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ENHANCED MWCNT HYBRID ECG ELECTRODES ON THE BIO-SIGNAL RESPONSES

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Abstract

The non-invasive instrumental diagnostic methods like ECG, use surface electrodes as an interface for wearability on patient body, similar to health monitoring systems. Traditionally, these electrodes are conductive metal like -Ag/AgCl based, hardly upgraded ever. Nonmetal- conductive carbon-based materials like CNT, GO, rGO, Graphene, Graphite etc. have been studied extensively for their versatility characteristics giving scope for changes. Present study used Graphite MWCNT hybrid ECG (GCHE) electrode fabricated by electrophoretic deposition (EPD) technique. In the course of fabrication, biocompatible surfactants were used as dispersant of highly hydrophobic MWCNT, to coat Graphite electrodes. The resulting GCH electrode is dry type and exhibit characters of wearable health systems.

Introduction

The instrumental diagnosis of electrophysiological signals like using ECG, EEG, EMG, EOG, etc. require electrodes and the most widely used are noninvasive surface electrodes. Among the noncommunicable diseases, the disease burden of cardiovascular diseases is highest, largely due to unhealthy modern day life practices. Therefore, a predictive onset diagnosis of symptom is vital always, needed round the clock monitoring even at non clinical setup, for averting an untoward health incident. Many attempts to answer this situation have resulted in development of wearable devices that need electrodes /sensors for this purpose.

An electrocardiography (ECG) surface electrode is the primary interface between the instrumental ECG device and the body of patient. The golden standard of ECG electrode is the disposable Ag/AgCl electrodes as they have a number of benefits like relatively inexpensive, reliability with test of time for ECG diagnosis. These electrodes have few drawbacks as well, they are wet gel type (inconvenient to wear), inconsistent diagnosis on long term usage (gel drying off), not conformal contact, so have motion artifacts in ECG diagnosis¹⁻⁴.

Over time various designs and materials for non-invasive surface ECG electrodes have been studied to alternate the wet gel Ag/AgCl electrodes. These types are broadly looked for following factors 1) reliable and quality signal sensing,2) long term on-body monitoring, 3) reusability, 4) conformal contact wear to reduce body motion artifacts, 5) remote (tele)monitoring for wireless physician assistance and 6) cost, etc. ^{5,6}. Even though all these factors are unjustifiable, important factors have been used in considering ECG electrode design and development. Due to conductivity characteristics of metals (Ag, Au, Ti,) & materials related to them in electrophysiological (ECG, EEG,.) techniques use them in surface electrodes design traditionally for diagnostic monitoring. But needs wet gel and corrosion

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related issues⁸⁻¹¹ correspond to nonmetal- carbon material like Carbon Nanotubes (CNT) due to its unique electrical, mechanical, thermal properties are also studied for electrophysiological surface electrodes for diagnostic monitoring.

There are many types of CNT in use – SWCNT, MWCNT ^{5,6}, to fabricate an ideal surface electrode ('forest/ brush' type)²⁰ for electrophysiological use. These types of electrodes have been fabricated on 1) metals-Cu ¹⁹, Au¹⁸, Pt¹⁷ etc., 2) textile wearable fabrics ^{7,13,16}, and 3) nonmetal-Si, ceramics & polymers ^{12,14} etc. as base for hybrid electrode material. A true "forest" type CNT^{13,14} electrode to fabricate, use and maintain is inviable due to lack of present-day techniques and rarely tried for bioelectrical usage.

Graphite¹⁵ is an allotropic form of carbon. It has electrically conductive property due to its delocalized electron & C-C sp2 hybridization structure. In engineering practices, it is used extensively due to its unique chemical and physical characters. There are a number of graphitic carbon²⁰ materials studied for ECG electrodes including Super B carbon black²⁵, Graphene^{21,24}, rGO²², GO²³, Graphite (pencil lead)^{25,26} etc. Due to the versatile nature of carbon and its compounds & the principles of biomimetics, we used graphite as base for our CNT hybrid ECG electrode.

