OPTIMIZATION AND FABRICATION OF CARBON NANOTUBES, GRAPHENE, AND THEIR HYBRID BASED ECG ELECTRODES FOR HEALTHCARE APPLICATIONS

A Thesis Submitted to

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In

Electronics and Communication Engineering

By

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DECLARATION

I declare that the research work reported in this thesis entitled **"OPTIMIZATION AND FABRICATION OF CARBON NANOTUBES, GRAPHENE, AND THEIR HYBRID BASED ECG ELECTRODES FOR HEALTHCARE APPLICATIONS"** for the award of the degree of *Doctor of Philosophy in Electronics and Communication Engineering* has been carried out by me under the supervision of Prof. N.S. Raghava, Department of Electronics and Communication Engineering, Delhi Technological University, Delhi, India.

The research work embodied in this thesis, except where otherwise indicated, is my original research. This thesis has not been submitted by me earlier in part or full to any other University or Institute for the award of any degree or diploma. This thesis does not contain other person's data, graphs, or other information unless specifically acknowledged.

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CERTIFICATE

This is to certify that the research work embodied in the thesis entitled "**OPTIMIZATION AND FABRICATION OF CARBON NANOTUBES, GRAPHENE, AND THEIR HYBRID BASED ECG ELECTRODES FOR HEALTHCARE APPLICATIONS**" submitted by **Ms. Bani Gandhi** with enrolment number **(2K16/Ph.D/EC/06)** is the result of her original research carried out in the Department of Electronics and Communication Engineering, Delhi Technological University, Delhi, for the award of **Doctor of Philosophy** under the supervision of **Prof. N.S. Raghava.**

It is further certified that this work is original and has not been submitted in part or fully to any other University or Institute for the award of any degree or diploma.

This is to certify that the above statement made by the candidate is correct to the best of my knowledge.

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ABSTRACT

As the world population is increasing rapidly, better health facilities are also in huge demand. Several health issues are unpredictable and the most common are problems related to the heart. Currently, Cardiovascular Diseases account for 32% of deaths globally. Therefore, timely detection of heart abnormalities can provide longer life to people suffering from heart diseases. ECG (Electrocardiogram) is a dominant, standard, and powerful method used to assess cardiovascular health because various heart conditions are reflected as disorders or abnormalities in the ECG signals. The most commonly used ECG electrodes are Ag/AgCl electrodes. These electrodes provide better ECG results for a short duration, as the gel dries up during the course of time, and long-term ECG monitoring is not feasible. Bulky equipment dissolves the chances of continuous ECG monitoring. Therefore, the need of the hour is to fabricate such ECG electrodes which are portable enough to monitor ECG continuously and in real-time. Carbon-based nanomaterials have been utilized due to operational principles and cost. Presently, functionalized CNTs are also been considered due to their advantages like being cost-effective when compared with gold and silver nanoparticles, better dispersing capabilities than unfunctionalized counterparts, better intermolecular bonding between the polymer and CNTs, and better conductivity because of the better flow of electrons.

Hence, Carbon-based Nanomaterials ECG electrodes have fascinated great attention due to their lightweight, comfortable for wearing which do not affect the mobility of the person, and highly conductive for acquiring weak ECG signals. Along with this, the major concern is the toxicity of the nanomaterials. Nanomaterials (Graphene and CNTs) received a green flag regarding this parameter. They are not only safe but provide long-term biocompatibility. If any deviation is recorded in the ECG signal, the report can be sent to the medical expert using various wireless technologies, and immediate help can be provided. Hence, the blend of nanotechnology, electronics, and communication has given health monitoring a new and refreshed outlook which created interest in developing different nano-materials instead of Ag/AgCl electrodes.

In this thesis, different combinations of CNT, Graphene, and their Hybrid have been fabricated using different fabrication techniques and their performances have been analyzed for biomedical applications.

CHAPTER 1

Introduction

Among non-communicable diseases, cardiovascular diseases apart from diabetes are one of the dominant causes of death globally, killing away millions of innocent people every year. To monitor one's heart's electrical activity, ECG is one of the most widely used techniques. In the former years, the ECG was measured with bulky machines and using wet electrodes. In this way, continuous and long-term ECG measurement was not possible. But now, since the emergence of nanoelectronics, improvements have been done in this domain. Nanoelectronics means blending nanotechnology with electronics and communication, where nanotechnology allows incorporating nanomaterials into the fabrication of ECG sensors. The advantage of including nanomaterials is that it will make the sensor portable, flexible, stretchable, and comfortable for the person to wear all day long without causing any uneasiness or distress. In case any abnormality in the ECG is observed, the signals can be sent to the doctor, made possible by advancements in the field of electronics and communication. As a result, the doctor can help the patient with a remedy. In this way, the person's life can be saved. To begin with, let's throw some light on the various nanomaterials.

1.1 Nanomaterials

Nanomaterials are referred to as materials that have an external dimension that measures the maximum value of 100 nanometers, and the internal measurements of 100nm or less. Nanomaterials with the same composition are termed 'bulk materials', but may possess different physical and chemical characteristics. Utilizing nanomaterials is a big step towards the miniaturization of devices and systems. There can be two approaches to producing nanomaterials; the '**top-down**' approach which produces miniature structures derived from large pieces of materials, and another is '**bottom-up**' approach which produces materials atom-by-atom or molecule-by-molecule. Various Nanomaterials and their evolution are depicted in Figure 1.1.

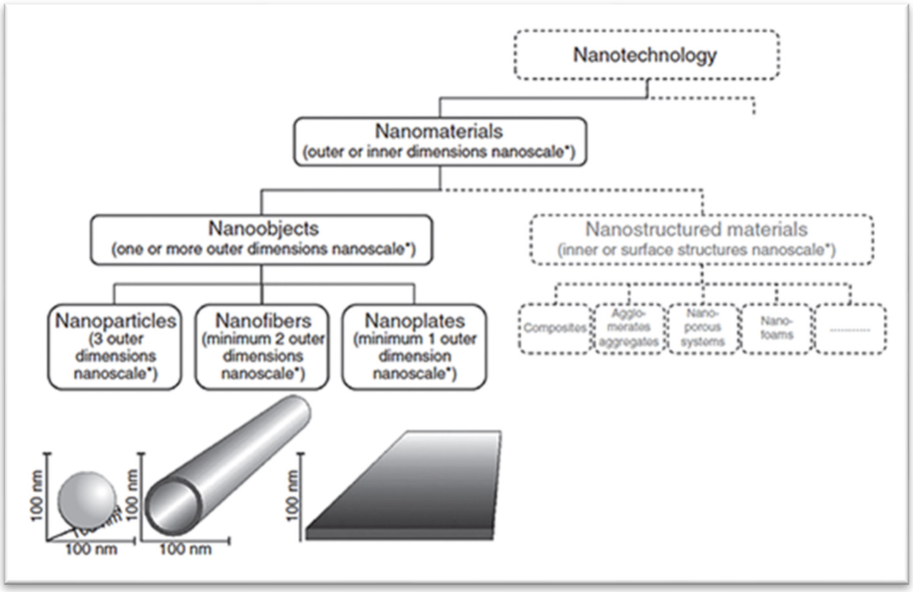


Figure 1.1: Evolution of Nanomaterials

1.2 Dimensions of nanomaterials

The number of dimensions of material outside the nanoscale (100 nm) range determines its classification as a nanomaterial as shown in Figure 1.2.

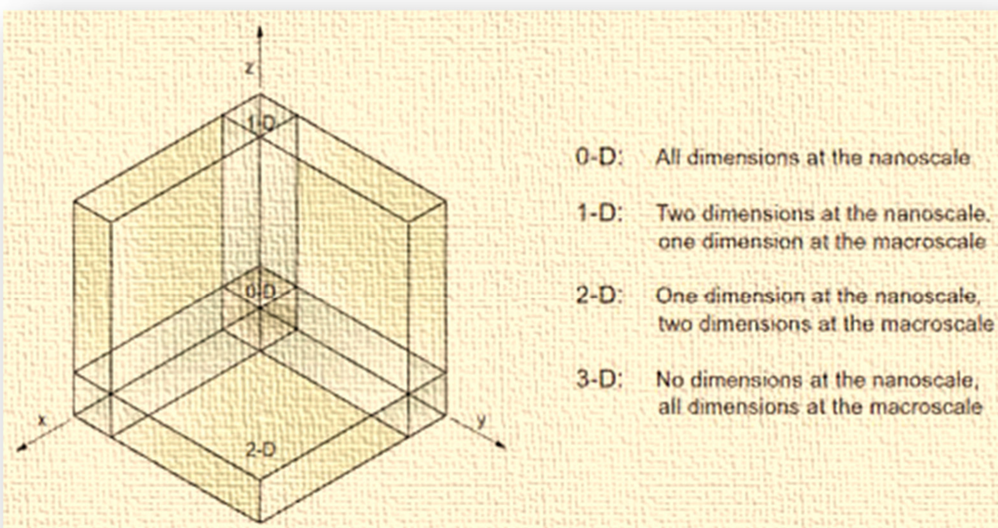


Figure 1.2: Dimensions of Nanomaterials

Therefore, in all dimensions of 0-D (zero-dimensional) nano-materials, no dimensions are greater than 100nm. Nanoparticles are the most common examples of 0-D materials. 1-D nanomaterials lie outside the nanoscale, where the dimensions are larger than 100nm. Carbon Nanotubes, Nanowires, and Nanorods are the most common examples.

2-D nanomaterials also lie outside the nanoscale in two dimensions. The most common examples include graphene, nanocoatings, nanolayers, and nano-films.

Materials that are not confined to the nano-scale in any of the dimensions are described as 3-D materials, the most common examples include nanowire bundles, multi-nanolayers, etc.

1.3 Types of Nanomaterials

A discussion on some examples of nanomaterials to better grasp their properties. For decades, 1-D nanomaterials like thin films and tailored surfaces have been developed and employed in industries including electronics, chemistry, and engineering. Figure 1.3 depicts various types of nanomaterials.

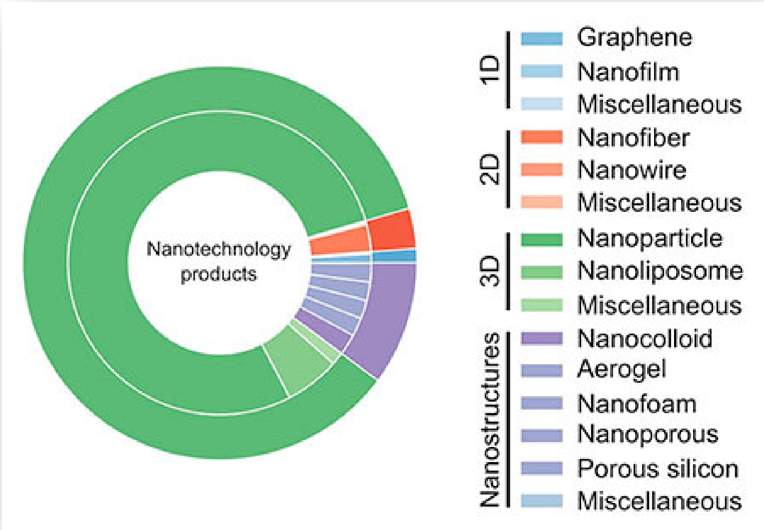


Figure 1.3: Types of Nanomaterials

a) Nanoscale in Zero Dimension

i. Fullerenes (carbon 60)

Carbon 60 (C 60), a new class of carbon material, was discovered in the mid-1980s. C60 molecules are sphere-shaped particles measuring 1nm in diameter, with sixty carbon atoms organized as twenty hexagons, and twelve pentagons also known as the ‘football configuration’.

By resistively heating graphite rods in a helium atmosphere, a technique for producing larger quantities of C60 was developed in 1990. Fullerenes could be used in a variety of applications, including minute 'ball-bearings' for lubrication of surfaces, drug-delivery vehicles, and electronic circuits.

ii. Quantum dots

Quantum dots were first invented in the early 1980s after being theorized in the 1970s. Quantum effects came into consideration when semiconductor particles are made small such that electrons and holes can exist even at low energies. Because energy is associated with wavelength (or color), the optical properties can be adjusted based on the particle size. Therefore, by only regulating their size, particles can be made to emit or absorb specific wavelengths (colors) of light.

Recent applications of quantum dots include solar cells, composites, and fluorescent biological labels, chemical reagents.

b) Nanoscale in One Dimension

i. Thin films, layers, and surfaces

Many products in silicon integrated engineering and manufacturing deeply depend on thin films to operate, and atomic-level control of film thickness is commonplace.

In chemistry, mono-layers are commonly manufactured and employed. Graphene is a well-known material in this novel class of materials. Even with very complex layers (such as lubricants) and nano-coatings, the development and behavior of these layers are relatively well understood from the atomic level upwards. This nanoparticle's huge surface area, along with its capacity to self-assemble on a support surface, could be useful in all of these applications. Surfaces with improved qualities should find use in the chemical and energy industries, even though they are minor advancements. Higher activity and improved selectivity in reactors and separation processes could provide benefits that go beyond evident cost and resource reductions, allowing for small-scale distributed processing (providing chemicals as close to the place of use as possible). In the chemical business, there is already a trend in this direction. Another application may be the manufacturing of high-value compounds like pharmaceuticals on a small scale on-site [1].

ii. Carbon Nanotubes (CNTs)

Sumio Iijima discovered CNTs in the year 1991. CNTs are long tubes made of rolled-up graphene sheets. CNTs are classified as Single-walled CNTs (SWCNTs) and Multi-walled CNTs (MWCNTs). Both of them are characteristically a few nanometers in diameter and a few micrometers to centimeters in length. Because of their novel chemical and physical properties, CNTs have assumed an important role in the context of nanomaterials. CNTs have Young's modulus exceeding 1TPa, making them mechanically strong and as stiff as a diamond while being flexible, and showing extremely high electrical conductivity. CNTs have a wide range of potential applications like nanoelectronics, display devices, sensors, and reinforced composites due to their remarkable properties [2].

CNTs can be grown using a variety of methods. The selective and uniform production of CNTs with specific dimensions and physical properties, on the other hand, has yet to be accomplished. Concerns have been raised about the safety of CNTs due to their potential size and shape similarity to asbestos fibers [3].

CNTs are cylindrical nanostructures with a length-to-diameter ratio of up to 132,000,000:1 and are promising candidates as conductive materials for flexible electronics [4]. CNT's unique nanostructure offers many features required in transparent electrodes, including high-intrinsic conductivity, flexibility, solution-processability, and potential low-cost production [5]. To date, CNTs have been applied to flexible devices like strain and pressure sensors [6], biological and chemical sensors [7], touch-screens [8], displays [9], solar cells, and supercapacitors [10-11]. However, CNTs are still faced with the issues of the high cost and scalability in the means of the separation of metallic CNTs from semiconducting CNTs.

iii. Inorganic nanotubes

Shortly after CNTs, inorganic fullerenes type materials and nanotubes, which were formulated on layered composites like molybdenum-disulfide were recognized [12]. They possess exceptional lubrication characteristics, are resistant to shocks, possess high storage capacities for lithium and hydrogen, and many more. The use of oxide-based nanotubes like titanium dioxide in catalysis, photocatalysis, and energy storage is being investigated [12].

iv. Nanowires

Nanowires are self-assembled ultrafine wires or linear arrays of dots. They can be made of a variety of materials. Gallium-nitride, Silicon, and Indium-phosphide semiconductor nanowires have confirmed extraordinary electronic, magnetic, and optical properties [1].

The nanowires are synthesized with sophisticated techniques like self-assembly techniques, in which atoms naturally organize themselves on stepped surfaces, Chemical Vapor Deposition, Molecular Beam Epitaxy, Electroplating, etc. Typically, the 'molecular beams' are derived from thermally evaporated elemental sources [3,5,8].

Nanowires could be used in high-density data storage, either as magnetic read heads or as patterned storage media, as well as electronic and optoelectronic nanodevices, for metallic interconnects of quantum devices and nanodevices [9-11].

v. Silver nanowires (AgNWs)

AgNWs are the most conductive nanomaterials and have gathered a lot of attention from researchers [13-14]. The diameter is approximately tens of nanometers or even less, and no restriction on the length. Due to its 1-D nature, it possesses high aspect ratio morphology and offers properties like optical transmittance and high DC conductivity [15] for applications of transparent and flexible electrodes. In addition, nanowire antennas show a superior high-frequency response [16]. However, there are some challenging issues with silver nanowires that limit their applications in flexible electronics. A highly non-uniform topography with AgNW electrodes can cause shorting through other layers, especially when AgNWs are used as lower electrodes in devices like solar cells [17]. Additionally, most flexible substrates are not resistant to relatively high annealing temperature (~200 °C) which is needed for higher conductance of AgNW electrodes.

vi. Nanoparticles

Nanoparticles possess diameters of less than 100 nm and have improved properties when compared to bigger particles of the same material [18].

Nanoparticles are abundant in nature, like byproducts of photo-chemical and volcanic activities, as well as those produced by algae and plants. Nanoparticles have been generated as a consequence of combustion, food-cooking, and vehicle exhausts. Nanoparticles are interesting because they have unique properties (such as chemical reactivity and optical behavior) when compared to larger particles of the same material. Nanoparticles have a wide range of potential applications, including cosmetics, textiles, and paints in the short term, and methods of targeted drug delivery to a specific site in the body in the long run. As nanoparticles can also be organized as multiple layers on surfaces, they make perfect catalysts, as enhancing the surface area improves surficial properties which enhances the surface area thus improving surficial activities, which makes them perfect catalysts. Nanoparticles are currently used in cosmetics, and their improved properties may have an impact on their toxicity [4].

c) Nanoscale in Two Dimensions

2-D nanomaterials like wires and tubes spawned a substantial amount of attention among researchers and scientists very recently, due to their novel and amazing mechanical and electrical properties in particular [5].

i. Graphene and other layered materials

Graphene is like a honeycomb structure of carbon atoms at the atomic scale. Because of its unique combination of excellent qualities, graphene is quickly developing as one of the most promising nanomaterials, with applications spanning from optics, electronics, biodevices, and sensors. Graphene-based nanomaterials, for example, offer a lot of potential in the field of energy. Here are some instances from recent years: Activated graphene provides excellent supercapacitors for energy storage; graphene boosts both energy capacity and charge rate in rechargeable batteries; Graphene electrodes could pave the way for low-cost, lightweight, and flexible solar cells, while multifunctional graphene could serve as a promising substrate in catalytic systems [4].

