring of glucose is essential for the diagnosis of diabetes[1-3]. Glucose detection is more significant in clinical diagnosis, food industry, and bio-technology. A large number of enzymatic electrodes have been employed as glucose sensor since the discovery of first glucose oxidase electrode in 1960s[4-8]. Enzymatic sensors are commercialized owing to its sensitivity and selectivity. Though these electrodes have higher electro catalytic activity, enzymatic sensor suffers from elaborated enzyme immobilization process, instability, pH and high cost of enzyme[9-11]. Hence, there is a need to develop sensors which are fast and accurate and paved the way for the non-enzymatic glucose sensors. Electrochemical non-enzymatic sensors have attracted intense interest due to their high sensitivity, good reproducibility and low cost. Various approaches such as hydrothermal[12-13], sol-gel[14], electrospinning[5,15], and electrophoretic deposition[16] have been applied to prepare the sensing material. However, these methodologies need sophisticated equipments and involve toxic chemicals. In addition to that glucose oxidation on conventional drop casted electrodes hinders the practical use of the material because of its sluggish reaction kinetics. The higher potential needed for glucose determination consequently decreases the sensitivity[6]. On the other hand, electrodeposition is a facile technique to synthesize nano materials due to its cost effectiveness, environment friendliness and ease of fabrication.[17-18] Nowadays transition metals[19], metal oxides[8,20], alloys[21] and spinel[5] have been used for glucose sensing. Of these transition metals gained considerable attention for constructing practical glucose monitoring system due to its availability and low cost. Particularly Cobalt and its oxides are well known for their electro catalytic activity towards the glucose oxidation owing to its higher catalytic activities, stability and multi valencies in alkaline medium[18,22-23]. Recently Carbon nanotubes (CNTs) are extensively utilized in electrochemical sensors because of its unique properties like high surface to volume ratio, good electrical conductivity and stability. Moreover, CNT present in the nano composite can furnish conductivity for faster electron transport along with electro active sites for spatial diffusion[24-27]. The composite material synthesized by adding highly conductive carbon nanotubes to the metal and metal oxides can expect to enhance the performance owing to their

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synergistic effect between the two components rather than one[28-29]. Furthermore, the nanoparticles incorporated composite always have an enhanced physical and mechanical properties. Fenghua su et al have prepared the Co-MWCNTs composite coatings by different electrodeposition techniques and concluded that the composites have higher hardness and corrosion resistance[30]. Susumu Arai et al have been synthesized Co-MWCNT by composite plating method and investigated its field emission properties[31]. Cobalt based composites are widely investigated for its tribiological properties. Mahdavi et al reported the characteristics of electrodeposited Co-TiO_2 composite coatings[32]. Although the electro catalytic behaviour of Co ions in alkaline solutions have been well studied, the effect of incorporating nano particles into Co matrix by electrodeposition and thus formed Co composite electrodes as non-enzymatic glucose sensor has not been studied. To the best of author's knowledge, there are no reports on electrodeposited Cobalt-MWCNT nanocomposites modified electrode for glucose detection.

In this work, an electrodeposition technique is employed to synthesize the Co-MWCNT composite electrode for detecting glucose selectively and in presence of interferences such as urea, dopamine, ascorbic acid and NaCl as a non-enzymatic sensor.

2. Experimental

2.1 Materials

All reagents were of analytical grade and were used 'as received' without further purification. MWCNT was purchased from Tokyo chemical industry, Japan. Cobalt (II) acetate and boric acid was purchased from Merck, India. Standard solutions of glucose were freshly prepared prior to each experiment using distilled water. Ascorbic acid and dopamine were obtained from Alfa Aesar. Urea and KCl were supplied from Otto inc. Mumbai, India.