The methods available for depositing CNT on other materials (metals ¹², carbon ^{1,3}, textiles ¹³, polymer ¹⁰) include drop casting, printing, compositing ⁹⁻¹¹ etc. This study used Electrophoretic Deposition (EPD) ^{17,20}, technique to deposit MWCNT on Graphite base for fabricating ECG electrodes. To EPD MWCNT²³, first we must disperse it in a solvent. Although, MWCNT dispersion has been tried with a number of solvents, for ECG studies water as solvent is needed. In general, CNTs are inherently hydrophobic in nature even on high energy physical method like ultrasonication. Therefore, to achieve dispersion of MWCNT in aqueous medium ²⁴, a biocompatible surfactant was used as agent ^{25,26}. Out of various available biocompatible surfactants, cetyl trimethyl ammonium bromide (CTAB) and sodium Cocoyl Glutamate (Na-CGN) ^{27,28} was studied due to their charged nature.

The Graphite MWCNT hybrid ECG electrode was evaluated and characterized for electrochemical properties and electrophysiological signaling metrics. The study found that this electrode material was uniquely different in its property, ideal for further work on wearable devices of biophysiological applications^{38,39}.

Materials and methods

To fabricate the ECG electrode, graphite (Merck KGaA, Germany) C- 99% 1cm diameter rod was machined to article of measure as depicted in Fig1. with the CNC lath facility to match the dimension of base of disposable Ag/AgCl ECG disposable electrodes¹⁻⁴. Following the machining the graphite base electrode, facilitation of porous structure formation through, gasification was carried out at 850°C using steam of water vapor for 1 Hr¹⁷. Further this base article was thoroughly cleaned with alcohol and distilled water followed by drying in Hot air oven (110°C, 1.5Hr)¹⁸.

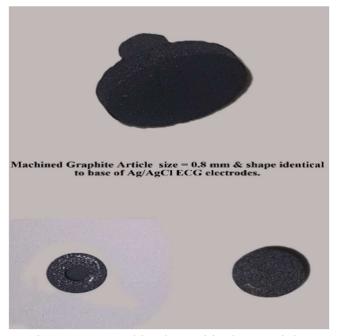


Fig 1. CNC machined Graphite base articles.

The graphite base article base was coated with MWCNT (SAT Nano Tech, China) 99% purity 50-90 nm diameter was used as it is condition using electrophoretic deposition technique ⁵⁻⁹.

The dispersion of MWCNT(1.5mg) in aqueous medium, was by sonication in ultrasonic bath (30min, 25°C) ¹⁵⁻¹⁸using biocompatible surfactant solution (d.d water) of a) cetyl trimethyl ammonium bromide (CTAB from Merck (90mg /100ml) and b) sodium Cocoyl Glutamate from Fengchen Co, China (87.3mg/100ml). The agglomerates were removed by centrifugation (1200rpm) for 20mins and further studies utilized the aspirate part of dispersant ²⁷⁻²⁹.

For Electrophoretic Deposition process³¹, the machined graphite base article was used as the substrate working electrode immersed in above mentioned both surfactant MWCNT suspension, placed at (≈ 2cm) equidistance from counter electrode (SS304). A precision (DC) power source (Tarson Inc, 0-300V/0.4-5A) was used, for our EPD process 15V supply (5min) was utilised. Further to uniformly deposit the whole setup was stirred (220rpm) using magnetic stirrer (IKA C-MAG model). Following the MWCNT coating Graphite base article where thoroughly washed of any residual surfactants by placing in double distilled water for one Hr and annihilated in hot air oven (115°C,1Hr). This procedure was repeated on average three times for each graphite article in each of the both surfactant MWCNT suspension, to minimise the statistical errors of processing. Further to validate the EPD process, gravimetric method was used before and after coating each graphite article.

The EPD coated Graphite MWCNT hybrid electrode article was studied using FESEM (ZEISS Gemini T Sigma300) with available detectors (inlense, SE2).

Electrochemical Impedance spectroscopic analysis of Graphite MWCNT hybrid electrode article was done using BioLogic (SP-300) EIS system³²⁻³⁴. The electrolyte used was physiological normal saline (sodium chloride 0.9%w/v), counter electrode (Pt wire) and standard Ag/AgCl electrode as the reference electrode. The working Graphite MWCNT hybrid electrode article was placed ≈3cm equidistance from

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other electrode while recording the EIS response. The galvanostatic EIS setting was used, with pretreatment range set at 10 nA to 500 mA, applied current range at 1 mA, frequency type scan. For each fabricated Graphite MWCNT hybrid electrode article, three repetitive measurements were conducted at frequency range of 7KHz to 10µHz. These data was later processed with relevant software – Origin lab (Orgin2024b) to average and computing processes.

