



# Water based homogenous carbon ink modified electrode as an efficient sensor system for simultaneous detection of ascorbic acid, dopamine and uric acid



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## ABSTRACT

Development of new sensor material suitable for simple, cost effective and quick practical application is a demanding research interest in electroanalytical chemistry. Carbon based inks prepared using different type carbon materials and binders as a gel or suspension have been often used as active sensor materials. Unfortunately, those systems are involved with complicated preparation route and usage of hazardous chemicals. Herein, we report a water based carbon ink composed of low cost carbon black powder, chitosan and acetic acid (prepared within  $15 \pm 2$  min) for electrochemical sensor applications. A new carbon ink modified electrode was fabricated in this work by drop-casting of micro-litre quantity of the CB-Chit ink on a cleaned glassy carbon electrode (GCE) followed by air drying in room temperature for  $10 \pm 2$  min. The CB-Chit ink modified GCE (GCE/CB-Chit) showed enhanced electrical conductivity, surface area and electrochemical activity than that of the unmodified GCE. Investigated simultaneous electrochemical oxidation and sensing of three biologically important molecules such as ascorbic acid, dopamine and uric acid on GCE/CB-Chit displayed excellent peak current signals at well-defined peak potentials with linear concentration ranges of 25–1600, 0.1–1400, and 5–1800  $\mu\text{M}$  and detection limit of 0.1  $\mu\text{M}$  ( $S/N=3$ ) for the all analytes. The newly fabricated sensor was validated by applying to the detection of AA, DA, and UA in vitamin C tablets, dopamine hydrochloride injection and human urine real samples. The sensor can be operated without any surface pre-treatment and analyte adsorption, unlike to the carbon nanotube and graphite based electrode systems with serious adsorption complications.

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## 1. Introduction

Recently, the emerging need for quick and low cost electrochemical sensor has turned the electrochemist to discover a new class of nanomaterial suitable for unconventional electrode fabrication and applications. In this regard, carbon based material which could show property and activity similar to the expensive graphitic materials such as graphene, graphene oxide and carbon nanotube, but having low cost, is highly desirable [1–3]. Carbon black (CB) is one such material widely used in tires, printing inks, rubber and plastic products. It has been prepared by incomplete combustion or thermal decomposition of gaseous or liquid hydrocarbons under controlled conditions and has shown

attractive physical properties similar to the graphitic material like high surface area ( $>1000 \text{ m}^2/\text{g}$ ) and low density (much lower than the theoretical value for graphite,  $2.25 \text{ g/cm}^3$ ) [4,5]. In electrochemistry, CB has been used as a binder in the carbon paste electrodes (CPEs) and screen-printed electrode preparations [6]. The CB chemically modified electrodes prepared by combining nafion<sup>®</sup>, chitosan, conducting polymers were showed appreciable electrochemical sensor activities to nicotinamide adenine dinucleotide (NADH), benzoquinone, epinephrine, cysteine, thiocholine and hydrogen peroxide [7,4,8,9]. Indeed, owing to practical difficulty in transferring quantitative amount of the heterogeneous carbon suspension from the stock suspension solution to the electrode surface, fabrication of reproducible carbon modified electrode surface is found to be highly challenging. Few of CB-Chit nanocomposite (heterogeneous) based reports were reported in the literature for dye removal [10] waste water treatment [11], fuel cell [12,13], sensors [14] and biosensors [15]

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applications. Herein, we report a homogenous carbon black-chitosan ink (CB-Chit) prepared by simple chemical mixing method for development of cost-effective and reproducible carbon modified electrodes. Simultaneous electrochemical sensing of ascorbic acid (AA), dopamine (DA) and uric acid (UA) in a neutral pH solution was demonstrated as a model system for the applicability of the CB modified electrode.

In general, carbon based inks are prepared by mixing carbon powder/particles, polymeric stabilizers such as polystyrene, isophorone, polytetrafluoroethylene, ethylene glycol, surfactant (Triton X100), polyionic liquids, poly(3,4-ethylenedioxythiophene) and binder (essential for adhesion, printing and dispersion) as a paste or suspension, which are then suitably casted on underlying substrate like screen-printed substrate [16], glassy carbon [17], carbon paste [18] and ceramic substrate [19] for fabrication of electrochemical sensors. For example, Osborne *et al.* prepared a carbon ink based on blending glassy carbon powder, polystyrene and isophorone at a ratio of carbon/polymer = 2.3 and modified on a polytetrafluoroethylene substrate [20]. Pech *et al.* prepared a carbon ink by mixing activated carbon of high surface area ( $1700\text{--}1800\text{ m}^2\text{ g}^{-1}$ ) with 5 wt.% polytetrafluoroethylene (PTFE) polymer binder, ethylene glycol solvent and Triton X100 surfactant, wherein, the activated carbon composition was set as 3 wt.% and the resulted gel like ink was used for electrochemical capacitor application [21]. Meanwhile, although certain carbon nanoparticles like quantum dots and carbon nano-fibre can dissolve freely in organic solvents like DMSO and THF [22,23], in general, bulk carbon materials like carbon black and graphite are not soluble in any solvents. It is noteworthy that the carbon ink modified electrodes often require activation procedures such as pre-anodization, laser treatment and acid treatment prior to the electroanalytical applications [18–20]. For example, a ArF UV excimer laser operating at 193 nm (LPX 200, Lambda Physik, Germany) delivering laser fluences from 100 to 400 mJ per pulse ( $0.05$  to  $0.2\text{ J cm}^{-2}$ ) at a frequency of 10 Hz was applied on the glassy-carbon ink modified electrode as a pre-treatment procedure [20]. Similarly, to enhance the electrochemical activity of the screen-printed carbon electrodes, pre-anodization at 0.9 to 1.5 V vs SCE in strong acid or alkaline solution as an activation step has been adopted [16,18,19]. Note that, some of polymers used in the ink formulations like polystyrene and isophorene are toxic and referred as potential carcinogens [20]. Thus, development of an elegant and non-hazardous carbon based ink suitable for simple modification on any substrate and can operate without any pretreatment procedure/s is a demanding research interest in the interdisciplinary field of electrochemistry. Herein, we report a water based carbon ink prepared by carbon black-chitosan polymer-acetic acid as an environmental friendly system for pretreatment-free carbon chemically modified electrode development.