2-D silicon would be an especially interesting graphene analog because it would be produced using better semiconductor techniques as it can be more effortlessly integrated with the prevailing electronic systems. Graphene is another allotrope of carbon which is a one-atom-thick planar sheet with a honeycomb lattice arrangement. It is rapidly emerging as conducting carbon-based nanomaterials for transparent electrode applications. In contrast to CNTs, graphene possesses better conductivity, higher transparency, and some other unique physical and chemical characteristics. For instance, Bae et al. [3] illustrated large-area-doped graphene sheets with a roll-to-roll transfer procedure which shows 30Ω/sq. In addition, graphene is chemically the most reactive form of carbon, owing to the high surface area and the highest ratio of edgy carbons in comparison with similar carbon-based materials [4]. As a result, Graphene is an ideal candidate for thin-film electrodes in chemical and temperature sensors.

d) Nanoscale in Three Dimensions

i. Dendrimers

Dendrimers are sphere-shaped polymeric particles devised by nanoscale-hierarchical-self-assembly. Dendrimers come in a variety of sizes, the smallest being several nanometers. Dendrimers are commonly used in inks and coatings, but they possess many interesting characteristics which include them in applications like drug delivery, and environmental clean-up as they can easily trap metal ions, from the water after ultrafiltration techniques and can be filtered out [19].

1.4 Applications of nanomaterials

Figure 1.4 depicts the distribution of products involving nanomaterials and nanotechnology across industries.

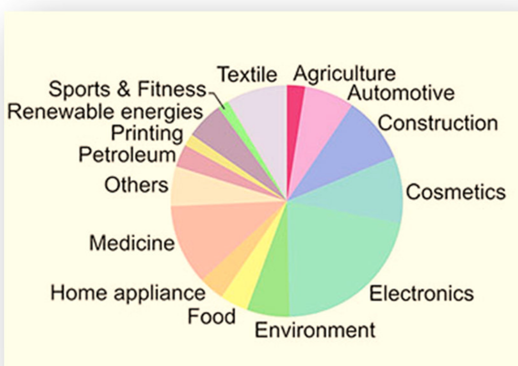


Figure 1.4: Applications of nanomaterials

When nanotechnology revenues are broken down by sector, it is clear that manufacturing and materials pitched in the most to the entire nanotechnology profits. This is to be anticipated, given that the primary phase of growth for any general-purpose technology embraces initial interdisciplinary research. Nevertheless, as nanodevices are combined with existing technologies, this trend is expected to shift more toward application sectors in the coming years.

i. Nanomaterials in Bioelectronics

Due to their high aspect ratio, surface area, and high-grade mechanical, chemical, and electrical properties, nanocarbons such as graphene and carbon nanotubes have emerged as novel materials for bioelectronics and biosensing [2,7]. Furthermore, nanocarbon has excellent flexibility for body conformable electronics and is the ideal material for wearable applications due to its lightweight. CNTs and Graphene nanostructures are beneficial for bio-electronics and bio-sensing applications.

Bioelectrical signals that can reflect heart function are widely studied using ECG, EEG, EMG, and apex cardiogram (ACG). As an example, in an ECG test, electrodes are attached to various parts of the human body to measure biopotential. ECG curve can be obtained after further analysis. Wet Ag/AgCl electrodes that are directly attached to the skin via electrolyte gel are widely used in ECG tests. The wet electrodes and electrolyte gel, on the other hand, may cause skin rashes and allergies in the subjects. Furthermore, traditional methods may restrict the subject's activities, which is inconvenient for real-time monitoring. Nanomaterial-based dry electrodes in wearable ECG sensors for real-time detection are being investigated to improve the situation.

1.5 Literature Survey

1.5.1 ECG electrodes using SWCNT

Using SWCNT, DNA, and Chitosan, Philip Whitten, et al. [20] created electrodes in 2006 that could be used as biosensors. Results showed that when compared to the bucky paper, DNA and Chitosan electrodes were found to be superior in terms of conductivity. The fact that these electrodes can be used in water without losing any of their mechanical properties makes them an excellent choice for many applications. The electrodes were only developed by the authors, but they have yet to be used practically. As a result, there is no evidence to support this claim because ECG portrayal is not there. Pacemaker and ECG pads can be made with these nanocomposites, according to the researchers who mentioned this in their paper. Because of the high conductivity of CNTs**,** in 2013 Penghua et al [21] fabricated a 3-in-1sensor that could sense temperature, gas, and a pressure-based ECG sensor. They developed this sensor using CMOS. They infused SWCNT between the Drain and the Source of the CMOS. Then after 2 years in 2015, Bashir et al [22] developed electrodes using Patterned Vertically Carbon Nanotubes (pvCNT). They also filed a patent for the same invention. The CNT pillars were synthesized and used. The CNT pillars were affixed on the surface of the substrate with unwavering shape, length, spacing, and size. The objective behind using CNT pillars (pvCNT) is that they maintain good conductivity on any kind of skin, including rough skin, or hairy skin. These are also capable of acquiring ECG signals with low noise. In addition to this, the fabricated pvCNT electrodes can function for extended periods, ranging from hours to days to weeks to months. Moving further with discoveries, an ECG electrode printing method was demonstrated in 2015 by Rao et al [23]. The ECG signal must not be interfered with by excessive potential or voltage in order to obtain an accurate reading. Field-Effect Transistors (FETs) and membranes that are ion-sensitive are therefore preferred. These electrodes allow for continuous monitoring as well as online monitoring of ECG signals. It is also effective in sweaty conditions. The author, on the other hand, only built the electrode and no discussion was there on the ECG monitoring All the merits of the CNTs led to another discovery of the ECG electrodes by Yamamoto. Et al [24] in 2016, where silver was used to fabricate the electrode. The PET substrate was used for the connectivity to the temperature sensor, which was then heat-cured to 70°C. After that, a solution of CNT-ink and PEDOT: PSS was applied to the substrate and left to dry at 70°C for a second time. PDMS provided the necessary adhesiveness. ECG results were quite commendable in both cases; pre-exercise and post-exercise. As another year passed D. Yamamoto et al [25] fabricated a 3-in-1 sensor platform that allowed measuring sensors like ECG, acceleration, and temperature. The materials used for fabrication were Silver nanoparticles (AgNPs) and CNT ink. After testing the electrodes, it portrayed great ECG results. CNT and Color Tunable Organic Led Emitting Diodes (CTOLEDs) were used to fabricate an ECG sensor in 2017 by [26] Ja Hoon Koo et al [26]. SWCNTs were used for a P-MOS inverter. Four CNT-based transistors were used to boost the ECG signal. Real-time recording of the ECG signals was done. Flexibility and conformability made the electrodes suitable for use on a wide range of skin types. LEDs were used to display colorimetric displays of ECG waves (normal or abnormal). As a result, abnormal ECGs are indicated by CTOLEDs that show a dark-red color, while normal ECGs show a dark-blue color. The software was used to display data from the wrist sensor, which was attached to a user's wrist for testing purposes. The SWCNT film was used to fabricate novel stretchable, flexible, conductive, biocompatible, and transparent hydrogels in 2018, according to E. P. Gilshteyn and colleagues [27] it was done in two ways: first, the SWCNTs were directly delegated to hydrogel, and then film deposition was applied to hydrogel that had already been stretched. ECG results were comparable with the commercial electrodes.

In addition to Adhikari et al [28] created new Electrically Organic Fibers (ECF) which are organic in nature. The advantage of these electrodes is that the fabrication process is simple and cost-effective. The procedure required staining the ECFs with SWCNT and regioregular poly(3hexylthiophene) (rr-P3HT). Here, two physiological signals were obtained namely EMG and ECG. After the fabrication of the sensors, people started incorporating the CNTs into the textiles. So, various textile-based ECG sensors using SWCNTs have been discussed as follows.

a. Textile-based ECG sensors

Electrodes with cotton-yarns and SWCNT were developed in 2018 by Zhao et.al. [29]. Sensors were created by dipping and drying. During the procedure, the cotton-yarn was soaked into the SWCNT mixture and then hung to dry at room temperature. Tests showed that they could mediate ECG signals with quite reduced distortion and attenuation after installing the sensor. In addition, the authors noted that the data was of high quality. A textile-based ECG sensor based on SWCNT, AgNWs, and Polyutherene (PU-nanoweb) was recently fabricated by E. Lee et.al [30]. Additionally, 15 grams of SWCNT solution was used to soak the PU-nanoweb, followed by 5g. of AgNWs. In order to increase conductivity, the sensor was treated with ethanol and placed in the oven.

1.5.2 ECG electrodes using MWCNT

To begin, J.H et.al [31] used MWCNT and PDMS to create polymer-based ECG electrodes that were both flexible and stretchable. With a 1.5% concentration of MWCNTs, the best ECG output was observed, due to good conductivity (electrical). Electrodes were able to operate for long periods of time because they were flexible. Results were also compared to those obtained using more traditional ECG electrodes, and the new electrodes were found to be highly successful. MWCNT and PDMS-based electrodes were created by H.C. Jung et al. [28] in the same year. It was possible to attach and use these electrodes with the same traditional ECG machines. Also, these electrodes worked well in sweat conditions and also while the person was in motion. In addition to this, erythema and itching were also not detected. Then, a year later in 2015, S.M. Lee et.al [32] again used MWCNT and PDMS for developing the ECG electrode and an EEG device which is in the form of a headphone. The ECG patch has a thickness of 200-300 micro-meter. The manufactured patch was flexible and adaptable to a variety of skin types, including wrinkled and rough ones. It was observed that the adhesion of the patch decreased slightly after certain attachments and detachments, but the adhesion was redeemed after wiping the patch with alcohol. Again, in the same year, B. Lui et al [33] developed electrodes with a 3-step process namely ultra-sonication, stirring, and in situ polymerization, in addition to replica technology. The finest combination was attained at 12 hours of sonication with 5wt% CNT content showing the best results. Mass production was possible, and expensive hardware wasn't really needed for the fabrication of this sensor's components. Another discovery of the ECG electrode was made in the same year by A.A Chlaihawi et al [34] produced electrodes using PDMS and MWCNT. To allow for greater flexibility MWCNT and PDMS composites were bar-coated onto the PET substrate after the silver ink was screen-printed onto it. The electrodes of three diameters 8mm, 12mm, and 16mm, were created. The electrodes with the largest surface area outperformed the standard Ag/AgCl electrodes. In both the resting and moving positions, the subject's ECG was recorded and analyzed separately. Also, in 2015 L. M. Rivera et al [35] developed a dry ECG sensor. It was made using Polyaniline (Pani) which was the main matrix. Then acrylic resin was used as the host matrix and then the material was blended with MWCNT to increase the strength and then further doping was done with Silver (Ag) and finally functionalized with CI (Chemical Ionization). When the calculations were done, it was observed that the electrical resistance was found to be 1000Ω and electrical conductivity was found to be 0.002S/cm for an area of $0.012m^2$. Although this electrode has not been tested for ECG measurements, the authors have mentioned in the paper that this composite will be useful to monitor ECG. Gel-less MWCNT and PDMS electrodes were also developed in [36] in 2015. Ag ink was used to screen print the sensor onto PET substrate by Amer et al. After that, the screen-printed sensor was bar-coated with a mixture of MWCNT and PDMS. This invention resulted in a better connection between the electrode and the skin. Also, it conformed to any sort of skin excellently. The results were obtained for both relaxed mode and movement mode. The results portrayed that these were more elastic than Ag/AgCl electrodes and motion artifacts were reduced in this case. Moving a year forward, one more invention was added to the bucket in 2016 by T. Sekitani et al [37]. The authors fabricated a highly conductive and completely biocompatible nanocomposite of the aqueous hydrogel and MWCNT. The electrodes were flexible when bending tests were performed. Also, good-quality ECG signals were obtained. In the same year, B. Liu et al [38] developed polymer-based electrodes using MWCNT, PDMS, and AgNPs to increase conductivity and flexibility. In addition to this, aPDMS (adhesive Polydimethylsiloxane) was also used to increase the electrode-skin contact, which further reduces motion artifacts. With this invention, the ECG signals could be transmitted wirelessly. The authors conducted an 11-day performance test of the electrode and it was observed that the signal degradation was not much during the course of time. Again in 2016, Levan et al [39], developed electrodes with acrylic paint, and MWCNT. The diameter of the electrodes was 20–30 mm and it was thick in the range of 0.005-1.5 mm. While submerged in water, the ECG electrodes maintained their electrical conductivity. PVDF (Polyvinylidene Fluoride) filters were coated with MWCNTs in 2016 by Juan et al [40]. As with the Ag/AgCl electrodes, the fabricated electrodes showed good conductivity and electrical resistance. 2016 witnessed another discovery by B.Liua [41], who developed a flexible nanocomposite with PDMS used for constructing the base, AgNWs, and an adhesive layer which is a blend of MWCNT and aPDMS. The acquired ECG signals could be transmitted wirelessly. The electrode was sturdy and could be easily conformed to the skin surface (any kind) with reduced motion artifacts. Also, an eight-day performance assessment was conducted and on the 6^{th} day, it was observed that the signal has deteriorated but the signal could be recovered by applying alcohol and cleaning it. Consequently, in the year 2017, R.P. Tripathi et al [42] fabricated stretchable, and flexible electrodes which were analogous to a skin-like film, basically for prolonged ECG monitoring. It was fabricated using PDMS and MWCNT. Their performance was superior to that of the standard Ag/AgCl electrodes. These electrodes were water-resistant. The size of the electrode was compact and also the cost of production can be decreased by making large quantities. In 2017, Lui et. Al [43] developed electrodes using AgNP, MWCNT, and PDMS. A fourteen-day performance test was done to evaluate the electrode performance and it was observed that these electrodes were well suited for long-term monitoring. Vests were made with three electrodes sewn into them. In addition to being flexible, the electrodes were also washable. Another discovery of elastomeric polymers was made by Yourack Lee et al [44] illustrating alumina–passivated non-percolating conduction via aligned-CNT elastomeric sheets. These sheets reduce low-frequency noise. The ECG was acquired using the “3-lead system” and the results obtained were similar to the standard Copper wires. Yet another discovery was made by Yuki Yamamoto et al [45] reporting the effect of thickness of the film for measuring skin temperature, reliability, and consistency of gel-less ECG electrodes and adhesive force. Also, the measurements were done in a real-time scenario by placing the sensor on the person's chest. The ECG sensor was made of MWCNT, PDMS, and Ethoxylated Polyethylenimine (PEIE). The temperature sensor and ECG sensor were integrated to gauge the health conditions. The ECG sensor was reliable, flexible, and wearable approximately 100 times without losing its adhesion between the electrode and the skin. The ECG was observed in three conditions- before exercise (without sweat), after exercise (with sweat), and after wiping the sweat. ECG results obtained were pretty decent. In recent years, ECG electrodes that are stretchable, flexible, and printable were developed [46] by Amer et al in 2018. A film-like structure was created by printing silver ink onto a PET substrate. It was then coated with PDMS and MWCNT using the bar-coating method with 8mm, 12mm, and 16mm radii electrodes. These electrodes were less noisy due to good conformality on the skin. Again, in the same year, Jeong Hun Kim et al [47] fabricated an efficient and effective MWCNT and PDMS-based electrode. The developed electrode was flexible, stretchable, and highly conductive. The process of fabricating the electrode was simple and cost-effective. Also, the ECG signals obtained were of good quality. In 2018, Yadhuraj S.R et al. [48] developed polymer-based ECG electrodes. PDMS was used in the fabrication process because it is cost-effective and biocompatible. Here, several variants of electrodes were developed namely, only PDMS-based electrodes, Nickel and PDMS-based, and MWCNT/PDMS mix. Later, the testing was done using the lead-II ECG configuration. The dimensions of the electrode were 2cm x 2cm and its results were compared to the conventional electrodes. The MWCNT and PDMS-based electrodes provided an accuracy of 94% and the signal amplitude was better than PDMS and nickel-based electrodes. Also, the motion artifacts were much higher in dry electrodes compared with Ag/AgCl electrodes. In the same year, Zhenlong Huang et al [49] report an architecture of 3-D integrated electronics by blending approaches and strategies in microfabrication and material design. The 3-D device is created layer-by-layer via transfer-printing-pre-designed flexible and stretchable circuits on elastomers and makes vertical interconnects with the help of laser ablation and controlled soldering technique. With the help of this framework, the authors created a human-machine interface device that is implemented on a 4-layer architecture and offers an 8-channel sensing port along with Bluetooth technology for wireless communication. The ECG sensor was made using MWCNTs Silicone and Polytetrafluoroethylene (PTFE) as substrate. The sensor was around 1mm thick. Also, the signals were of good quality. After numerous measurements, the electrodes could be recycled by cleaning them with IPA to eliminate any dead skin cells or dirt. Again in 2018, Anubha Kalra et al [50] prepared and mechanically tested the thin polymer electrodes to estimate and measure proficiently the physiological signals while taking care of the skin-conformity. Here, PDMS is used in preparing thin-film electrodes with different Young's moduli. The electrodes were fabricated with two different conductive materials- MWCNT and Graphene Nanopowder (GNPs). Thereafter, sensors were made wet in N-N Dimethylformamide (DMF) before dispersing in PDMS in an ultrasonic mixer. It was done to dodge the lumping effect in the PDMS and also to encourage unvarying dispersion. The dimensions of the patch were 30mm x 30mm x 2mm and the conductivity was found to be 1µS. The conductance of the fabricated electrodes was compared with Ag dry and Ag/AgCl electrodes. Out of the 4 sensors, the MWCNT and PDMS sensors stood third in the line for conductance. MWCNT and PDMS electrode was found to be the most stretchable and flexible. Now recently in 2019, Jung et al [51] developed self-adhesive, flexible, and dry electrodes made up of MWCNT/aPDMS. A 7-day test was done to analyze the performance of the sensor, it was observed that it did not cause any itchiness or any sort of skin irritation. Also, the signal degradation was not very much over time. Again in 2019, MWCNT and PDMS were used by M. Chi et al [52] to create a gel-free, flexible electrode. These electrodes were useful in the case of long-term monitoring. The fabricated electrodes had better outputs concerning motion artifacts, i.e., motion artifacts were more in Ag/AgCl electrodes than the fabricated ones. After a week, the skin did not show any sort of irritation.

a. Pin-shaped Electrodes:

In 2005, Giulio et al. [53], developed and designed Electrode Sensor for Biopotential Measurement Applications (ENOBIO) in this study. It was fabricated to eradicate the noise levels and aggravation associated with using conductive gel. ENOBIO was developed using the concept of nanotechnology, basically to remove the noise caused by the electrolytic gel, and also to maintain a decent contact impedance to further minimize the interference noise. Here, the authors make use of a CNT array/forest-based bio-potential sensor. The array-based CNTs penetrate the outer layers of the skin, thus enhancing the electrical contact between the sensor and the skin. This electrode was not specific to ECG monitoring, but in 2006 the same group [54], designed electrodes wherein the tip of the electrode was enclosed with the MWCNT forest. It was a brush-like assembly of the MWCNT forest. This was done to enhance the conductivity of the sensor by increasing the contact between the sensor and the skin. Again, in this paper, they did not report any results of the bio-potential signals. ECG electrodes were tested on humans in 2007 by this group, and wireless transmission of the signals was also tested [55]. This was followed by the fabrication of pvCNT-based ECG electrodes on stainless steel foil in 2015 by M.J.A. Saude and colleagues [56]. There was a 100-200nm gap between each of the pillars of MWCNT growth. Individual pillars are more breathable. Polypyrrole (PPy) (conductive polymer) in the previous electrodes, was coated with a thin layer by the same group in 2018. This was used to enhance the mechanical properties of the sensor and the results were similar to the Ag/AgCl electrodes. The output exhibited stable, reliable, and low-impedance through long and short periods of measurements when the comparison was made with the conventional electrodes. These improved electrodes had increased impedance but were more strong and more stable in mechanical adhesion with the substrate. Therefore, making it safer and more hygienic to use on the skin surface. Although the accumulated ECG waveform using pvCNT possessed more noise, in a nutshell, all the ECG wave components were visible and decent enough [57]. Again in 2015, H.L. Peng et al [58] developed a flexible electrode with CNT micropillars viz Micro-Electro-Mechanical Systems (MEMS) technique. After electrode testing for 2 days, the results were stable while the person was at rest or in motion. Then a year later in 2016, T. Kimet et al [59] designed and developed Conductive Dry Adhesives (CDAs). This was a carbon nanocomposite, which was elastic in nature and had a hierarchical structure stimulated by gecko activity. The ECG monitoring pad was devised by the authors of this paper. The pad adheres to all types of skin, including wrinkled, rough, and dry. The benefits of using this ECG pad are that it is water-resistant and had excellent conformality with the skin surface. Also, it had a self-cleaning capability and can be re-used plenty of times.

b. ECG sensors embedded into textiles

In 2013, Lam et al [60], fabricated dry fabric-based ECG sensors using MWCNT. The textile on which the sensor was fabricated was cotton. Here the authors applied MWCNT on the cotton cloth and for adhesion purposes, tapioca starch was used. The ECG results of the fabricated electrodes had better results than conventional Ag/AgCl electrodes. The process of fabrication was simple and inexpensive. Three years later in 2016, McKnight et al [61], developed cost-effective, flexible, and dry ECG electrodes. The ECG output of this sensor was compared with two more sensors one of which is made of stainless-steel conductive thread and the other one is made of 3-D printed conductive fiber. These electrodes displayed better ECG results than the two of them and were of good quality.