2.2 Preparation of Cobalt-MWCNT nanocomposites electrode

Mild steel plates of 7 cm x 1 cm x 0.5 mm were mechanically polished with grade 4, 5, 6 emery papers and then degreased with trichloroethylene. The unexposed area was masked with stop-off lacquer to acquire required area for deposits. Further, it was subjected to alkaline electro-cleaning followed by acid pickling in 5% HCl solution. After that, the substrate was washed in the running water and dipped into the electrolyte containing 100gL^{-1} cobalt (II) acetate, 40 gL^{-1} boric acid and required amount of MWCNTs with the particle size of 10-20 nm (diam). Before each experiment, the electrolyte was stirred continuously for 8 hours to blend the MWCNTs with the plating solution to get uniform suspension. Fig 1 shows and confirms the dispersion level of MWCNTs in the composite electrolyte. As is seen, the particles are uniformly suspended in the composite electrolyte. Initially the electrocodeposition was performed by varying the concentration of MWCNT from 1gL^{-1} to 4gL^{-1}, Current density ranging from 2A/dm2 to 4A/dm2, temperature from 30^0C-50^0C and pH from 2 to 4. By judging the quality and adherent nature of the deposit, the deposition parameters were optimized under which further co-deposition was performed using 2gL^{-1} MWCNT at the optimized conditions 4A/dm^2,30^0C and pH 4 for 15 minutes using a two electrode system in which graphite plate acts as anode and pre-treated mild steel as cathode. The deposited film, about 10 μm thicknesses was washed with distilled water and dried in air. 0.5 cm^2 geometrical areas of the electrodeposits were used for glucose determination.

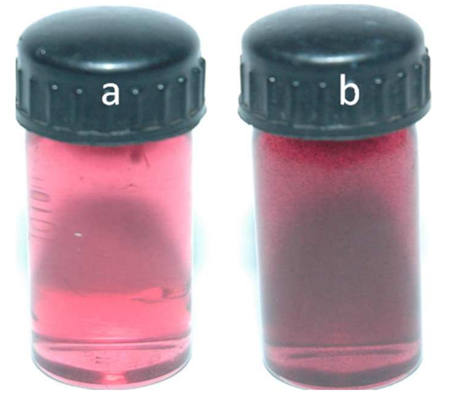

Fig 1. Photographs of a) pure cobalt and b) MWCNTs dispersed in cobalt solution

2.3 Electrochemical characterization

The surface morphology and the elemental composition were analyzed using scanning electron microscopy and energy dispersive X-ray spectrometer (Hitachi, model 3000H). The preferred orientation was evaluated by an XPERT PRO X-ray diffractometry (XRD) using Cu kα radiation having the wavelength of 1.5406 Å. The Scanning range is between 20^0 and 90^0 with a step size of 0.04°.

The electrochemical experiments for glucose sensing was carried out in a conventional three electrode set up containing deposited cobalt and cobalt-MWCNT nanocomposites as working electrodes, platinum as counter electrode and Ag/AgCl as reference electrode using SP-150 potentiostat, Biologic Make (France). Cyclic voltammetry and chronoamperometry were employed to study the electro oxidation of glucose in 0.1 M NaOH aqueous solution.

3. Results and discussion

3.1 Characterization of Cobalt-MWCNT nano composites electrode

The SEM images of pure cobalt and cobalt-MWCNTs are shown in Fig 2. In the case of pure cobalt the surface morphology (Fig.2a) seems to be of cobalt nano flakes that are uniformly distributed all over the substrate. As seen in Fig 2b, Cobalt-MWCNTs nanocomposites were also appeared as nano flakes which are interconnected to form 3D hierarchical flower like porous structure. This unique morphology existence is because of the MWCNTs added in to the bath which acts as surface modifier and made the aggregated nano flakes to form a flower-like morphology which is in agreement with the cited literature[33].