To determine the cytotoxic nature of the study articles, the MTT cytotoxicity test³⁶ was performed on NHDF (Cat noC-14031; PromoCell Gmbh) cell line with the Graphite MWCNT hybrid electrode article utilising the institutional inhouse facility.

The Graphite MWCNT hybrid electrode article is finally made wearable after adhering them to the extruded foam base of commercial Ag/AgCl ECG electrode, whose gel coating are removed completely. This modification facilitated Graphite MWCNT hybrid ECG (GCHE) electrode to securely attach to subjects' body as well as to the clamp of the ECG machine.

The SCHILLER AT-2 model ECG machine with 12 lead recording was performed on each participant after getting their informed consent by experienced hospital clinician personal. Institutional hospital resources were utilised with healthy volunteering male participants of age 20-60 yrs range. For the ECG study⁵⁻⁸, prior institutional Human ethical and Biosafety committee approval was obtained. For each participant, 12 lead resting ECG data recording each was done using – standard commercial Ag/AgCl ECG electrode (Blank)and - Graphite MWCNT hybrid ECG (GCHE) electrode (both as a) GCHE-CTAB and b) GCHE-Na Cocoyl Glutamate surfactant type). These ECG data where statistically correlated and interpreted ³⁰.

Results:

To assert the EPD coating of MWCNT on the Graphite base electrode in the GCH electrodes article^{29,30}, FESEM provides an indication of the same. The fig2 a) shows the FESEM of Pristine Graphite base electrode material. This is similarly in Fig 2 b) (i) &(ii) show the EPD of MWCNT on the graphite base electrode material using surfactants i) CTAB and ii) Na cocoyl Glutamate respectively.

Table 1: Comparative analysis of Fabricated Electrodes with Reference and Blank

Electrode article	Dimensional	Electrode
	thickness (cm)	Inference.
Pristine Graphite	pprox 0.8	Blank
Graphite MWCNT hybrid (dispersant	≈ 0.8	Working
CTAB)		
Graphite MWCNT hybrid (dispersant Na	pprox 0.8	Working
Cococyl Glutamate)		
Commercial Ag/AgCl disposable	≈ 1.0	Reference

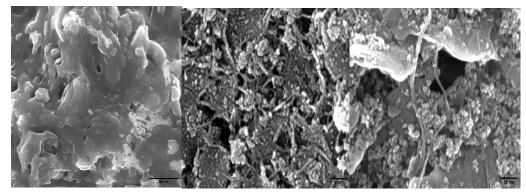


Fig2. FESEM Imagery a) Pristine Graphite base, b) Graphite MWCNT Hybrid electrode (i) CTAB and (ii) Sodium Cocoyl Glutamate surfactants.

Electrochemical Impedance spectroscopy (EIS) technique³¹⁻³⁴ was used to determine the electrochemical responds of the Pristine Graphite system and Graphite MWCNT hybrid (GCH) electrode article system.

To study the bioelectrical signalling using ECG responses of Graphite MWCNT hybrid (GCH) ECG electrode recording was performed on each participant was compared with that of pristine graphite base electrode (blank) and as well with commercial Ag/AgCl ECG electrode(control) ^{37,39}. These ECG studies were performed on healthy participants. Fig 5 is the ECG response of a) commercial Ag/AgCl ECG electrode, b) Pristine Graphite base and c) Graphite MWCNT hybrid (GCH) ECG Electrodes used as ECG electrodes.