1.6 Motivation and Objective of the thesis

In today's world of a pandemic where there are a lot of diseases that need to be taken care of, this work concentrates on easing the monitoring of at least one of the major non-communicable diseases, which is CVDs. Some of the following challenges are motivating factors to conduct research work in this field:

i. Dispersion of nanomaterials was a tedious task
ii. No experimentation with new combinations of nanomaterials, which would require less concentration of nanomaterials
iii. Wireless transmission was done in limited research works
iv. High electrode-skin impedance was observed
v. Only health professionals were able to see the ECG data
vi. IoT systems were not incorporated with CNT-based electrodes

Based on the above factors, the objectives of this study were formulated, which are discussed as follows:

i. To select the cost-effective and efficient material for fabrication
ii. To identify a simple and cost-effective fabrication technique
iii. To design and fabricate, and measure nanomaterials-based ECG electrodes with varying concentrations and combinations
iv. To perform a topological analysis of the materials
v. To develop an ECG monitoring system
vi. To compare the ECG results with the various fabricated group of ECG electrodes

1.7 FABRICATION AND DESIGN CONSIDERATIONS

Nanomaterials-based ECG electrodes are fabricated for continuous, long-term monitoring. The fabricated electrodes should be flexible so that they can be used in robust conditions, and most importantly should be bio-compatible so that it does not cause any skin irritation or allergy. Therefore, the following considerations should be taken into account to support all the parameters as mentioned.

A. Selection of materials:

It is extremely important to check the feasibility of the nanomaterials if they can be incorporated to make substantial ECG electrodes. Materials must be extremely conductive to capture feeble ECG signals. The selected material should possess great dispersion capabilities because nano-materials have Vander wall forces, which restrict them to mix with any polymer. The nanomaterial should be lightweight so that the system can be made portable. The nanomaterial should possess highly flexible and stretchable traits. Lastly, the materials should be cost-effective.

B. Equipment Requirements:

The equipment required should be readily available. The machines need to be user-friendly, easy on the hand, and pocket friendly. Also, minimum equipment should be required for the fabrication process.

C. Fabrication process:

The fabrication process should be simple, and the number of steps should be limited. It is very important to do proper calculations for making proper composites and to identify where the percolation threshold has been achieved. It is important to note the time required to keep the solutions in the oven or sonication or air-dry, etc.

D. ECG monitoring system:

The ECG monitoring system should be able to provide continuous and long-term ECG results. The hardware equipment for the system should be cost-effective, durable, and reliable. Also, the software involved in the system should be easy to create and should provide accurate results with high reliability and minimum or no delay.

1.8 Organization of the thesis

Chapter-1: In the first chapter, a brief introduction to the evolution of nanomaterials has been discussed. Following this, Graphene and CNTs have been discussed along with their characteristics. The importance of Graphene and CNTs as an integral part of the technological advancement in the medical field, especially in the sensor industry has been discussed. Later, the importance of Nanomaterial-based ECG sensors has been explained along with ECG basics and the importance of wireless communication (IoT) with nanotechnology has been briefly described.

Chapter-2: In the second chapter, various CVDs have been discussed. Along with this, ECG measuring techniques have been described in detail, and their patterns, and how the electrodes are placed for the various lead system of the ECG systems have been discussed. Following this, the evolution of ECG electrodes has been discussed including Pre-gelled electrodes, Polymer-based electrodes, Fabric electrodes, Bulb/Clamp Electrodes, Polymer, and Fabric-based electrodes, Pin-shaped electrodes, and Bulk sensors. In this work, a 3-lead system has been employed because this work aims to deploy a system where remote, long-term, and continuous ECG monitoring can take place.

Chapter-3: In the third chapter, the overview of flexible electronics has been described. A detailed description has been done of how CNTs and Graphene were taken into account for fabricating ECG electrodes for this work. Carboxylic-acid functionalized CNTs, which include both MWCNT-COOH, SWCNT-COOH, and Graphene have been employed to fabricate ECG electrodes of various concentrations. Since polymer-based electrodes have been fabricated, a detailed description of various polymers has been discussed, as well as the reason for using Polydimethylsiloxane (PDMS) in fabrication. The importance of Internet-of-Things (IoT) has been discussed for providing a remote ECG monitoring system. In addition to this, a discussion on software and hardware components required for this system has been done. In this research, the framework of an intelligent ECG tracking system which is based on the IoT cloud is anticipated and implemented. The accumulated ECG facts and figures from the patient are communicated to the IoT cloud platform with the help of (Wireless Fidelity) Wi-Fi. Also, for convenient and on-time access to the data, HTTP has been deployed in the IoT-cloud platform. An (Application Programming Interface) API was created for implementing the Graphical User Interface (GUI). The ECG data was stored in the API using a database MongoDB. The system performed effectively, and the results were obtained successfully.

Chapter-4: In the fourth chapter, various fabrication techniques like Spin Coating, Bar Coating, Doctor Blade technique, Metal Patterning, Screen Printing, Mold casting, and other unconventional techniques have been discussed in detail with their advantages and disadvantages. In this research, the mold-formation technique has been employed as it is economical, easy to employ, and materials are readily available. The equipment(s) required are not bulky. After fabricating the molds, the person is only required to pour the composite into the mold and after thermal curing the electrodes are ready. Once the molds are cast, several electrodes can be developed via the 'replication' process.

Chapter-5: In the fifth chapter, two groups of electrodes are fabricated MWCNT-COOH/PDMS and SWCNT-COOH/PDMS using Fabrication Technique-I. In this technique magnetic stirrer, probe-sonicator and oven are used. First, the molds are fabricated, then CNT-COOH with IPA is first distributed using the magnetic stirrer, then probe sonicated with IPA, and later with PDMS. The final composite is poured into the molds and thermally cured, and lastly, the electrodes are demoulded from the mold. Thirteen different concentrations 0.1wt%, 0.25wt%, 0.5wt%, 0.75wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt%, 3wt%, 3.5wt%, 4wt%, 4.5wt%, and 5wt%, for both the groups were fabricated. Physical characterizations like Transmission Electron Microscopy (TEM), Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FITR), and Raman Spectroscopy were performed. Tests like capacitance, resistance, and impedance were also carried out. The ECG was monitored using the system developed in Chapter 3. The ECG results were compared with the Ag/AgCl electrodes. Of the two groups, the best results were achieved from 3.5wt% concentration with SWCNT-COOH/PDMS. The peaks were well defined when compared with 4wt% MWCNT-COOH/PDMS electrodes. Also, better waveforms are achieved with less concentration of the nanomaterial.

Chapter-6: In the sixth chapter, two groups of electrodes are fabricated- Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS(G-II), and SWCNT-COOH/MWCNT-COOH/PDMS(G-III), and Graphene/PDMS, Graphene/MWCNT-COOH/PDMS (G-IV), and Graphene/SWCNT-COOH)/PDMS(G-V) using Fabrication Technique-II. In this technique, only a bath-sonicator and oven are used. First, the molds are fabricated, then Nanomaterials with IPA are first distributed using the bath-sonicator, and later with PDMS. The final composite is poured into the molds and thermally cured, and lastly, the electrodes are demoulded from the mold. Thirteen different concentrations 0.1wt%, 0.25wt%, 0.5wt%, 0.75wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt%, 3wt%, 3.5wt%, 4wt%, 4.5wt%, and 5wt%, for both the groups were fabricated. Physical characterizations like TEM, SEM, FITR, and Raman Spectroscopy were performed. Tests like capacitance, resistance, and impedance were also carried out. The ECG was monitored using the system developed in Chapter 3. The ECG results were compared with the Ag/AgCl electrodes.

Chapter-7: The seventh chapter consists of the overall conclusion and future scope of the presented work. In this work, novel and various kinds of ECG electrodes were fabricated using various permutations and combinations of the nanomaterials. Also, an IoT system was deployed and used for continuous ECG monitoring.

CHAPTER 2

Electrocardiogram

2.1 Introduction

Recent advancements in technology in medical sciences have improved everyone's life. The use of wireless technologies in the medical field can facilitate swift and essential healthcare services. Currently, heart diseases have taken a toll and caused many deaths. Heart diseases are caused due to abnormalities in the heart. Therefore, early detection of heart abnormalities can add a number of years to people's life. The most efficient diagnostic tool to measure the heart's electrical activity is ECG [62]. ECG provides precise diagnostic data regarding heart-functioning and cardiovascular health and system. For a person living in a rural area or a place far from the doctor; new and advanced types of equipment and devices are required so that the information reaches the medical expert and appropriate treatment or suggestion can be provided to the person. This is only possible with the help of mobile healthcare systems [63, 64]. The remote healthcare model solves the problem of immediate help from the medical expert but also save traveling cost and time; delay in getting medical attention [64, 65], hence improving healthcare quality and patient safety.

2.2 Cardiovascular Diseases (CVDs)

CVDs are one of the major non-communicable diseases which are responsible for approximately 32% of deaths from non-communicable diseases. According to a report from World Health Organisation (WHO) approximately 17.9 million people died from CVD in 2019. Of the total number, 85% of deaths were caused due to heart attack and stroke under the age of 70. Therefore, it becomes necessary to detect CVDs at a very early age [66]. Also, it is anticipated that by 2030, 23.6 million people will die from CVDs. One of the major reasons is the doctor-to-patient ratio in low-to-middle income countries. For instance, in India, approximately 60 doctors are available to 1 lakh people, as compared to 250 doctors to 1 lakh people in developed countries [67, 68]. Sometimes the symptoms of CVDs are difficult to detect like pain in the arm, the people can confuse with gastric issues. Bold symptoms like chest pain, shortness of breath, and fatigue are also important symptoms to be taken into consideration. Various CVDs are discussed as follows [68]:

1. **Coronary heart/artery disease:** This type of CVD occurs due to plaque in the coronary blood vessels. Plaque consists of cholesterol, fatty acids, and fibrin. This blocks the supply of oxygen and blood to the heart.

2. **Angina pectoris:** It is a chest pain caused by an insufficient supply of blood flow to the heart, it can also be considered the symptom of coronary heart/artery disease.

3. **Congenital Heart Disease:** Commonly termed as 'heart failure', caused when the heart fails to pump a sufficient amount of blood to various parts of the body.

4. **Arrhythmias:** Defined as an irregular heartbeat that can be slow or fast. The normal heartbeat is approximately 72 beats per minute (bpm) [62]. A very slow heartbeat is termed 'bradycardia' and a fast heartbeat is coined 'tachycardia' [69]. When the heart rhythm is irregular, the heart is not able to pump enough blood to all the body parts, which can damage vital organs like the brain, heart, kidney, lungs, etc. Symptoms include feeling pauses between the heartbeats, weakness, fatigue, sweating, and uncomfortable breathing. Causes of arrhythmia include smoking, excessive alcohol intake, too much intake of caffeine/nicotine, stress, and high blood pressure. Various risk factors include heart attack, heart failure, narrow heart valves, congenital heart defects, diabetes, etc.

i. Types of arrhythmias:

a. Premature beats: This is the most common and harmless type of arrhythmias. The symptoms include skipped beat or a little wavering in the chest. In this case, the person does not require medical attention or treatment for the same.

b. Supraventricular arrhythmias: These are tachycardias which are fast heart rates that begin in the atrioventricular (AV) node or the atria.

c. **Ventricular arrhythmias:** As the name suggests it starts in the ventricles and requires medical attention. It includes ventricular tachycardia and ventricular fibrillation.

d. **Bradyarrhythmia:** This type of arrhythmia does not supply enough blood flow to the brain, which causes the heart to beat slower. This state causes unconsciousness.

e. **Cardiomyopathy:** This occurs due to inadequate pumping of the heart because of structural changes in the heart muscle or the weakening of heart muscles.

f. **Ischemic Heart Diseases:** This occurs due to damage in the major blood vessels in the heart and causes heart attacks. It is a condition in which a shortage of oxygen and other nutrients can affect organs like the heart (Cardiac ischemia), brain (cerebral ischemia), limbs (Critical limb ischemia) or intestines (Intestinal or Bowel ischemia)

Various CVDs have been discussed, to detect abnormalities in the heart and to avoid such brutal effects, ECG is the best method to measure the electrical activities of the heart.

2.3 Electrocardiogram (ECG) and Types of ECG Systems

ECG is a pain-free, non-invasive technique to support the diagnosis of various common heart issues. A healthcare provider might use an ECG to detect or determine:

i. Arrhythmias
ii. Coronary artery diseases- are causing chest pain/ heart attack
iii. Any previous heart ailment
iv. If on pace-makers, then how well they are working

An ECG is required if the following symptoms are observed [70]:

i. Chest-pain
ii. Giddiness

iii. Light-headedness
iv. Palpitations
v. Furious pulse-rate
vi. Breathlessness
vii. Fatigue

If these symptoms appear and disappear, there is a chance that the issue may not be detected at that particular instant. Therefore, the medical expert might suggest remote and continuous ECG monitoring. The devices may include the following:

i. **Holter monitoring:** A Holter monitor (Figure 2.1) records the heart's activities constantly for 2458 - 48 hours, while performing everyday chores or while sleeping. It can be used when the heartbeat is either too fast or too slow or the person misses the heartbeat. This monitor can be worn if the person is detected with a heart condition. It is like a small box with wires and sensors attached to the person's chest [71].

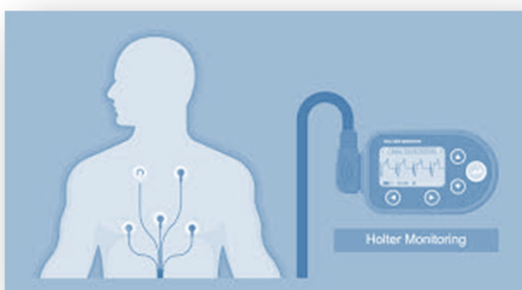


Figure 2.1: Holter monitor

ii. **Event monitor:** It is a portable device as shown in Figure 2.2, very similar to a Holter monitor, but does not provide a continuous recording process, instead it records for only certain times and a few minutes only. It can be worn for approximately 30-days, which is longer than a Holter monitor. Some devices automatically start recording when an irregular heart activity is detected (memory looping monitor), and devices need to be switched on (symptom event monitor). One has to stay away from electronic devices (approx. 6 inches) while the monitor is recording, as the electronic signals interfere with the ECG signals [72].

Figure 2.2: Event Monitor

An ECG waveform can provide the following details about the heart's electrical activities to the medical expert:

(a) **Heart rate:** Generally, it can be measured by monitoring the pulse rate. An ECG can be beneficial for the identification of an extremely slow heart rate (bradycardia), and extremely fast heart rate (tachycardia).

(b) **Heart-rhythm:** An ECG can help in the detection of irregular heartbeats known as arrhythmias.

(c) **Heart attack:** An ECG can demonstrate evidence of a prior heart attack. The ECG patterns may also detect which portion of the heart is damaged and also the extent to which the damage has been done.

(d) **Blood supply and oxygen supply to the heart:** An ECG measured while a person has symptoms may determine the cause of the chest pain due to reduced blood flow.

(e) **Heart structure changes:** An ECG can offer signs of an enlarged heart, heart defects, and other heart problems.

The above section discussed ECG, the types of devices that can be used for ECG detection, and abundant information about the heart that the ECG offers. In the consequent section, types of ECG have been discussed.

2.3.1 Chest ECG

ECG is an unobtrusive surficial measurement of the electrical activity of the heart (Figure 2.3). Augustus Waller devised the first-ever human ECG using a capillary electrometer in 1887 [73]. In 1902, Willem Einthoven testified the galvanometric measurements of the ECG from the limbs (arms and legs) which are immersed in the saline solution(s), and this marked the beginning of the ECG as a "clinical tool" [74]. Contemporary measurements of the ECG using gel-based electrodes are typically derived from the AgCl because of their low-half-cell potentials. The ECG waveforms are generally of the amplitude of 1mV and are improved using low noise instrumentation amplifiers.