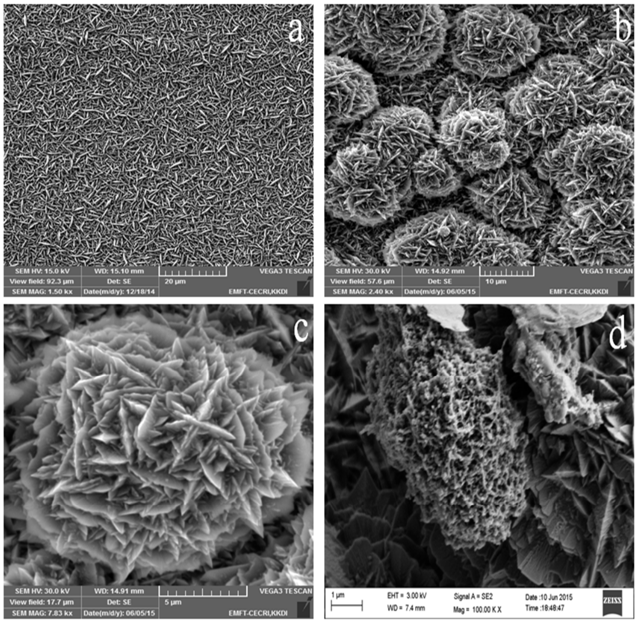

Fig. 2 SEM images of a) pure cobalt b) Co-MWCNT nanocomposite c) single flower like morphology of Co-MWCNT d) FESEM image of Co-MWCNT

Fig 2c shows the single hierarchical architecture revealed the growth mechanism which is originated from the core. The MWCNTs are well embedded on the layers of cobalt which is clearly visible at 100K magnification in FESEM images shown in Fig 2c. The MWCNTs are entangled and well embedded on the layers of cobalt which is clearly visible at 100 K magnification in FESEM images shown in Fig 2d. The possible mechanism for the flower like structure is elucidated as the strong physical adsorbing tendency of multi walled carbon nanotubes[34] that are easily adsorbed on the electrode surface providing active sites for the nucleation and growth of cobalt nuclei. Fig 3 is shown the back scattered SEM images of pure cobalt and Cobalt-MWCNTs which clearly revealed the distribution of MWCNT in the composite coatings.

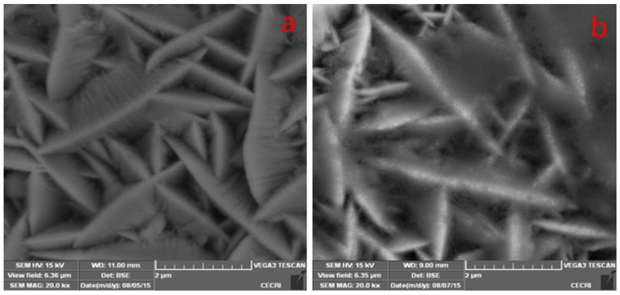

Fig 3. Back Scattered SEM images of a) cobalt and b) Co-MWCNT nanocomposites coating.

The chemical composition of pure cobalt and cobalt-MWCNTs nanocomposites electrodeposits are shown in Figs 4a and 4b. Pure cobalt thin films is comprised of cobalt, and oxide while the Cobalt-MWCNTs thin film contained cobalt, carbon and oxide with the wt% of 67, 30, 3 respectively. The EDAX spectrum analysis of the composite deposit confirmed 30 wt% distribution of MWCNTs in the composite coatings.