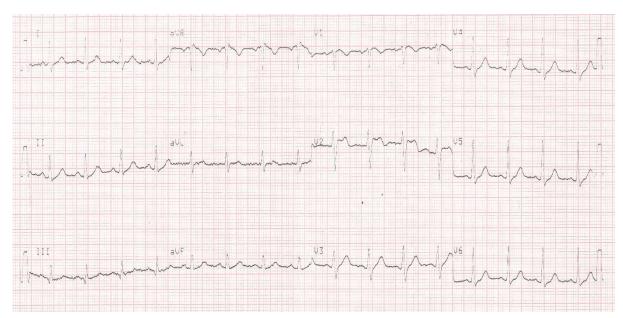


Fig 3. The healthy & resting ECG signal response of commercial Ag/AgCl ECG electrode, Pristine Graphite base and Graphite MWCNT hybrid Electrodes

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The the MTT cytotoxicity test³⁶ result of the Graphite MWCNT hybrid Electrode material, with NHDF (Cat noC-14031; PromoCell Gmbh) cell line system. The blank and the test samples were studied for cytotoxic response prior to designated participant level ECG studies³⁸.

Discussions:

The electrophoretic deposition of MWCNT on the graphite base electrode was confirmed through the FESEM results of the samples, showing clearly that MWCNT is coated on the Graphite base electrode as substrate. The SEM micrograph of the three samples namely, a) pristine graphite base electrode, b) Graphite MWCNT hybrid Electrode (dispersant CTAB) and c) Graphite MWCNT hybrid Electrode (dispersant Sodium Cocoyl Glutamate) articles. In Fig3a has pristine graphite base electrode which show clearly the substrate surface of plain carbon plates arrangement of dimension $\approx 350 \text{nm} - > 1.5 \mu\text{m}$. The other fig3b-i & ii shows MWCNT deposited sparsely in both the graphite substrate surface. Also, the diameter of the MWCNTs varies widely from \sim 25 – 90 nm. A hierarchical carbon structure was created with graphite carbon pores with deposits of MWCNT network to channel the signal responses. The Electrochemical Impedance Spectroscopic frequency-response for the a) Pristing graphite base electrode, b) Graphite MWCNT hybrid Electrode (dispersant CTAB) and c) Graphite MWCNT hybrid Electrode (dispersant Sodium Cocoyl Glutamate) are markedly variable in bode plot they represent. The Fig 4 a) & b) indicates the fact that MWCNT in the GCH electrode and the corresponding values of impedance of each electrode corresponding its response electrochemically. On the other hand, the Graphite MWCNT hybrid Electrode (dispersant- CTAB) and Graphite MWCNT hybrid Electrode (dispersant -Sodium Cocoyl Glutamate), bode plots are markedly similar but with impedance value corelates to the charged nature of surfactants being used. The cationic CTAB surfactant impedance is notably varying to that of the anionic nature of Sodium Cocoyl Glutamate dispersant. This variance is correlated & comparable to the ECG signal pattern in the study.

The ECG signals of the a) Commercial Ag/AgCl ECG surface Electrode, b) Pristine graphite base electrode, c) Graphite MWCNT hybrid Electrode (dispersant CTAB) and d) Graphite MWCNT hybrid Electrode (dispersant Sodium Cocoyl Glutamate) indicate a slight varying respond. Upon studying the ECG signal characteristic and the corresponding EIS response the variabilities can be interpreted to the underlaying physicochemical variability of the nature of surfactant used and their characteristic interaction with the MWCNT and Graphite structures. MWCNT being a linear sp2 carbon structural frame, can interact with van der waals force to surfactants that is explainable by click chemistry. These charged surfactants electrochemically respond as shown in there corresponding Bode plot as well as electrophysiological signal as in the ECG responses by a limitedly understood mechanism, which is to be further studied to understand the mechanism.

Conclusion:

Biomedical non-invasive diagnosis methods like ECG, EEG, EMG etc, relay on surface electrode. The use of surface electrodes for wearable applications and for long term health monitoring needs a dry type biocompatible wearable electrode. Carbon only Graphite and MWCNT based ECG hybrid electrodes,

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are bio-electrically conductive and low cytotoxic in nature. Hybrid fabrication with electrophoretic deposition and characterization validation prove the integrity of molecular structure. These technique needs use of a surface tension reducers like biocompatible amino-acid based surfactants. The use of their surfactant agents induces a relative and correlational change in electrochemical response and as well as bioelectrical ECG signal response. The use of charged biocompatible amino-acid based surfactants correlate to these signal-response variations. These modulated changes in this study shall pay way to future developments in physiological wearable devices.

CONFLICT OF INTEREST:

The authors declare no conflict of interest.

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