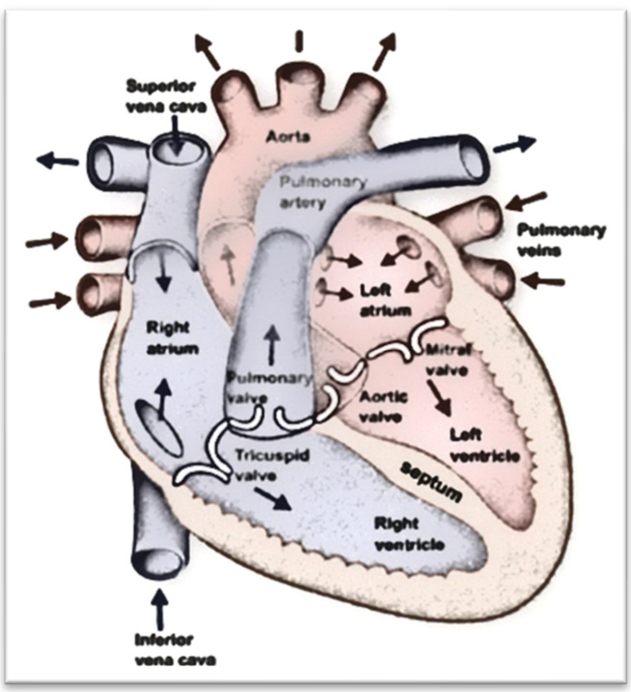


Figure 2.3: Anatomy of the Heart

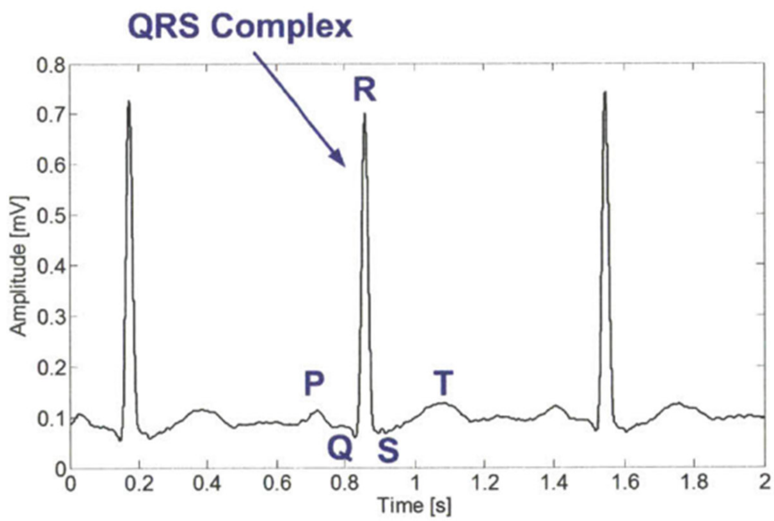


Figure 2.4: A standard ECG waveform (lead II)

In Figure 2.4, the ‘P’-wave depicts the depolarization of the atria, the ‘QRS-complex’ depicts the depolarization of ventricles, and the ‘T’- wave depicts the re-polarization of the ventricles. Since the highest signal quality is required, all such ECG leads and monitors are placed on the chest. The placement of the leads is shown in Figure 2.5. Every differential vector evaluation of the ECG is coined as ‘lead’. The standard clinical ECG monitoring system utilizes a 12-lead system as depicted in Figure 2.5. The 6-leads which lie in the plane of the body are called ‘frontal leads’. The remaining six leads which are from V1-V6 are ‘precordial’ leads that are perpendicular to the body.

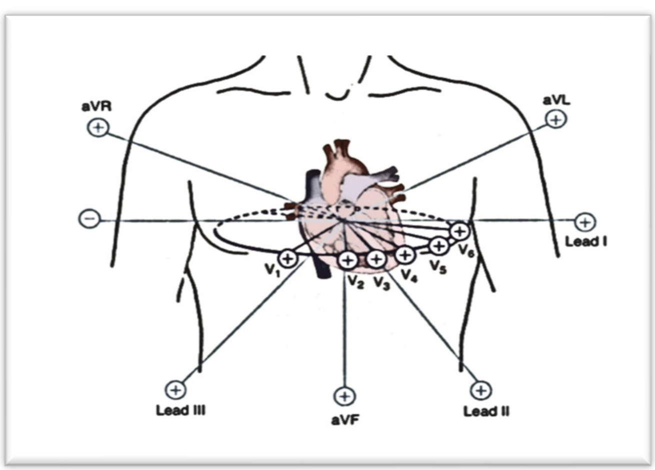


Figure 2.5: The standard 12-lead ECG

For monitoring and diagnosis objectives, it is important to monitor and track long-term ECG throughout the day for the patient and his/her daily events.

2.3.2 The Head ECG

As the human body is a conductive medium, the ECG signals can be obtained from any part of the body but will be distorted and attenuated. First electrode is located in the mastoidal region, and the 2nd electrode is positioned on the upper middle part of the neck. The mastoid's bony assembly and the neck's flat surface allow a very firm electrode contact. Together, the 2-electrode system works as a single-lead ECG system. Generally, a 3rd Driven Right Leg (DRL) electrode is necessary for the Common-Mode-Feedback (CMFB). The DRL electrode intends to condense the common-mode power-line interference and regulate the circuit's DC-potential to the body's potential. Since a limited skin area is near the ear, the DRL electrode is excluded [75].

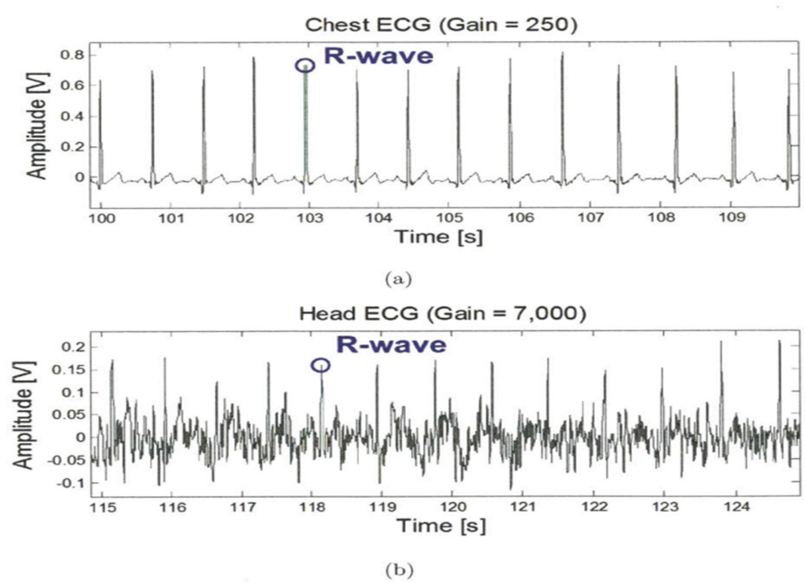


Figure 2.6: (a) Chest ECG, (b) Head ECG

Figure 2.6 (a), represents chest ECG which has a magnitude two times as large as the ECG signals from the head in the range of 30_pV_p - 40_pV_p as shown in In Figure 2.6 (b). The diminution is because of the ECG field lines at the head, which produce a minute potential difference when projected on the mastoid lead. Due to this attenuation, the head-ECG has a reduced Signal-to-Noise Ratio (SNR) and only R-waves are proximately evident. Therefore, the best way to measure ECG is through chest leads. Let's discuss the various ECG lead placement configurations [75].

2.4 ECG Lead Configuration Systems

The surface electrodes help in measuring the electrical activities of the heart [76]. The electrode potential is the reason for heart conduction to the body surface. Standardized and referenced electrode placements and positions are used to record and monitor the ECG. The following are defined as follows:

1. Bipolar limb leads (Standard leads)
2. Augmented unipolar limb leads
3. Chest leads

1. Bipolar Limb Leads - Standard Lead-I, Standard Lead-II, and Standard Lead-III

Bipolar Limb leads are also referred to as the 'Einthoven' lead system. 2-electrodes record and monitor the ECG signals. It can be seen in Figure 2.7, that four body locations of the body namely Right Leg (RL), Right Arm (RA), Left Arm (LA), and Left Leg (LL) potentials are recorded.

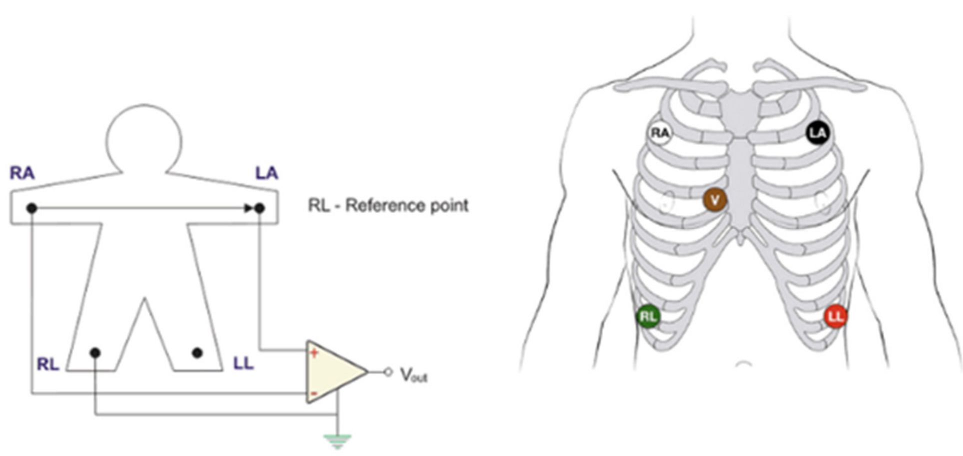


Figure 2.7: ECG lead placement (Bipolar Limb Leads)

The final ECG result is the differential value between these two values. The right leg is considered the ground reference electrode.

Lead-I: The voltage drop is across the left arm and right arm**,** referred to as ‘V-I’.

Lead II: The voltage drop is across the left leg to the right arm, referred to as ‘V-II’.

Lead III: Voltage drop occurs from left leg to left arm, referred to as ‘V-III’.

Einthoven Triangle

It is a closed pathway devised between the left arm, right arm, and left leg. Einthoven described that the cardiac electric vector is 2-D along the frontal plane of the body as shown in Figure 2.8. Along with the projections of the triangle, vector sums on the 3-sides of the triangle are ‘0’. Also, by Kirchhoff’s law, the amplitude of the R-wave along lead-III is equal to the sum of the amplitude of the R-wave along lead-I and lead-II [77].

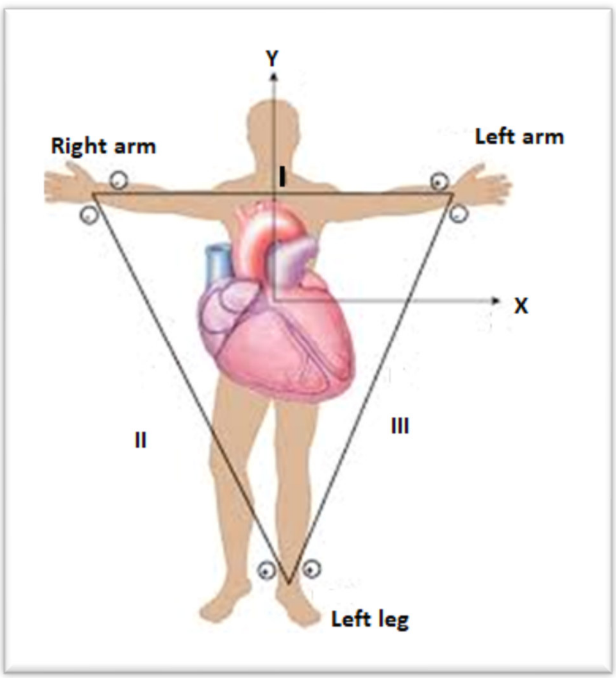


Figure 2.8: Einthoven Triangle

2. **Augmented Unipolar Limb Leads**

Unipolar Limb Leads were introduced by Wilson. In this system, ECG is recorded between a single exploratory electrode and central terminal. As the name suggests, 'Central terminal' is associated with the center of the human body. Two large and equal resistors are employed. The limb lead pair are connected to the resistors. This assembly and connection formed at the center joint of this resistive network establish the central network. The residual segment of the limb electrode formed the exploratory electrode. In this lead system, a minute surge in the ECG voltage was observed. Three kinds of connections are utilized [78].

i. **aVR Lead**

2- resistors are connected to the left leg and the left arm. The middle (center) of the resistor point is connected with the negative terminal. The right arm is connected to the positive terminal of the amplifier. The right leg is considered the reference terminal.

ii. **aVL Lead**

2- resistors are connected to the right arm and left leg. The middle (center) of the resistor point is connected with the negative terminal. The left arm is connected to the positive terminal of the amplifier. The right leg is considered the reference terminal.

iii. **aVF Lead**

2- resistors are connected to the left arm and right arm. The middle (center) of the resistor point is connected with the negative terminal. The left leg is connected to the positive terminal of the amplifier. The right leg is considered the reference terminal [78].

3. **Chest leads**

V-1 – 4^{th} inter-costal space of the right-sternal margin

V-2 – 4^{th} inter-costal space of the left-sternal margin

V-3 – Mid-point between V-2 and V-4

V- 4 – 5th inter-costal space at the midclavicular line

V-5 – Similar to the V-4 position but placed on the auxiliary line

V-6 – Similar to the V-4 position but placed on the mid-auxiliary line [78]

2.5 Basic ECG Patterns

As depicted in Figure 2.9, the electrical activities of the heart are measured and monitored by employing the electrodes on various body surfaces of the person. The electrical activities measured by the electrodes are spread across the atria to the ventricles. The abnormal electrical activities determine vulnerable heart conditions. Various ECG segments and waves are described below:

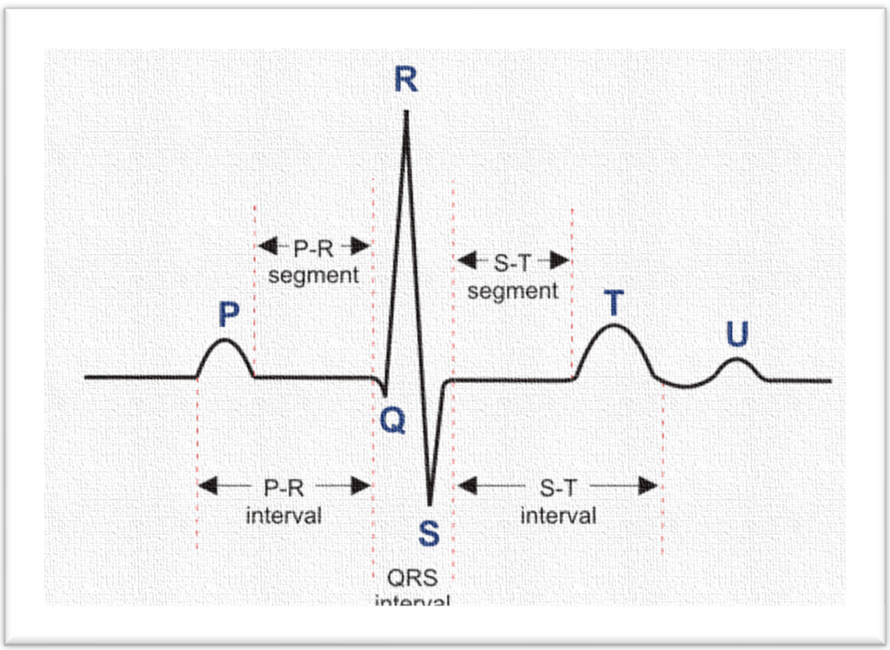


Figure 2.9: Basic ECG Pattern

i. **P -wave:** It arrives first, and signifies de-polarization of the atria. During the P-wave, the electrical impulse begins with the sinoatrial node to the atrioventricular node which spreads across both the atria [62].

ii. **QRS-complex:** It signifies de-polarization of the ventricles, and is considered the strongest wave in the ECG waveform. The QRS complex comprises of three peaks: ‘Q’ and ‘S’ are the negative peaks, and ‘R’ is the positive peak [62].

iii. **PR-interval**: It is addressed as the delay between the P wave and QRS complex [79].

iv. **T-wave:** It signifies the re-polarization of the ventricles, and is a positive deflection just after the QRS complex [80].

v. **ST-segment:** It occurs between the depolarization and repolarization of the ventricles, and the time duration is between the S-wave and the outset of the T-wave [79].

vi. **U-wave:** It is an extremely minute deflection right after the T-wave and signifies the re-polarization of the Purkinje fibers [79].

Usually, one cycle of the ECG signal comprises primarily four waves which are the P-wave, QRS complex, T-wave, and U-wave (only observable only occasionally). The baseline or the isoelectric line is measured as the distance of the tracing following the P-wave and the PR segment after the T-wave. Table 2.1 provides the ECG waves in a nutshell.

Table 2.1: Various Waves, Complexes, and Segments of ECG waves

Waves and Complexes [81]			
S. No	**Type**	**Description**	**Range/Healthy wave (ECG Interpretation)**
1.	P wave	It represents atrial de-polarization (left and right).	Amplitude ≤ 3 mm; Upright (positive) and uniform.
2.	T wave	It indicates ventricular re-polarization (left).	The standard T-wave is generally in the same direction as the QRS except in the right pre-cordial leads; Duration < 0.2 seconds.
3.	Q wave	It is the first downward deflection after the P wave.	Duration < 0.04 seconds; Amplitude < 2.5mm.
4.	R wave	It is the initial positive deflection.	It has an amplitude lower than the S wave.
5.	S wave	It is the negative deflection following the R-wave.	In the normal ECG, there is a large S-wave in V-1 (precordial or chest leads) that gradually becomes smaller, to the point that almost no S-wave is present in V-6 (precordial or chest leads)
6.	QRS	It represents the	The normal QRS complex is very variable in the

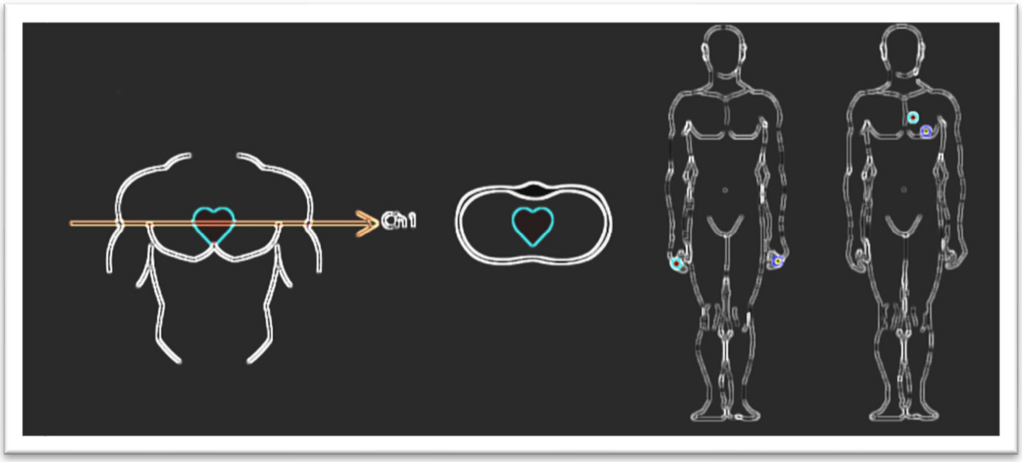
	Complex	ventricular de-polarization (left and right).	frontal leads (Augmented unipolar limb leads) and quite uniform in the horizontal leads (Unipolar chest leads); Duration: 0.06ms-0.12ms.
Intervals and Segments [81]			
S. No	**Type**	**Description**	**Range/Healthy wave (ECG Interpretation)**
1.	PR Interval	This interval starts from the P wave to the beginning of the QRS-complex.	Duration: 0.1s - 0.14s.
2.	PR Segment	It leads starts from the end of the P-wave till the start of the QRS complex.	A normal PR segment is present in the ECG waveform but is invisible in case of any atrial injury: Duration 50ms-120ms.
3.	J Joint	The junction between the QRS complex and ST-segment.	Elevation or the depression of the J-joint is seen with the various causes of ST-segment abnormalities.
4.	QT interval	Begins with the QRS-complex till the end of the T-wave.	Duration: 300ms-430ms
5.	QRS Interval	Begins with the QRS-complex till the end of the QRS wave.	Duration $\leq$ 0.12 s
6.	ST segment	Begins with the end of the QRS complex till the start of the T-wave.	Duration: 80ms-120ms.