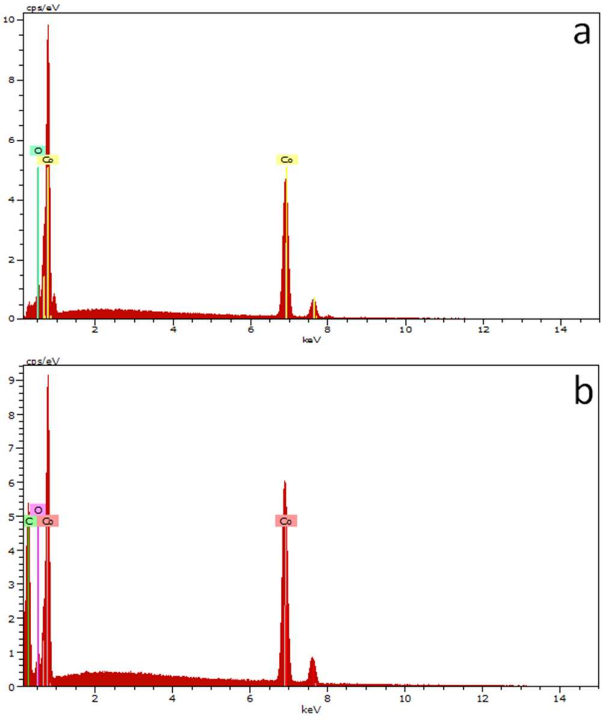

Fig 4. EDAX spectrum of a) pure cobalt b) Co-MWCNT nano composite coating.

3.2 XRD analysis

For Co-MWCNT nanocomposites the preferred orientation changed from (100) plane to (110) plane and its intensity was increased compared to pure cobalt suggesting the reduction of grain size and the existence of preferable crystal structure.[35] Moreover the existence of new peak at (101) indicated the 'hcp' texture. The incorporation of MWCNT not only changed the orientation but also decreased the grain size[30]. The peak located at 2θ value of 31.6751 and 44.6725 could be assigned to the crystal structure of Co_3O_4 which is corresponding to the (220) and (400) planes of the standard JCPDS 42-1467.[1,33] The peak remained at 2θ= 28.4278 could be indexed to the CNT.

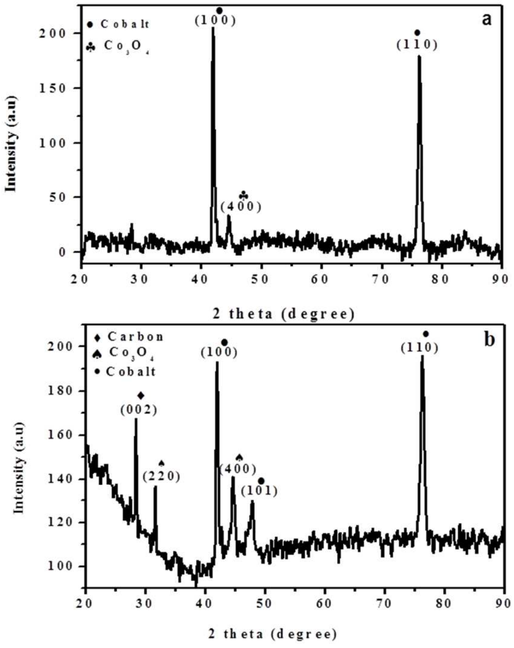

Fig 5. XRD pattern of a) pure cobalt b) Cobalt- MWCNT nanocomposite coating.

3.3 Co-MWCNT electrodeposits as a glucose sensor

Fig 5a shows the cyclic voltammograms for pure cobalt and Co-MWCNT nanocomposite electrode in the absence of glucose in 0.1 M NaOH solution at a scan rate of $50mVs^{-1}$. In both cases, two well defined redox pair was obtained. Peak I at 0.2 V could be attributed to the conversion of Co^{2+} to Co^{3+} and Peak II at 0.5 V is assigned to the oxidation of Co^{3+} to Co^{4+} which is in good agreement with the cited literature.[36-39] Oxidation current density for Co-MWCNT (0.45 $mAcm^{-2}$) is higher than that of pure cobalt (0.16 $mAcm^{-2}$) indicating the synergistic effect of cobalt and MWCNTs which is highly useful for electrochemical sensing applications. Fig 5b shows the CVs for the oxidation of 1mM glucose added into the 0.1 M NaOH solution at a scan rate of $20mVs^{-1}$. In the case of cobalt the onset potential of glucose oxidation starts at E= 0.4 V whereas for Co-MWCNT modified electrode the onset potential is E= 0.27 V ascribed to the enhanced performance due to MWCNT. On addition of 1 mM glucose into 0.1 M NaOH, oxidation peak (II) current increases from 0.36 mA cm^{-2} to 0.66 $mAcm^{-2}$ and the potential shift towards negative direction revealed that glucose is oxidized to gluconolactone by CoO_2/CoOOH redox couple[20,39]. As expected, addition of MWCNT into the cobalt matrix increases the oxidation current and lowering the over potential for glucose compared with pure cobalt. Fig 5c represents the cyclic voltammograms of Co-MWCNT nanocomposite electrode for different concentrations of glucose. It is seen that the oxidation current increases with the increase in the glucose concentration.