Ascorbic acid (AA) (Vitamin C) is a potential biological antioxidant that has been widely used for the treatment of common cold, scurvy, mental illness, infertility, cancer and AIDS [24]. Dopamine (DA) is an important catecholamine neurotransmitter which plays a critical role in the function of central nervous, hormonal and cardiovascular systems [25]. Low level of DA in blood may result in Huntington's disease and neurological disorders such as Parkinson's disease and schizophrenia [26]. Uric acid (UA) is the primary end product of purine metabolism. For a healthy human being, concentration of UA in urine and blood are  $\sim 2\text{ mM}$  and  $120\text{--}450\text{ }\mu\text{M}$  respectively [27]. Abnormalities of the UA level may lead to several diseases such as Lesch-Nyhan syndrome, hyperuricemia, gout and pneumonia etc [28]. Moreover, it has been found that AA, DA and UA coexist in the extra cellular fluid of the central nervous system and serum in mammals. Selective, quick and separation-less determination of these

biochemicals is a demanding research interest in medicinal analytical chemistry. Several analytical methods have been developed for the simultaneous determination of two or three components of AA, DA, and UA [29–33]. Chemiluminescence [29], high-performance liquid chromatography [30], electrochemical method [31], spectrophotometric [32] and capillary electrophoresis [33] have been often employed for this purpose. Most of these procedures are complicated, time consuming and usually required specialized instruments. Among these methods, electrochemical method has been drawn great attention due to their low-cost, easy operation and high sensitivity [34]. Unfortunately, these three molecules are oxidized at almost the closer potentials on the conventional solid electrodes and hence resulted into overlapping of the voltammetric peak current signals. To overcome this problem, various chemically modified electrodes including noble metal nanoparticles [35,36], metal oxides [37,38], carbon nanomaterials [39,40] polymers [41,42] and composites based on the above materials [43–45], have been developed for the simultaneous determination of AA, DA and UA. Unfortunately, they are suffering from some serious drawbacks such as requirement of tedious preparation [31,44,45], pre-treatments [35], perm-selective membrane like Nafion [39] and use of expensive metal nanoparticles such as Au, Ag, Pd and Pt in their modified electrode formulations [35,36,46,47]. For instance, a Pt/RGO nanocomposite modified GCE was prepared by *in situ* chemical reduction of GO and  $\text{H}_2\text{PtCl}_6$  using  $\text{NaBH}_4$  and was used for the detection of DA and UA in presence AA [46]. Similarly, a hydrothermal based preparation of Ag nanoparticle-decorated RGO composite was used as an active material for the simultaneous determination of AA, DA, UA [47]. In this work, a new carbon black ink modified electrode prepared within  $15 \pm 2$  min showed an elegant detection of these biochemicals with appreciable peak-to-peak separation, good linearity and low detection limit values. Applicability of the working sensor was demonstrated by detecting AA, DA and UA in different kinds of real samples such as vitamin C tablets, dopamine injection and human urine samples with good recovery values.

## 2. Experimental

### 2.1. Reagents and solutions

Carbon black (N330 grade) was obtained as a gift sample from Phillips Carbon Black Ltd. Kochi, India. Chitosan was obtained from Sigma-Aldrich. Ascorbic acid, dopamine and uric acid were purchased from, Merck, India. All reagents were of analytical grade and used as received without any further purification. 0.1 M phosphate buffer solutions (PBS) of pH varying from 1 to 11 were prepared by mixing standard stock solutions of  $\text{Na}_2\text{HPO}_4$  and  $\text{NaH}_2\text{PO}_4$  and adjusting the pH by the addition of either 0.1 M  $\text{H}_3\text{PO}_4$  or 0.1 M NaOH. Prior to each electrochemical experiment, all the solutions were deoxygenated with extra pure  $\text{N}_2$  gas for about  $15 \pm 1$  minutes. Aqueous solutions were prepared using deionized and alkaline potassium permanganate distilled water (designated as DD water).

### 2.2. Apparatus

The electrochemical measurements were carried out using an electrochemical workstation (CH Instruments, Model 760C) with a conventional three electrode cell using glassy carbon electrode (GCE) (CH instruments) as a working electrode (area  $0.0707\text{ cm}^2$ ), Ag/AgCl as an reference electrode and Pt wire as a counter electrode. Raman spectroscopic analysis was performed using AZILTRON, PRO 532, (USA) with a 532 nm laser excitation. Morphology studies were carried out using a scanning electron microscope (SEM) (JEOL JSM 6390). Transmission electron

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