2.6 ECG Lead Placement

Leads are different vectors that give a different appearance to the electrical activities of the heart. The electrical potentials are attained by placing electrodes at different positions on the body surface and producing different ECG vectors. Different types of lead placement sections are discussed as follows [82]:

2.6.1 1-Lead ECG

These types of lead placement are very common to measure the ECG. It is depicted in Figure 2.10. The monitoring can range from a few seconds to 14 days. The medical significance of a 1-lead ECG signal is restricted.



1 vertical and 0 horizontal visual axes

Figure 2.10: 1-Lead ECG System

The Diagnostic capabilities include basic heart monitoring for research/educational purposes, to check the effect of exercise. Ischemia cannot be detected, and the detection of arrhythmias is very limited. Also, atrial fibrillation/ excessive ventricular beats cannot be recorded.

2.6.2 3-Lead ECG

3-lead ECG systems are mostly used for recording and analyzing 24-hour readings that are often used for the analysis of heart issues and are considered a long-term measurement ECG system. In a 3-lead configuration, the placement is as follows: one electrode on the right arm, another on the left arm, and the 3rd electrode on the right ankle as depicted in Figure 2.11 [83].

3 vertical and 0 horizontal visual axes

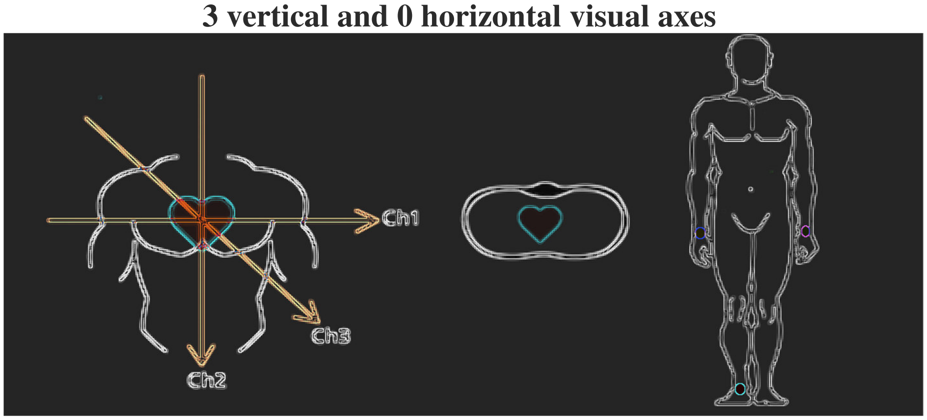


Figure 2.11: 3-Lead ECG System

The Diagnostic capabilities include basic heart monitoring to check the effect of exercise. Ischemia cannot be detected, and the detection of arrhythmias is very limited. However, it can detect atrial fibrillation in general practice.

2.6.3 5-Lead ECG

In this configuration, all four limb leads are placed on the chest, and a supplementary electrode is placed in one of the chest lead sites. 5-lead ECG system can record any of the 6 limb leads + 1 pre-cordial-lead. V1 is insensitive for detecting severe myocardial ischemia. 5-lead ECG system as depicted in Figure 2.12; more than one precordial lead cannot be displayed at the same time. This system involves the electrode placement of 3 electrodes as in the 3-lead system and adding the 4th electrode next to the right side of the 4th inter-coastal region, and the 5th electrode on the person's lower right abdomen.

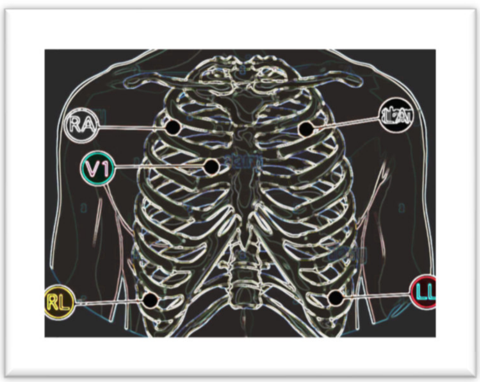


Figure 2.12: 5-Lead ECG System

All the limb lead ECGs can be attained through a 5-Lead ECG system, although only one chest lead is used at a particular time. 5-Lead ECG configuration can detect the rhythm abnormalities, and the ST segment deviations appropriately.

2.6.4 6-Lead ECG

The 6-Lead ECG system comprises of 6-electrodes which include 4-limb and 2-chest electrodes. It helps in monitoring Bipolar-leads and augmented leads. C_a and C_b should be positioned at two places between C1 and C6. The subsequent permutations and combinations that can be applied are C1:C3, C2:C5, C3:C5, C1:C4, C2:C4, C3:C6, C1:C5 as shown in Figure 2.13. It can detect atrial fibrillation/normal heart rhythm, and cardiac arrhythmias [84].

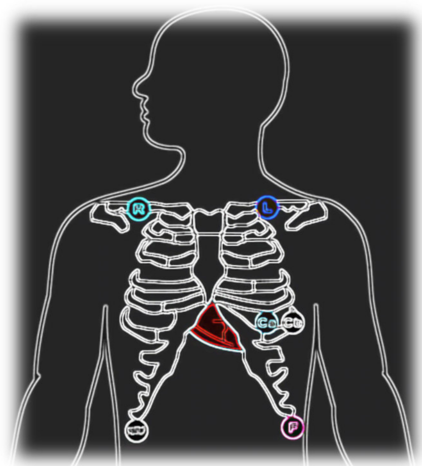


Figure 2.13: 6-Lead ECG System

2.6.5 12-Lead ECG

The 12-lead ECG system entails the positioning of 12 electrodes on the person's body. 4-electrodes, on the person's limbs, including the right-arm electrode (R-lead), left-arm electrode (L-lead), right leg electrode (N-lead), and the left leg electrode (F-lead) as shown in Figure 2.14. 6 chest electrodes (C1- C6) are positioned on the person's thorax at several body parts. This system comprises of 3-augmented-leads: a-VR, a-VL, and a-VF, and 3 bipolar leads. Lead-I, Lead- II, Lead- III, and 6-unipolar precordial leads to complete the 12-lead ECG system [85].

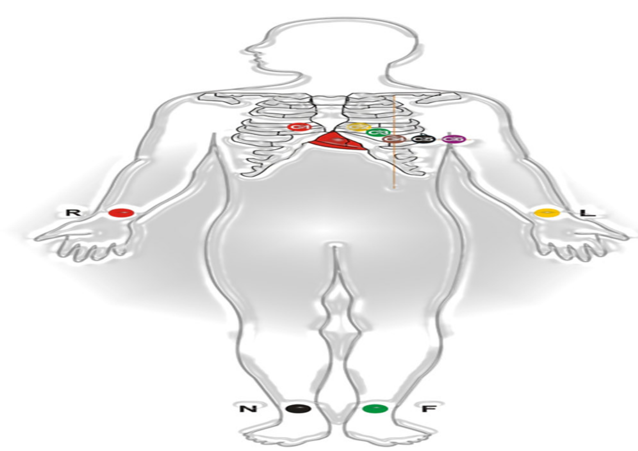


Figure 2.14: 12-Lead ECG System

The ECG placement systems have to be discussed, which is very important before one knows how to measure the ECG. Following this, another important parameter is the selection of electrodes, since different types of electrodes can be suited to different ECG lead systems. Therefore, the next system discusses the various types of electrodes which can be used for ECG monitoring.

2.7 TYPES OF ECG ELECTRODES: A WALK-THROUGH

The conventional method of recording the electrical activities of the heart is ECG. Typically, to measure ECG electrodes/sensors are attached to various parts of the body, like limbs and the chest. Further, these sensors measure the potential differences throughout each cardiac cycle. Then with these potential differences, an ECG waveform with P, QRS, and T components can be seen [59]. Therefore, to attain the waveform, a wide variety of electrodes can be employed. Hence, these categories are discussed below.

2.7.1 Pre-gelled Electrodes

The most commonly worn electrode for ECG recording is the conventional Ag/AgCl electrodes as shown in Figure 2.15. These electrodes already have a conductive paste on the electrode. This gel is used for providing and improving conductivity between the skin and electrode. Also, the gel contributes towards dispensing adhesion between the skin and electrode and reduces the skin-to-electrode impedance. These electrodes are lightweight and can be worn comfortably by the patient. However, as far as long-term ECG monitoring is taken into consideration, these electrodes cannot be used. The key reason being the gel on the electrode dehydrates as time elopes. Therefore, the ECG signal quality is degraded. Also, these electrodes are not reusable. After a few attachments and detachments, the adhesive properties of these electrodes tend to decrease. Hence, reducing the grip between the skin surface and electrode [86]. Also, utilizing these electrodes can incite skin irritation due to the existence of harmful compounds or sensitizing compounds with really high concentrations. Also, irritation can be caused while removing the electrode from the skin surface [87]. It was also reported that recurrently using these electrodes can cause allergy to dermatitis [88]. These gelled electrodes also form an artifact, which is termed charge sensitivity [86].

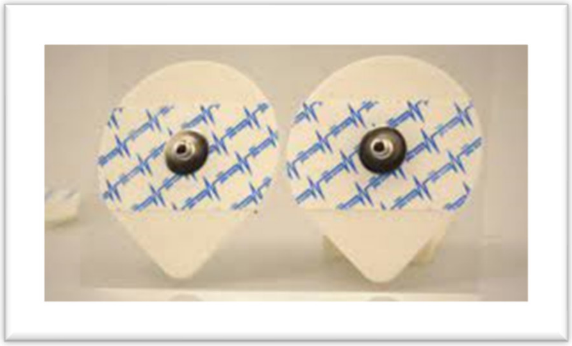


Figure 2.15: Pre-gelled Electrodes

2.7.2 Bulb Electrodes/Clamp Electrodes

These electrodes are typically used in hospitals or medical centers. The bulb of the electrodes is usually made with rubber, and the metal part at the bottom can be of silver, brass, or silicon. The metal part is attached to the conduction interface between the skin surface and the electrode. Similarly, the clamp or clip ECG electrodes have a metal part on one side of the clip, which is used for providing the interface between the skin and electrode as depicted in Figure 2.16. The procedure for recording the ECG signals starts with shaving the hair (not in all cases) before applying these electrodes to the skin surface. This induces sensitization as the skin surface was abraded [89]. In various works, it was observed that as many electrodes are placed close to each other, the conductive gel applied might create a conductive path between the neighboring electrodes. This could lead to a short-circuit of the electrodes [90].

Additionally, the machines or monitors on which the ECG waveform can be seen are bulky, hence not supporting remote ECG monitoring. The ECG test in the medical center is also relatively expensive. These electrodes also diminish the possibility of long-term ECG measurement since the person cannot stay in the hospital/medical center for long durations. It was also observed that the gel applied could cause skin allergies and irritation [91]. The use of these electrodes also rules out the possibility of persistent and continuous ECG monitoring as the gel dries up soon and has to be applied over and over again. This also degrades the signal quality since the electrode loses contact with the skin due to degraded adhesivity.

Several issues regarding pre-gelled and bulb/clamp electrodes have been discussed. Hence, to overcome these issues, dry or gel-free electrodes have been explored [92]. Also, there has been an upsurge in interest in wearable and flexible ECG sensors due to bio-compatibility, convenience, and comfort [50, 58, 93]. This concept facilitates the self-help approach to managing health. It has led to financial independence, along with reduced time and infrastructure constraints. Also, it enabled continuous communication with the medical fraternity. Hence, this leads to remote monitoring of real-time ECG signals.

Figure 2.16: Bulb/Clamp Electrodes

2.7.3 Dry electrodes

To surpass the limitations of pre-gelled/bulb electrodes, dry electrodes were brought into the picture. These sensors do not require any involvement of conductive gel between the electrode and the skin surface. To recap, the gel introduces smearing issues and cross-talk problems. Another issue is that the gel dries over time and has to be applied so that the adhesion is proper, and the signal quality is maintained [94]. Dry electrodes overcome these disadvantages. Hence, these electrodes are an excellent candidate for continuous and long-time ECG monitoring systems. Also, these electrodes possess excellent stretchability, conformality, flexibility, and allow remote ECG monitoring services. Therefore, various types of dry electrodes are elaborated as follows and depicted in Figure 2.17.

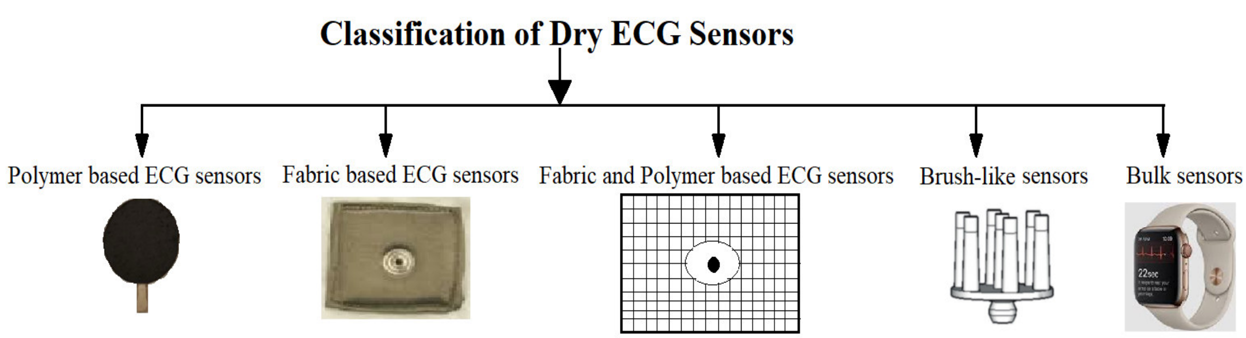


Figure 2.17: Types of Dry Electrodes

a. Polymer-based ECG sensors

Here, the polymers are incorporated with the materials. Polymer-based sensors have captivated much attention for fabricating sensors because it makes the electrodes flexible, conformable, and cost-effective [95]. These sensors are resistant to signal deterioration and skin allergy [96]. Also, they possess exceptional chemical and physical characteristics in addition to the substantial potential for fabricating lightweight, economical, and large-scale biosensors [97]. Also, surface modification is easy, and due to large surface areas, these are apt for fabricating electronic sensors [98].

b. Fabric-based ECG sensors

The ECG sensors are fabricated using various fabrics such as polyester, gauze, nylon, cotton, and so on. The sensors are fabricated and have seized the attention of researchers. The conducting materials can be either embedded into the fabric or the electrodes can be sewed into wearables like a shirt or vest, belt, etc. Also, the fabric or yarn can be dipped into the conductive solution and further dried up before use [22]. The advantage of using these sensors is comfortability, as it allows the effortless wearing of the garment. It encourages long-term and secures ECG monitoring. These sensors easily conform with the person's body type.

c. Fabric and Polymer-based ECG sensors

A combination of both fabric and polymer for developing the ECG sensors is extremely riveting. This can be achieved by forming a mixture of the conducting material and polymer, and applying the paste to the fabric. It incorporates both the advantages of polymers and fabrics. The polymer makes the sensor flexible and stretchable and including fabrics makes the sensor comfortable and easy to wear.

d. Pin-shaped sensors

These sensors are fabricated with the help of macroscale pins. The brush-like structure holds the skin firmly in addition to up-surging the contact area between the sensor and the skin surface. Also, these can be grown in the form of forests. These forests can be grown on the substrate. One of the significant advantages of using this sensor is the excellent grip on the skin and exceptional electrode-to-skin contact, even on the hairy skin surface. These sensors can be termed capacitive electrodes. Since these do not require a conductive gel to be applied before using these sensors [22].

e. Bulk Sensors

These sensors include devices such as a wristband, watches, and earphones for monitoring one's ECG. These can also be termed as smart-devices. Bulk sensors are fabricated using flexible materials and substrates so that they can easily conform to any kind of skin surface like a wrist, ear, or even arms. This enhances skin contact, hence improving the quality of the ECG signal. Also, the devices are sturdy and stable enough so that they can be worn persistently without any major issues.

2.8 Conclusion

This chapter discussed various CVDs and their effects on the world population. To reduce the number of deaths, ECG helps to discover the abnormalities. In this work, a 3-lead system has been utilized to measure the ECG. A 3-lead system is the best configuration for this work because it provides good results and helps in making the system portable and excellent for long-term and continuous ECG monitoring. To achieve the portability of the ECG system the materials used for fabrication should be able to provide flexibility and elasticity to the electrodes.

CHAPTER 3

Flexible Electronics

3.1 INTRODUCTION

This research focuses on developing reusable, comfortable, and lightweight ECG electrodes for biomedical sensing. These biomedical sensors should be flexible, conform to human skin, environment friendly, easy to use, and provide highly accurate results of the ECG signal.

Flexible electronics can be defined as a technology where electronic circuits can be embedded on flexible substrates like films, polyimide, polymers, and polyester. In the recent past, an evolving segment of flexible electronics has outdone their rigid substrate analogous counterparts because of their exceptional characteristics like light-weight, proportionally shaped, more durable, less expensive, and large-area manufacturing capabilities [99]. Numerous demonstrations of flexible electronics promise the availability and accessibility of portable, compact, light-weight, robust, and pocket-friendly electronics in near future. The speedy progression of flexible electronics is driven by the budding facilitation of nanomaterials including a wide range of applications in displays [100], RFID tags [18], thin-film transistors [5], sensors [101], antennas [1], photovoltaics [2], light-emitting diodes [102], electronic paper [103], and numerous potential and probable applications. Nanotechnology provides outstanding features like tunability in performance, low processing temperature, and solution processability, which makes it extremely appropriate for flexible electronics processes and manufacturing. Various conductive nanomaterials have been covered in the first chapter. Since each of the carbon-based Nanomaterials possesses different properties and characteristics, different nanomaterials are suitable for different applications. The properties required for making conductive sensors are conductivity, flexibility, and robustness. Therefore, the materials offer the characteristics close to the requirements for this study are discussed as follows:

3.2 Conductive nanomaterials for fabricating ECG Electrodes

A. Functionalized CNTs

1. Cost-effective when compared to gold and silver nanoparticles
2. Better-dispersing capabilities than unfunctionalized counterparts
3. Better intermolecular bonding between the polymer and CNTs
4. Better conductivity because of the better flow of electrons

B. Graphene

1. Safe for skin
2. Long-term biocompatibility
3. Excellent strength
4. Light-weight

3.3 Other materials for fabricating ECG Electrodes

A. PDMS

1. Biocompatibility
2. Non-toxicity
3. High gas penetrating capacity
4. Flexibility

B. Isopropyl Alcohol (IPA)

1. Both the materials are soluble
2. Solvents like methanol, which are non-polar, worked well for dispersing the CNTs but swelled up the PDMS
3. Bubbles are easily removed from the composites
4. Evaporates almost instantly
5. Non-toxic when compared to other solvents like methanol

The fabricating materials were selected because of their nature, characteristics, and benefits they have to offer. Another important thing in the selection of the materials is the behaviour of the materials with each other. This is a very important aspect while fabricating the sensors because their characteristics might change when they are sonicated or thermally cured. Also, these materials are selected keeping in mind the requirements for the portable ECG monitoring system for continuous and remote ECG signal monitoring. Therefore, in the next segment the system architecture, hardware, and software requirements of the consistent ECG monitoring system have been discussed.