$$\mathrm{Co_3O_4 + OH^- + H_2O \longleftrightarrow 3CoOOH + e\text{-}}$$
$$\mathrm{CoOOH + OH \longleftrightarrow CoO_2 + H_2O + e\text{-}}$$
$$\mathrm{2CoO_2 + C_6H_{12}O_6 \longrightarrow 2CoOOH + C_6H_{10}O_6}$$

The results revealed that the potential shift and anodic current increment is due to the synergistic effect of Cobalt and MWCNT.

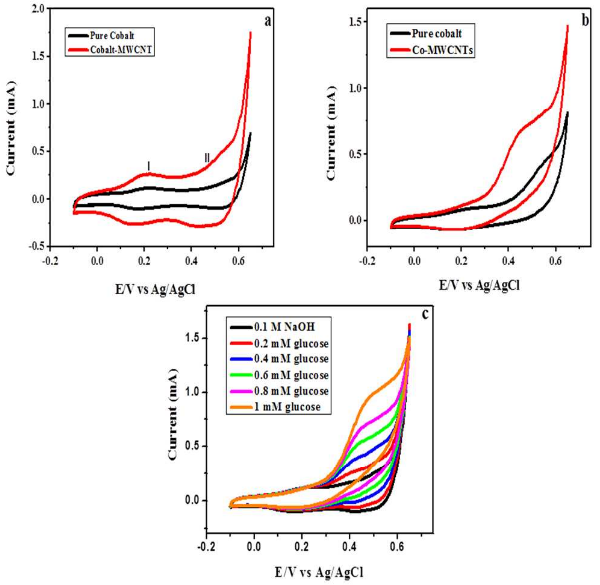

Fig 5. a) Cyclic voltammograms of pure cobalt and Co-MWCNT nano composite in 0.1 M NaOH at a scan rate of 50 mVs^{-1} b) in the presence of 1 mM of glucose at a scan rate of 20 mVs^{-1} c) with the presence of increasing concentration of glucose at a scan rate of 20 mVs^{-1}

3.4 Amperometric detection of glucose

All the amperometric experiments were carried out at 0.43 V vs Ag/AgCl which is optimized from the CV experiments. Fig 6a shows the amperometric response of Co-MWCNT nanocomposite electrode in the presence of glucose in the stirred solution of 0.1 M NaOH. For each addition of glucose, current increased steeply followed by the attainment of steady state current which represents the constant increment in oxidation current upon different concentration of glucose. The calibration curve for glucose detection was obtained by plotting the current values with glucose concentration. At the applied potential of 0.43 V, the sensor displays two linear detection ranges, one in the low concentration and another in high concentration range. Two linear ranges had also been reported in the literature in glucose sensing reasoning that increasing the glucose concentration blocked the mass transfer that affected the diffusion of fresh glucose molecules nearer to the electrode surface. At concentration higher than 0.1 mM the electrochemical response is flattened[40]. Consequently two discrete linear ranges were obtained for Co-MWCNTs modified electrode. As shown in fig 6b the calibration curve is linear for glucose concentration ranged from 5µM to 100 µM and another linear behavior for high concentration of glucose ranged from 200 µM to 3600 µM (fig 6c). At higher concentration of glucose the amperometric curve reached its saturation point hence some amperometric current response is declined due to the adsorbed intermediates could block the active catalytic sites[41]. Furthermore, limit of detection for lower concentration of glucose was calculated as 0.009µM whereas for higher concentration range, the detection limit was estimated to be 0.3 µM at a signal to noise ratio of 3 with the correlation coefficient of 0.992 and 0.989. The sensitivity was calculated from the slope of the calibration curve and it was found as 727 µA $mM^{-1}cm^{-2}$ and 37.05 µA $mM^{-1}cm^{-2}$. The steady state response time for each addition of glucose is reached within 3s revealed the rapid electron transport on the electrode surface. In particular, while increasing the glucose concentration, the anodic current increases but the non-linearity above 3600 µM may be due to the limit generated from the oxidation rate or the saturation of number of catalytic sites for oxidation[5]. The analytical performance of proposed sensor was compared with other glucose sensors reported in the literature and are presented in table 1. As is seen, the analytical parameters with this glucose sensor are better than the other sensors reported in the literature. Particularly limit of detection for lower concentration is 9 nM which is superior to the other modified electrodes reported in the literature.[18,25,28] The enhanced analytic performance is because of the three dimensional hierarchical porous structure of the Co-MWCNT nanocomposites electrode.