3.4 Internet-of-Things (IoT)

IoT is the inter-networking of physical devices, buildings, and vehicles. In simple words, it is the communication of the devices. These devices are embedded with sensors/actuators, IoT development boards, and wireless communication protocols which enable the device/object for collection and exchange of data. IoT can also be considered where physical attributes and intelligent interfaces are needed to be connected seamlessly. IoT may be considered a hot topic but it is not a new concept. Kevin Ashton started the journey by linking Radio Frequency Identification (RFID) information online. The applications of IoT are numerous including healthcare, smart homes, smart vehicular applications, smart traffic management, smart parking solutions, and smart pollution systems, among other applications. In this segment of the thesis, the importance of IoT is discussed.

3.4.1 IoT for Healthcare Applications

IoT is a consumer-driven technology and its necessity is predominantly visible in the healthcare domain. IoT has brought a lot of differences in the manner in which health is monitored, recorded, and managed, with devices that exchange data. It has created a platform where people or patients are self-supervising and managing their health vitals [104]. Specifically for heart-related issues, smart healthcare is required which measures the ECG signals, and sends the signals to the medical expert, so that the expert can check if any abnormality is present and some suggestion/remedy can be sent to the person/family/care-taker. Many users of remote ECG monitoring include older people [105, 106], people with special needs [107,108], infants [109-111], fetal monitoring [112-116], and people when driving [117-119]. Therefore, keeping all these things in mind, a smart healthcare system based on IoT has been developed, and its system architecture is described below.

3.5 System Architecture

A block diagram is presented in Figure 3.1 which presents an ECG monitoring system based on IoT for continuous monitoring. The system comprises an ECG signal acquisition system in which sensors (electrodes in this case) are attached to the person's body and an ECG acquisition circuit, then an analog-to-digital converter sends the acquired digital ECG signals to the medical expert's smart device. After careful analysis of the signals, the expert can provide his/her diagnosis of the health condition, and/or suggests remedial measures. This block can be explained in four parts, Sensors, Sensor-to-machine communication, Gateway, and Machine-to-machine communication. These topics are discussed in detail.

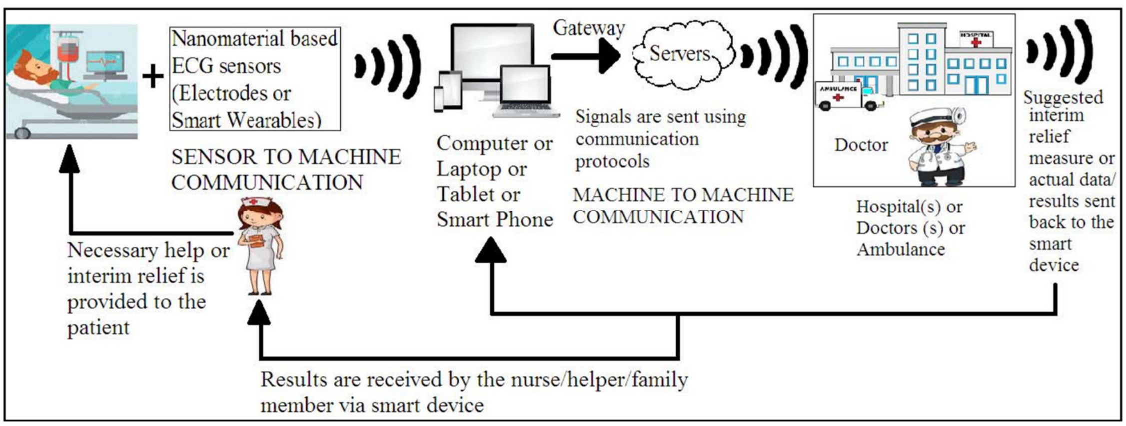


Figure 3.1: System architecture of the IoT-based Healthcare System

A. Sensors

Here the sensors are ECG electrodes, which are attached in a 3-lead configuration, left wrist, right wrist, and right leg. There are two types of electrodes, wet and dry. Figure 3.2 depicts dry electrodes.

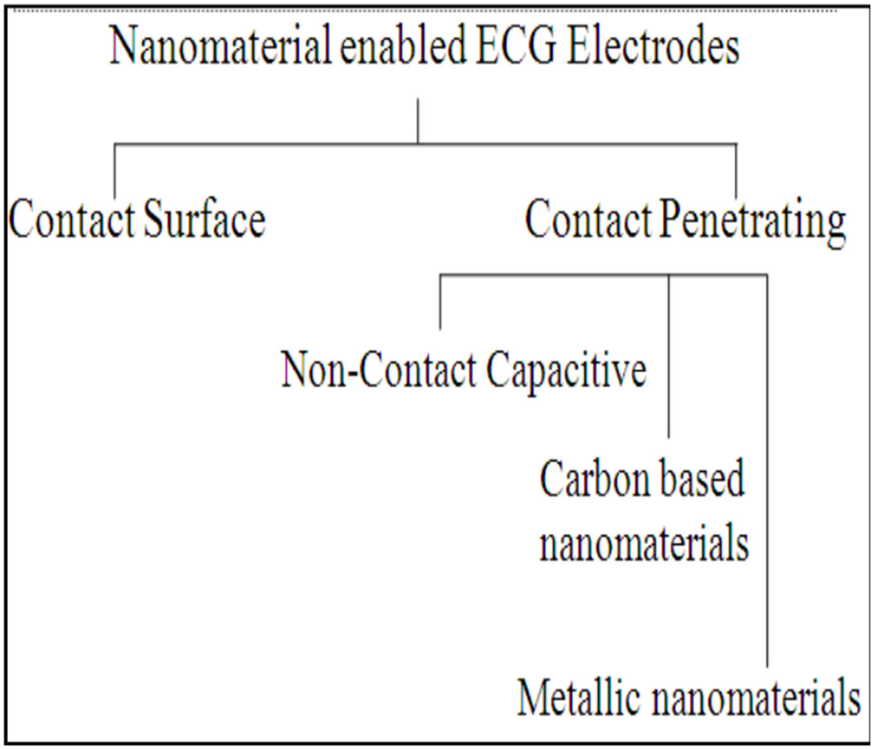


Figure 3.2: Types of Dry Electrodes

Due to the disadvantages of wet electrodes, dry electrodes are used in this system. A comparison is depicted in Table 3.1. Once the electrodes are placed on the body of the person, the signals are to be sent to the device from the sensor. The sensor-to-machine communication is explained in the consequent segment.

Table 3.1: Wet Electrodes vs Dry Electrodes

S.No	Specification	Wet Electrode	Dry Electrode
1.	Use of gel	Yes, makes it inconvenient because it dries with time	No inconvenience because no use of electrolytic gel.
2.	Toxicological concerns due to the gel	Yes	No
3.	Motion Artifacts	Yes	No
4.	Initial Skin Preparation	Clinicians have to apply the gel before starting with the ECG monitoring process.	No need for such engagements.
5.	Continuous Monitoring	Not possible because after a few hours the gel has to be applied again because it dries up, and the whole apparatus is huge, and people cannot carry it with them everywhere.	It is possible, that a dry sensor can be embedded into the wearables.
6.	Long term monitoring	Not possible due to the fact already	Possible.

		mentioned that the gel applied dries up over time.	
7.	Signal Degradation	Signal degrades over time.	No signal degradation.
8.	Remote monitoring	Not feasible	Possible

B. Sensor-to-Machine Communication

When the sensor is fabricated, the ECG signals are transmitted to the machine, which can be any smart device. The sensors/electrodes require connectivity to the 'sensor-gateway' for transmitting the ECG information. The various protocols can include Local Area Networks (LAN) like Ethernet, Wireless Fidelity (Wi-Fi), Personal Area Networks (PAN) like Bluetooth, Near Field Communication (NFC), Zigbee, etc. Some sensors do not require connectivity to the sensor gateways, thus, their connections to the back-end applications/server may be provided through Global System for Mobile Communication (GSM), Long Term Evolution (LTE), and General Packet Radio Service (GPRS) which are Wide Area Network (WAN) protocols. Nowadays, an interesting technology is Wireless Sensor Networks (WSN), in which the sensors require low data and low power for interconnection, which covers large coverage and retains a good battery life [120]. A list of wireless protocols is described in detail in Table 3.2.

Table 3.2 List of Protocols

		Parameters							
S.No	**Name of protocol**	**Frequency**	**Range/ Speed**	**Data Rate**	**Authentication**	**Data Protection**	**Power Consumption**	**Bandwidth**	**Topology**
1.	Wi-Fi	2.4 GHz, 5 GHz	100 m	54 Mbps	WPA2 (802.11i)	32-bit CRC [108]	100–350 mA [109]	22 MHz	Star
2.	Blue-tooth	2.4 GHz	10 m	1 Mbps	Shared secret	16-bit CRC	1–35 mA [109]	1 MHz	Point-to-Point
3.	ZigBee	868/915 MHz; 2.4 GHz	10m-100m	250 Kbps	Chaining Message Authentication Code	16 bit CRC	0.035706 W (24 bytes of data)	0.3/ 0.6/2 MHz,	Star, Tree, Mesh
4.	Ultra Wide Band (UWB)	3.1 – 10.6 GHz	10m	110 Mbps	Chaining Message Authentication Code	32 bit CRC	-41.3 dBm/ MHz [109]	500 MHz-7.5 GHz	Peer-to-peer [109]
5.	NFC	13.56 MHz [110]	5cm (approximatel	106, 212,and	AES Encryption [110]	Soft protection and	2.5 mA [110]	±7kHz-1.8MHz	Peer-to-peer

			y) [110]	424 Kbps [110]		Hard protection			
6.	6LoWPAN (IPv6 over Low-Power Wireless Personal Area Networks)	2400MHz [111]	10m-30m [112]	250 kbit/s	AES-CCM-128 security	Access Control List (ACL) and Secure mode	3dbm	250/40/20 Kbps (Low bandwidth)	Star, Mesh
7.	GPRS	1900, 1800, 900, 850 MHz	15-40 Kbps (speed)	56–114 Kbps	GPRS Encryption Algorithm	-	100-200 mA	296 Kbps	Point-to-Point, Point-to-Multipoint
8.	Ethernet	125 MHz	100 m	10 Mbps	Eap over Lan or EAPOL	-	50 W to 80 W	10 Mbps	Star, Bus
9.	Wireless Local Area Network (WLAN)	2.4- 5 GHz	32m	11-54 Mbps	AAA	WEP	25.5 W(elements of WLAN)	2.4 GHz	IBSS, ESS, BSS
10.	2G (SMS, MMS)	1.44Kbps	Covers a wide distance	50 Kbps	Ciphering key generator	-	400 mW	25Hz	Star, Bus, Ring, Mesh
11.	3G	1.8 GHz	Covers a wide distance	3.1 Mbps	AKA	-	0.5 W	25 Hz	-
12.	4G	2-8 GHz	Covers a wide distance	100-300 Mbps	AKA	-	0.5W	100Hz	-
13.	LTE	1749.9 MHz	350 to 500 km/hr (speed)	100 Mbps	EPS authentication and key agreement (AKA) procedure	-	0.5 W	1.4 MHz	MPLS
15.	GSM	1900, 1800, 900 MHz	35km	9.6 Kbps	Authentication Center	-	1.5 mA	200KHz	Mesh

C. Gateway Network

A reliable and robust wireless channel is required for the transmission of humongous ECG data. A gateway is required to do such a job for machine-to-machine communication. Recently, with the necessity of delivering a huge variety of IoT-based applications, various networks with a wide bandwidth of access protocols and technologies work on heterogeneous platforms. These networks may be private, public, or hybrid for communication purposes [120].

D. Machine to Machine Communication

The gateway acts as an interface for machine-to-machine communication, which means the information reaches from the patient's device to the doctor's device.

The framework has been discussed, and now the implementation of the system has been discussed in the consequent segment of the chapter.

3.6 Smart ECG tracking framework

3.6.1 Hardware Description

A generic architecture of an intelligent ECG tracking framework has been depicted in Figure 3.3. It describes the principal elements and elementary modules of an intelligent ECG monitoring network. Here, the whole system has been bifurcated into four stages. Stage 1 consists of the ECG sensing elements. Stage 2 is for acquiring the ECG through an acquisition circuit and then further processing the received signal. Stage 3 is used for transmitting the processed ECG signal. Once a perfect ECG signal is transmitted, a display device to observe the signal, here stage 4, is required, which helps in the visualization of the signal.

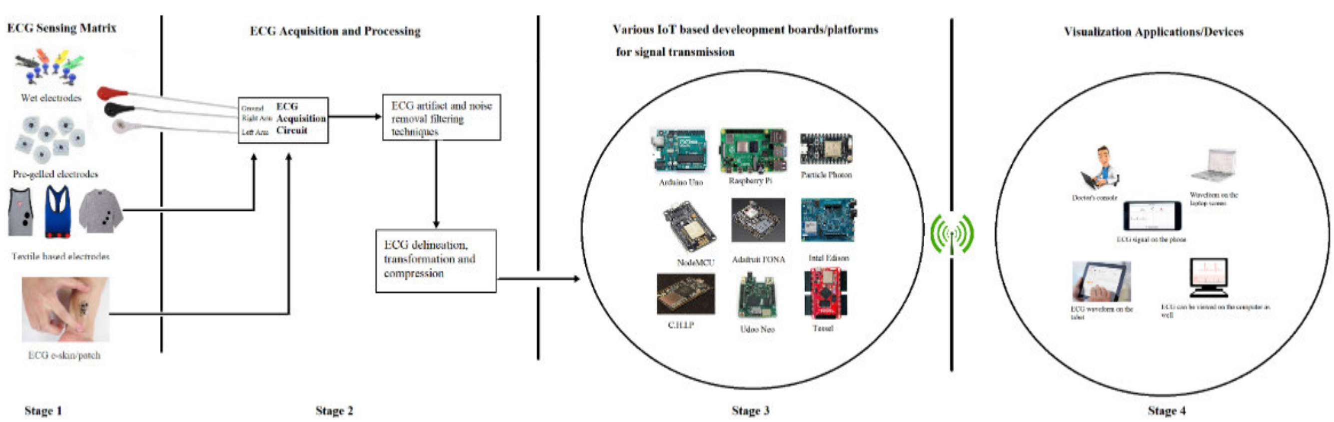


Figure 3.3: Framework of a SMART-ECG monitoring system

3.6.2 ECG Sensing Matrix

Stage 1, which is used for sensing the ECG signal, is the substratum of the entire framework. It holds the responsibility for accumulating ECG signals. The signals can be acquired with the help of wet electrodes [121, 122], which require cables that transfer the signals to the data acquisition circuit. Another variety of sensors are pre-gelled (Ag/AgCl) electrodes [118, 119, 123], and polymer-based electrodes [118, 119, 125, 126, 127, 128, 130-132]. These electrodes require cables as well. Various other sensors can be either textile-based or fabric-based [33, 42], e-skin, or patch type [32, 40, 132-134]. These types of sensors mostly do not require cables for transmission. The sensors can be placed on different spots of the person's body like limbs, chest, wrists/arms for sensing the ECG for wet or polymer-based or pre-gelled electrodes. Another way is by wearing a full-fledged garment like a t-shirt, a vest, or an undershirt, which is embedded into the garment, or the sensors are sewed into the garment. Another method is by placing an adhesive patch on the skin and acquiring the signal. The sensors need to be conductive enough since ECG is a weak signal; therefore, the construction of the sensor/electrode should be done accurately, so that precise ECG results are attained.

3.6.3 ECG Acquisition and Processing

Once the ECG signal has been sensed, it needs to be passed through a data acquisition circuit, so that only the ECG signal is visible and other signals like Electromyogram (EMG) and other noises are filtered out. The processing techniques improve the quality of the raw ECG signal. It also enhances the signal accuracy of prediction. Also, it removes various kinds of noise that may be present with the ECG signal like baseline wander, muscle contractions, powerline interference, motion artifacts, electrode contact noise, variations from breathing and also, and interference from the ECG signals [135-137]. This can also be termed data cleaning since various filters are employed to cut out the external noise. Also, other techniques like delineation, compression, and transformation can be done to enhance further the characteristics of the ECG signal [138, 139].

3.6.4 IoT-based Development Boards

The road to creating products for IoT is paved with the help of development boards/platforms. These smart boards, including SoC-based, microcontroller-based, or single-board computers, have employed economical prototyping and play a crucial role in the development of interconnected entities. The purpose of using these boards is to let people prototype their ideas into innovations. In recent years, it has enriched the market since the developers have leveraged the communication attributes of various and different protocols and systems. Therefore, a combination of microprocessors/microcontrollers and wireless chips creates a ready-to-use programming package. The main idea of enabling IoT-based boards is to contact the outside world. The foremost requirement of developing an IoT-based application is sensor nodes or motes. These devices possess some computational intelligence. These nodes/motes can be programmed and interfaced with the ECG sensors.

The nodes can be interconnected using the internet via any protocol stack [140]. Various development boards can be utilized for interconnectivity like Arduino Uno [141], Raspberry Pi [142], Beagle Bone [143], Intel Galileo [144], IBM Watson [145] so on and so forth.

3.6.5 Visualization: Graphical User Interface

This phase of the monitoring process is accountable for signal visualization, and management. It dispenses simple data access which is present in the storage/cloud. People can view the data on any of the smart devices available. This process permits the users to interact and review the received ECG signals in a real-time scenario or the offline mode [146, 147]. This aspect of the monitoring system helps human beings to analyze the signal and detect abnormalities. There can be two ways where GUI can be implemented, through a browser, desktop applications [148], or mobile apps [149].

3.7 Smart ECG Monitoring Framework: Implementations

The framework of the smart ECG system has been presented in Figure 3.3 and an analogous system has been implemented. The system has been realized using conventional Ag/AgCl electrodes, AD8232, NodeMCU, Arduino IDE, API, and AWS cloud services. Further, a discussion on various components and the flow of working of the system is presented as follows.

3.7.1 ECG Sensing Node

The ECG sensing node(s)/mote(s) is utilized for accumulating the ECG signals from the patient. Here, in this research, conventional Ag/AgCl electrodes from Medico as in Figure 3.4 and fabricated electrodes have been used for acquiring the ECG signal. These electrodes belong to the category of non-polarizable electrodes since they allow the flow of charge to pass through the skin-electrode interface with the least interference [150]. Also, it has a lower electrode-skin interface impedance [151-153].

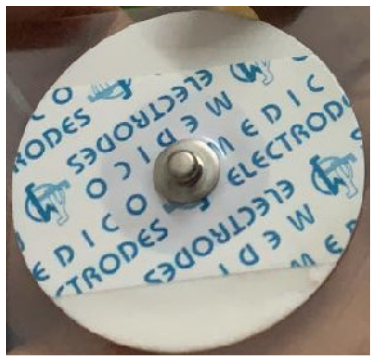


Figure 3.4: Medico Ag/AgCl electrodes

3.7.2 ECG Sensing Module

The sensing element is the base for the sensing node(s)/mote(s). It is an acquisition circuit for the ECG signals. In this work, AD8232 as shown in Figure 3.5 has been utilized to acquire and condition the ECG data. It is an integrated front-end for extracting, amplifying, and filtering the ECG signals in the presence of noise. The noise can be due to various factors like motion, muscle contraction, breathing, and so on. It comprises of right leg drive amplifier, an instrumentation amplifier, and a mid-supply reference buffer. It also has an Analog-to-Digital Converter (ADC) or a micro-controller for attaining the output signal. The dimensions of the device are 4mm x 4mm [154].

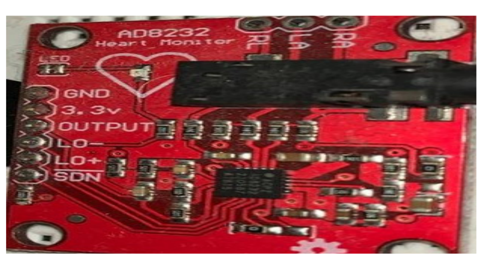


Figure 3.5: AD8232 ECG acquisition board

3.7.3 Controller Module

The controller module is employed to process the accumulated ECG data. In this work, NodeMCU has been employed for this purpose. It is an open-source, programmable, simple, low-cost, interactive, and Wi-Fi-enabled development board as seen in Figure 3.6. Its hardware design is open to building, editing, and modifying IoT-based applications. It consists of an ESP8266 Wi-Fi-enabled chip. It has Arduino-like Analog and Digital pins on its board. It also supports SPI, UART, and I2C serial communication protocols. Applications can be built using Arduino IDE.

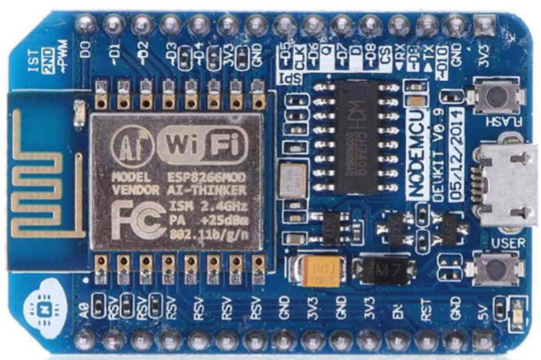


Figure 3.6: NodeMCU Development Board

3.7.4 Power Module

It provides a reliable power source for all the modules of the system. Here, a USB cable was used to supply power to the NodeMCU and AD8232. These both require a 3.3V power supply to function.

3.7.5 Application Program Interface (API)

An API was developed and framed to build context-based applications, which are used to interact with the physical world. It is used to connect the ‘things’ to the ‘internet’. Therefore, it allows interaction between the IoT device, the internet, and all other elements which are present within the network. Here, Node.js [155] is used as a programming language for developing the API. It is an open-source a cross-platform. JavaScript runtime environment executes JavaScripts code to the external web browser.

3.7.6 IoT Cloud Platform

After, the system has acquired the data via the sensing module. The IoT cloud platform is required for speedy, reliable, and convenient display and storage of ECG signals as and when needed.

i. Amazon Web Services (AWS) for cloud platform

In this work, AWS has been utilized as a cloud platform. A cloud-based application can be either created in the cloud or can be relocated from a prevailing infrastructure. Here, AWS cloud services have been employed to run applications and web servers in the cloud for hosting dynamic services. Also, for viewing the ECG data at all times [156].

ii. Hypertext Markup Language (HTTP) Server

In this implementation, an HTTP server is responsible for providing a GUI for the ECG waveform. HTTP is based on the conventional concept of request and response. Therefore, it can accept the user's responses and then acknowledge them accordingly. It is used to publish messages [157]. 'GET' request is sent by the user to access the IoT-cloud platform through a URL.

Later, a file that is scripted in the HTML is sent to the browser via HTTP. Next, the browser is then capable enough to transform the HTML file into a user-friendly GUI for logging on to the server and later viewing the ECG data.

iii. Storage Database

A storage database is required to stock the ECG files and data in the database so that the data can be viewed perpetually. This is done for convenient, reliable, and timely storage of the ECG data, which are significant functions of an IoT cloud [158]. In this work, MongoDB is used as the storage database. It is a document-oriented, cross-platform database. It provides high availability, high performance, and is easily scalable. It runs on the concept of collection and documentation. Since it stores data in documents, it is more adaptable and flexible to real-world requirements. Also, indexing improves the performance of the searches [159].

iv. Visualization Applications

In this work, the ECG signals can be viewed on a laptop, IoT-based platform, or smartphone. The signal on the laptop can be observed through the NodeMCU. The programming is done using the Arduino IDE platform. Also, the signal can be viewed on the AWS cloud platform, where the user can log in with their credentials. Further, the same ECG graph can be obtained on the smartphone.

All the major elements and components of the intelligent ECG tracking system have been scrutinized; in the following section of the paper, the process flow, and experimental results have been discussed.

3.8 Experimental Results and Analysis

Initially, the conventional Ag/AgCl electrodes are connected to the volunteers on the right wrist, left wrist, and right ankle as depicted in Figure 3.7.

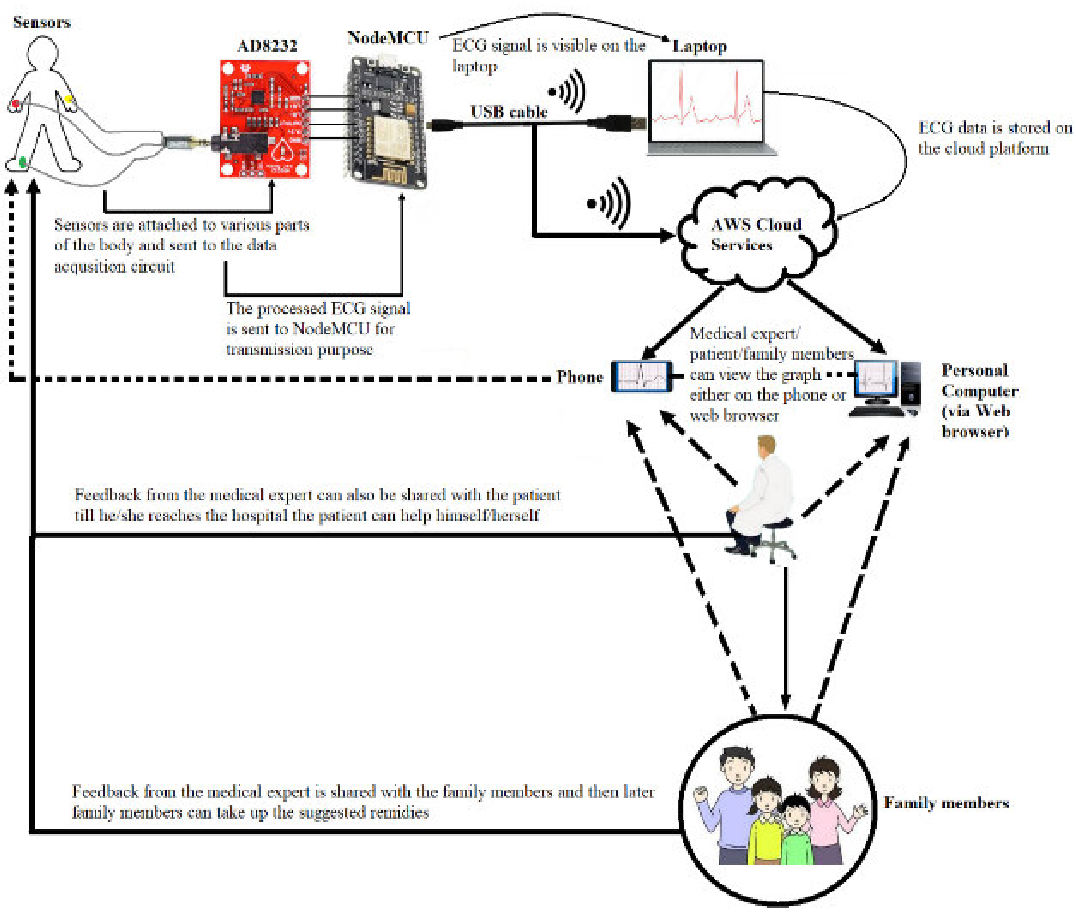


Figure 3.7: Pictorial depiction of the implemented smart ECG monitoring system

The three wires of the cable are then connected to the data acquisition circuit (AD8232). Later, after the signal has been filtered and processed, it is sent to the IoT-based development board (NodeMCU). This board is equipped with a Wi-Fi module. In the next step, NodeMCU was programmed using the Arduino IDE, and the connection to the Wi-Fi with the login ID and password. As a result, ECG signals were visible on the laptop screen. Then, the ECG value was pushed to the API for data transfer using 'HTTP Post'. Additionally, the latest value from NodeMCU is pushed to the API on the AWS cloud platform. At the backend, the value is received from the NodeMCU via 'HTTP Get'. The complete circuit is shown in Figure 3.8. After the value has been received, the application displays the ECG graph, as shown in Figures 3.9, 3.10, 3.11, and 3.12. The ECG signal can also be viewed using the web browser.

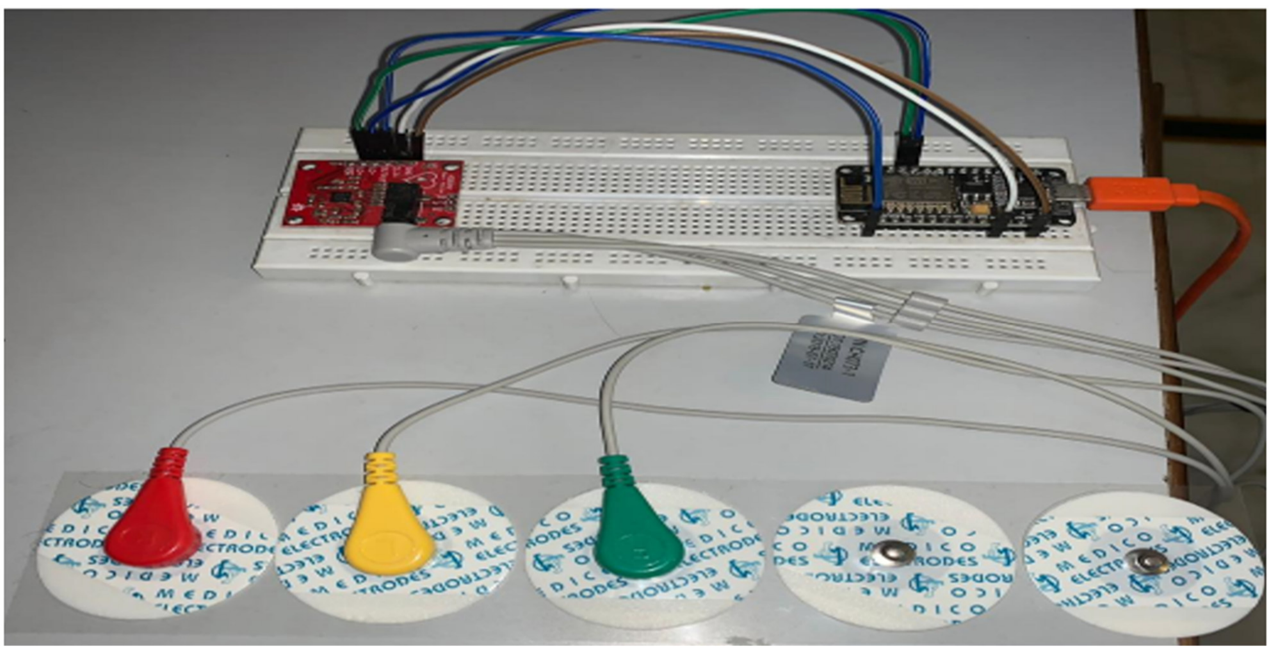


Figure 3.8: Complete circuit using AD8232, NodeMCU and Ag/AgCl electrodes

The experiment was performed on two female volunteers aged 29 years and 48 years. A 3-lead ECG configuration has been used since the system had to implement for long-term, continuous, and remote monitoring. Figure 3.9 displays the results attained from the 29-year-old volunteer.

Figure 3.9: Results attained from a 29-year-old volunteer

As depicted in Figure 3.10, the system displayed a clear ECG waveform. All the components of the ECG waveform, i.e., P, QRS, and T waves [160], are visible on the screen. A more explicit ECG graph is depicted in Figure 3.10.

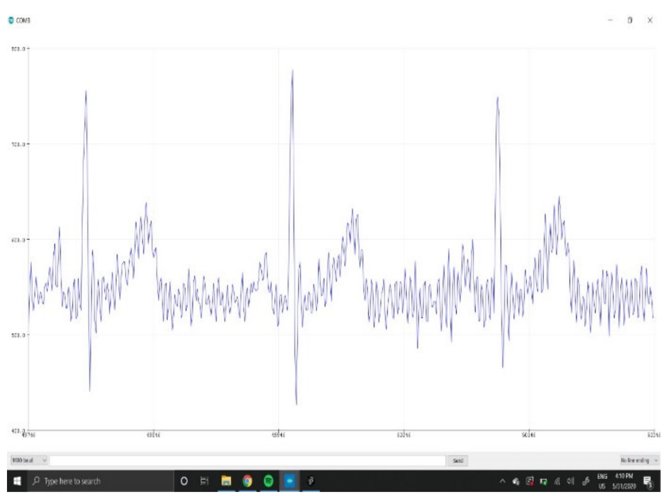


Figure 3.10: ECG graph screenshot from the laptop (29 years)

One more experiment was done with a 48-year-old female volunteer. The experimental setup was the same as discussed previously. Figure 3.11 displays the obtained ECG graph.

Figure 3.11: Results attained from a 48-year-old volunteer

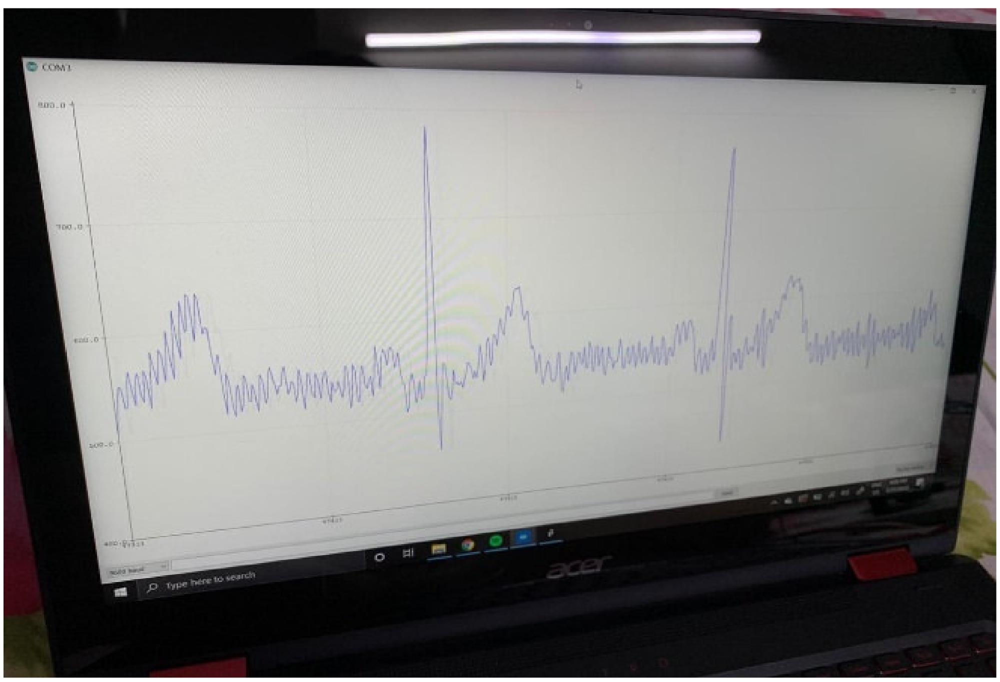


Figure 3.12: ECG graph screenshot from the laptop (48 years)

The above ECG results were obtained on the laptop screen. Later, the ECG results were viewed on the application, which was developed for remote viewing of the ECG signals. The results are displayed in Figure 3.13.

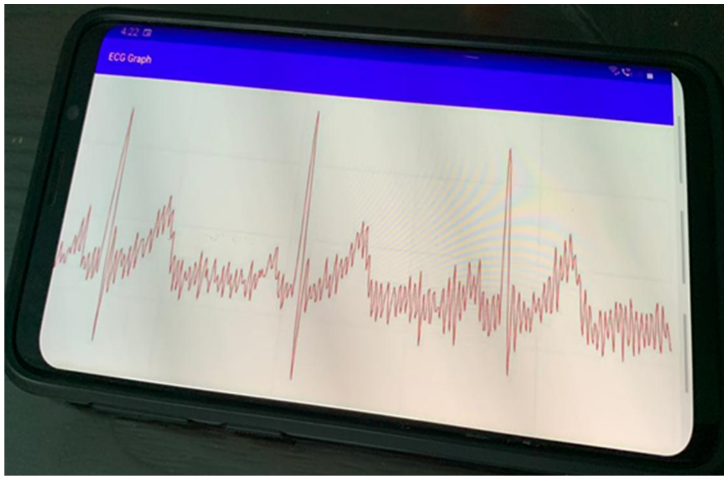


Figure 3.13: ECG waveform obtained on the smart-phone via an application

The ECG signals obtained on the smartphone are of good quality, and all the ECG components are visible. Hence, the implemented system portrays a good quality ECG signal and can be employed for long-term, convenient, and remote ECG monitoring. This helps reduce mortality due to heart diseases. If any sort of abnormal ECG is obtained, the medical expert can immediately intimate the family members/ patient with the remedy for the time being. This can be beneficial for the healthcare system as a remedy suggested by the doctor can save the life of the patient, and if the condition is not that critical, the patient/family member can follow the precautionary measures to reduce the severity of the condition. A significant benefit is that it will decrease the monetary load on the person, along with a reduction in the healthcare infrastructure for the nation. Secondly, the burden on the doctor is also reduced.