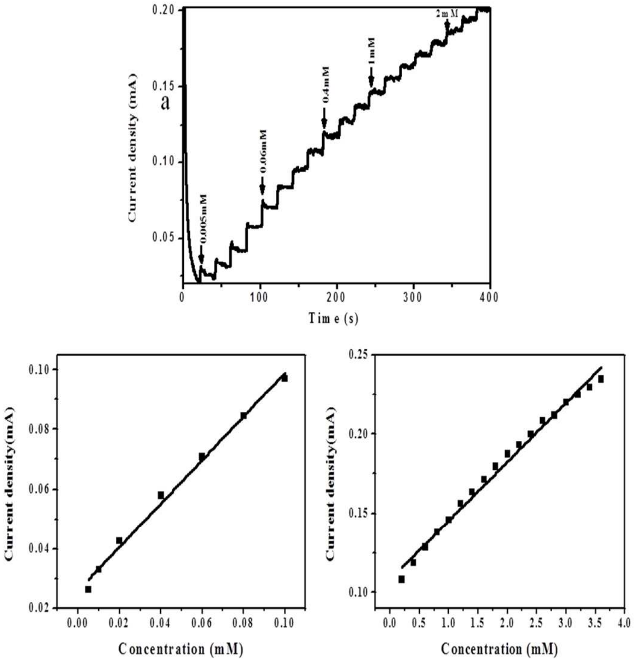

Fig 6. a) Chrono amperometric current response of Co-MWCNT nanocomposite for successive addition of glucose into the stirred 0.1 M NaOH. b) The calibration plot for lower concentration range of glucose. c) The calibration plot for higher concentration range of glucose.

3.5 Interference test

In order to establish the selectivity of Co-MWCNTnanocomposite electrode for glucose detection, commonly interfered species like ascorbic acid, dopamine, and urea were added along with glucose. The physiological levels of these interferents are very less than glucose. In addition to that chloride ion poisoning is an issue associated with non enzymatic glucose sensor.[29,34] Hence 0.1 M of NaCl is added in order to investigate the effect of chloride ions. 0.1mM concentration of glucose was added initially and the oxidation current increases whereas for the addition of 0.1 M concentration of inteferrents has no obvious current response is seen in fig 7.

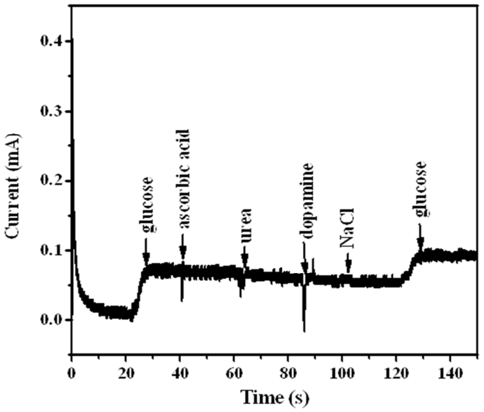

Fig 7. Chrono amperometric current response of Co-MWCNT nanocomposites for the successive injection of 0.1 mM glucose followed by 0.1mM interferents at a fixed potential of 0.43V vs Ag/AgCl.

The current response of the inteferrents are very low compared to glucose, is an evident for the selective detection of glucose molecules. Hence, this modified composite electrode can be used for sensing of glucose even in the presence of other interferents.

3.6 Reproducibility and stability

In an attempt to evaluate the reproducibility of the Co-MWCNT nanocomposite electrode, five different electrodes were prepared under similar conditions and the amperometric response was recorded on injection of 45µM glucose. The relative standard deviation (RSD) of 3.03% was obtained indicating the good reproducibility. The sensor was stored in the desiccator in order to establish the long term usage. The long term stability of the proposed sensor was evaluated for every five days for the addition of 60 µM glucose. 97.3% of current was retained after 45 days which revealed its excellent stability.

3.7 Real sample analysis

To determine the practicability of the sensor, Co-MWCNT nanocomposite electrode was used to analyze the glucose in blood serum. One serum sample was collected from our institute clinic and the amperometric experiment was performed. Fig 8 represents the amperometric response of 0.1 mM and 0.2 mM standard glucose solution followed by human blood serum sample. 100µl of blood serum was added into 10 ml of 0.1 M NaOH and the amperometric response was recorded. The amount of glucose present in the serum was estimated as 6mM by comparing the known current value of standard glucose concentration. The clinically reported value is 5.8 mM which is close to the estimated values using this sensor material indicating the practical use of this sensor.

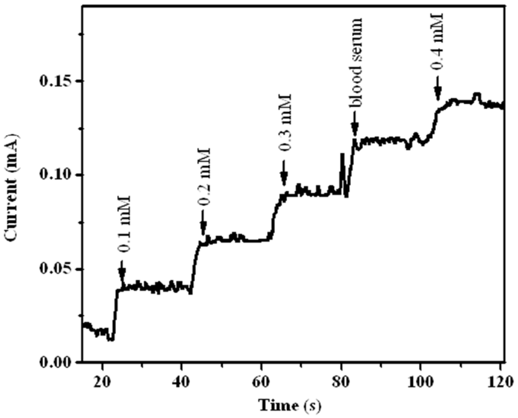

Fig 8. Amperometric current response of Co-MWCNT nano composite in the presence of various concentration of glucose followed by blood serum in 0.1 M NaOH.

4 Conclusions

In this study, versatile and cost-effective electrodeposition technique is adopted to synthesize Cobalt and Cobalt-MWCNT nanocomposite electrodes and the fabricated material is successfully demonstrated as non-enzymatic glucose sensor. Hierarchical porous structure and the incorporation of CNT into the Co matrix synergistically enhanced the electron transfer resulting to lower detection limit of 0.009µM with high sensitivity of 727 µAmM^{-1}cm^{-2} and good selectivity towards glucose detection. The proposed sensor is highly stable for over a month. Hence, it can provide greater pathway for real time application for monitoring glucose.

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Table 1.

Comparision of analytical parameters with other sensors

reported in the literature.

Electrode materials	Applied potential (V)	Sensitivity (μA mM^{-1} cm^{-2})	Linear range (mM)	Detection limit (μM)	References
Co-MWCNT nanocomposite	0.43	727,37	0.005-0.1, 0.2-3.6	0.009, 0.3	This work
CoOOH nano sheet	0.5	967	Up to 0.5	10.9	38
Co_3O_4-RGO	0.5	1145.2	0.1-0.9	10	1
Co_3O_4 nanofibers	0.5	36.3	Up to 2	0.97	39
CoOOH nano sheet arrays	0.5	526.8	Up to 1.109	1.37	22
3D Graphene-Cobalt oxide	0.5	3390	Up to 0.08	0.025	36
MWCNT-CoTsPc	0.3	0.1225	0.01-6.34	0.14	25
CoOx.nH_2O-MWCNT	0.55	162.8	Up to 4.5	2	18
CoOxNPs/ERGO	0.6	79.3	0.01-0.55	2	29