3.9 Conclusion

A smart ECG monitoring system has been designed and implemented using IoT-based methodologies and techniques. The architecture was designed and implemented using conventional Ag/AgCl electrodes by employing a 3-electrode configuration, which gathered ECG signals of good quality. Then, the ECG signals were sent to the cloud-based system via Wi-Fi through NodeMCU. Wi-Fi is used to provide wide-coverage areas and high data rates. The IoT cloud platform is responsible for storing the ECG data and making it visible to medical experts/patients/family members. The ECG graph could be viewed on the laptop screen, and/or a mobile phone using an application and web browser via the AWS cloud services. This system leads to convenient and remote ECG monitoring. This system can reduce the financial and infrastructural burden on the healthcare system around the globe.

CHAPTER 4

Fabrication Techniques

4.1 Introduction

Substitutions to the traditional pre-gelled Ag/AgCl ECG electrodes are extremely important in the medical community, largely when consistent and continuous monitoring of ECG signals is required. The current sedentary lifestyle of a significant portion of people attracts heart diseases, and it becomes appropriate to manage a healthy lifestyle, that is not restricted by parameters like time, space, and distance. This work requires ECG sensors that are highly conductive, light-weight, wearable, and flexible. Hence, it becomes essential to explore and analyze to fabricate ECG electrodes. This chapter focuses on several fabrication techniques like Spin coating, Screen Printing, Bar Coating, Mold casting, Metal Patterning, and many more.

4.2 Techniques for fabricating ECG Electrodes

4.2.1 Spin Coating

Spin coating technology is utilized for crafting paper-thin films while unloading the materials/composites on a flat and leveled surface known as the 'substrate'. It is achieved by rotating the substrate using centrifugal force. This process can be achieved in 4-steps as shown in Figure 4.1.

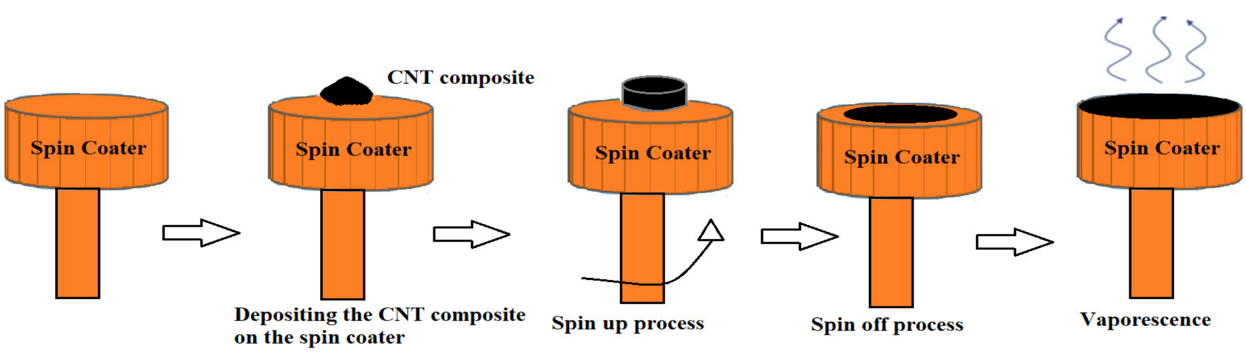


Figure 4.1: Spin coating process for electrode fabrication

The step first is placing the composite, then 'spin-up' the composite so the that maximum solution evaporates. The intention is to make the substrate wet. Following this, the next step is a 'spin-off' that removes the solution from the boundaries of the substrate. The final step is 'evaporation', although evaporation is a continuous process. Rapid rotation is necessary to dry off the solvent completely. The degree of how smooth or rough the final film depends on the evaporation process [161, 162]. Therefore, the highly volatile components which are the solvent (mainly) get removed from the substrate. The thickness of the accumulated layer depends on the viscosity of the composite and the rotational speed of the spin coater [163], and a uniform film was fabricated. Also, a comparison has been presented of different electrodes fabricated using this technique in Table 4.1.

Table 4.1: Comparison between Spin-coated Electrodes and conventional Ag/AgCl electrodes

S.No	Ref	Comparative results	SNR
1.	[31]	ECG signals of the fabricated electrodes at 1.5wt% were comparable with the conventional electrodes. The fabricated electrodes were more flexible, stretchable, and suitable for long-term monitoring.	-
2.	[32]	ECG signals from the dry fabricated electrodes were comparable to the conventional Ag/AgCl electrodes.	-
3.	[59]	The experimental ECG wave was comparable with the 3M (commercial) ECG electrodes	-
4.	[50]	The conductance of the developed electrodes was less than the Ag/AgCl electrodes, nevertheless, the results were satisfactory.	SNR of fabricated electrodes: 39.8 dB which was better than the conventional electrodes
5.	[48]	ECG signals from the dry fabricated electrodes were comparable to the conventional Ag/AgCl electrodes.	-
6.	[30]	ECG signals from the dry fabricated electrodes were comparable to the conventional Ag/AgCl electrodes.	-

To wrap up, the spin-coating technique is beneficial for fabricating thin film. Thin electrodes can be integrated with portable devices. The process of procuring the electrodes is not difficult. The only requirement is to place the composite on the spin-coater and within the limited period, uniform electrodes/film is obtained. The only drawback lies with the initial capital required to procure the spin coater, which is quite heavy on the pocket. Nonetheless, the return on investment can be sustained when electrodes are fabricated on a large scale.

4.2.2 Metal Patterning

Patterning is a procedure for generating replicas of an article that is to be crafted. Henceforth, it is utilized for constructing a 'cavity' into which the composite is poured over, and can be extracted after fabrication as depicted in Figure 4.2.

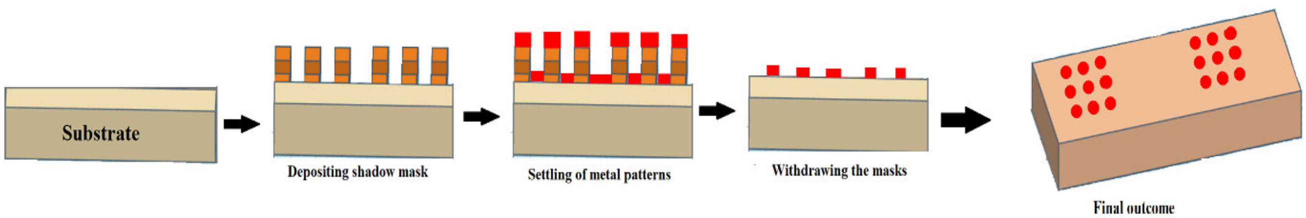


Figure 4.2: Electrodes fabricated with the metal frame

Patterns can be crafted with metals, wood, metals, etc. Using metal casts is better since these are long-term durability and endurance, and most importantly not greatly affected by atmospheric moisture. Table 4.2 gives a comparative analysis with other works.

Table 4.2: Comparison between metal-patterned electrodes and Ag/AgCl electrodes

S. no	Ref.	Comparative evaluation	SNR
1.	[164]	ECG signals from the dry fabricated electrodes were comparable to the conventional electrodes.	-
2.	[42]	ECG signals from dry fabricated electrodes were better than conventional Ag/AgCl electrodes.	-
3.	[51]	ECG signals from the dry fabricated electrodes were better than conventional Ag/AgCl electrodes, without any attenuation or distortion even when worn for a week.	-

4.2.3 Mold Casting

This procedure depicted in Figure 4.3 employs the formation of a mold using any polymer like PDMS [165]. A substrate like glass/Teflon/Acrylic sheet is affixed to the center of the petri dish along with the snap clips. Then the selected polymer is poured over the substrate in the petri-dish and placed in the oven for thermal curing. Subsequently, after the thermal-curing process, the polymer mold is de-molded from the petri-dish. Composite/mix was then poured into the mold and put inside the oven for evaporation and thermal curing.

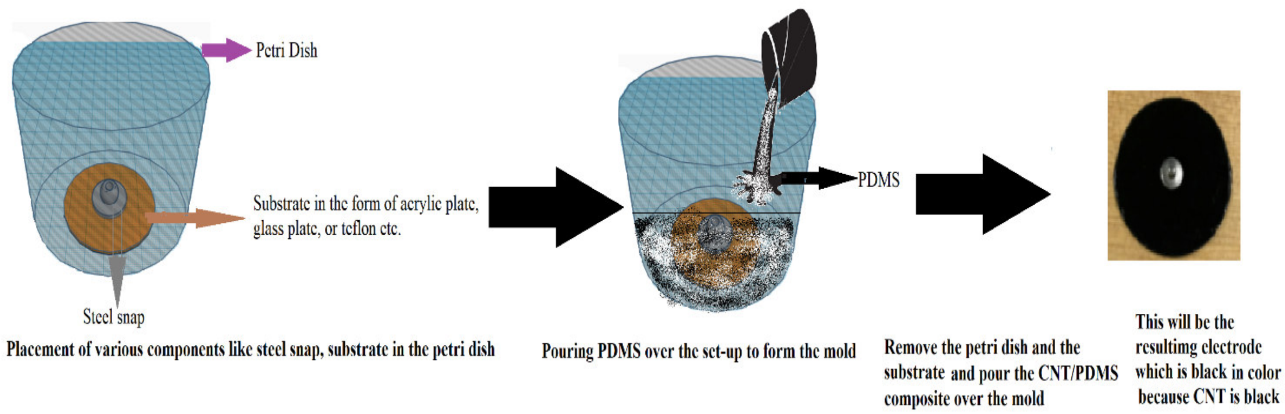


Figure 4.3: Mold casting technique

The electrode is the end product and can be used for biomedical sensing. Also, a comparative analysis of various works can be seen in Table 4.3.

Table 4.3: Comparison between conventional Mold-Casted electrodes and Ag/AgCl electrodes

S.No	Ref	Comparative results	SNR
1.	[166]	ECG signals from the dry fabricated electrodes at 4.5wt% were better than conventional Ag/AgCl electrodes.	SNR of the fabricated electrodes at 2wt%: 45.8 dB almost equal to Ag/AgCl conventional electrodes: 44.5 dB.
2.	[33]	5wt% CNT/PDMS electrodes with 12 hours of sonication and conventional Ag/AgCl electrodes provided almost comparable ECG results.	SNR was 31.2 dB (ultrasonication time:16 hours)
3.	[52]	ECG signals from the dry fabricated electrodes were comparable to conventional Ag/AgCl electrodes at a concentration above 4wt%.	-

The advantage of utilizing this technique is its ease of use and it is inexpensive. The reason is that once a mold is fabricated, numerous electrodes can be fabricated. The only problem is that with a single mold, only a single electrode can be fabricated, at least 5-6 molds should be fabricated. The complete procedure is not much time-taking. Likewise, pricey machines and resources are not required for electrode(s) fabrication.

4.2.4 Screen Printing

The Screen Printing procedure comprises the direct deposition of ink/composite/mixture on the surface of the substrate. The imprint to be reproduced is photographically transferred to an extremely thin fabric identified as the ‘screen’. The green blocks are the non-printing segments, and the fabric performs the role of a stencil, as depicted in Figure 4.4.

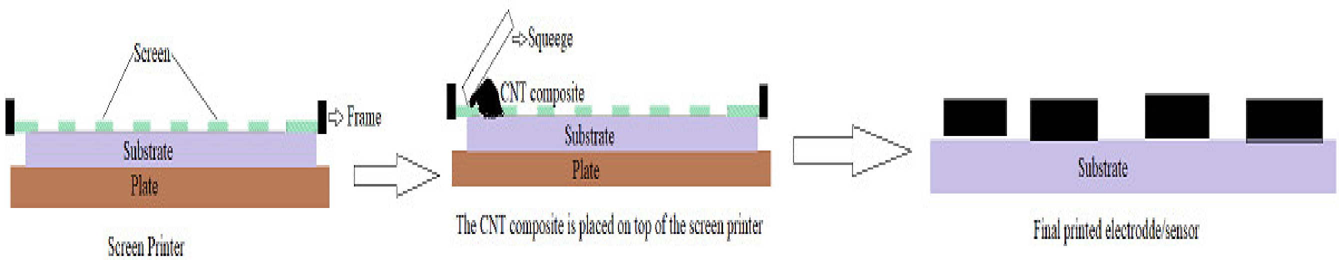


Figure 4.4: Screen printing technique

The consequent step was spreading the ink/composite/mixture across the whole screen, which drifts across the unblocked segments, and lastly arrives at the substrate. The procedure was easy, and many electrodes can be fabricated in a minimum time frame. Various fabrications done using this technique are discussed in Table 4.4.

Table 4.4: Comparison between Screen Printed electrodes and conventional Ag/AgCl electrodes

S. no	Ref.	Comparative evaluation	SNR
1.	[133]	-	-
2.	[167]	-	-
3.	[39]	-	-
4.	[168]	-	-
5.	[25]	-	-

To wrap up, the screen-printing method is extremely simple. People can choose the substrate on which they have to print the composite. One drawback is the investment in the screen printer, although it's a one-time investment. The benefit of using the screen printers is that the output is a smooth and uniform image and is not time-consuming and the electrodes can be produced in bulk.

4.2.5 Doctor Blade Technique

It is one of the effortless, convenient, and easy methods for the fabrication of thin ECG films. The procedure begins with acquiring a glass substrate that is commonly used, and then placing the scotch tape across and along with the thickness of the substrate as seen in Figure 4.5.

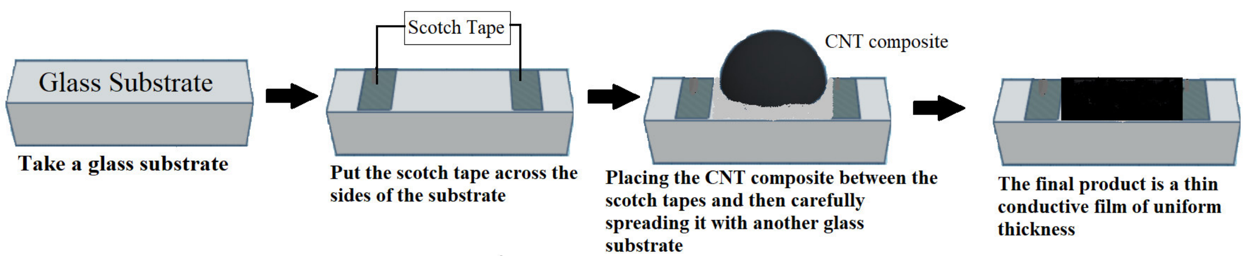


Figure 4.5: Doctor Blade Technique

The thickness of the electrode is determined by the number of layers of scotch tape which is spread over the substrate. After smearing the scotch tape, the prepared mixture is deposited between the scotch tape. Next, with the help of another glass slide, the mixture is spread throughout the substrate. The end-product is a thin film paper electrode(s), which is simply detached from the glass substrate. Various works are done using this technique and their comparative analysis is discussed in Table 4.5.

Table 4.5: Comparison between electrodes made of Doctor Blade technique and Ag/AgCl electrodes

S. no	Ref.	Comparative evaluation	SNR
1.	[38]	The signal amplitude of the developed electrodes was less than the Ag/AgCl electrodes	-
2.	[43]	The signal amplitude of the developed electrodes was less than the Ag/AgCl electrodes	-

This method is very simple and economical. The resources required for fabrication are readily available.

4.2.6 Using CNTs and aqueous-hydrogel

'Hydrogels' are a category of polymers with a cross-linking system that includes 90% of water. These polymers can occur naturally or can be produced synthetically. Hydrogels are highly compatible and can be used for biomedical applications. A comparison table of various works is presented in Table 4.6.

Table 4.6: Comparison between electrodes made of Aqueous hydrogels and Ag/AgCl electrodes

S. no	Ref.	Comparative evaluation	SNR
1.	[37]	Developed electrodes did not cause any inflammation when compared with conventional electrodes	-
2.	[27]	ECG signals from the dry fabricated electrodes were better than conventional Ag/AgCl electrodes.	Fabricated electrodes had better SNR (35dB) when compared with the Ag/AgCl electrodes (30dB)

The benefits of utilizing hydrogel-based electrodes are their smooth and reliable properties. These are excellent for prolonged applications. These sensors offer low-impedance, and a high SNR, which is the most important when physiological measurements are taken into consideration [169].

4.2.7 Bar Coating

A Bar Coater as shown in Figure 4.6 is utilized for developing composite-based or mixture-based films. It is utilized to equally and identically spread inks or composites or paints over the substrate. Every bar-coater possesses "theoretical-wet-film-thickness," which governs the thickness of the films. The equipment is available with many options sizes and costs.

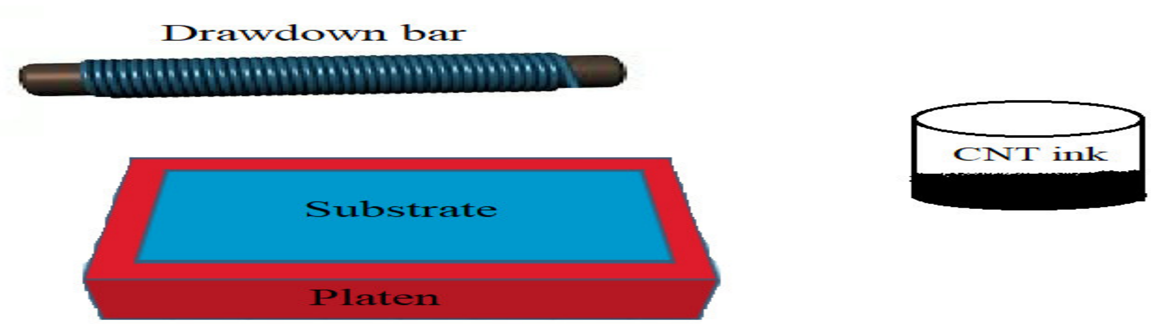


Figure 4.6: Bar coating process

The bar-coater provides a uniform and smooth electrode, unlike the doctor blade technique, which sometimes gives rough and uneven surfaces. A comparison in Table 4.7 is presented below.

Table 4.7: Comparison between Bar coated electrodes and Ag/AgCl electrodes

S. No	Ref	Comparative results	SNR
1.	[34]	ECG results were better than the conventional electrodes.	-
2.	[36]	ECG results were better than the conventional electrodes.	-
3.	[46]	ECG results were better than the conventional electrodes.	-

To conclude, the cost of the bar-coater is quite high, but a uniform, smooth and thin film is fabricated. Also, this fabrication process is efficient in terms of time required for fabrication, electrodes can be fabricated at a faster pace, and there are bright chances for bulk production. The procedure of fabrication is not difficult.

4.2.8 Dipping and Drying technique

The method involves dipping the yarn or fabric into the MWCNT or the SWCNT solution. Subsequently dipping the material/fabric/cloth is dried up and later utilized for monitoring the physiological signals as shown in Figure 4.7.

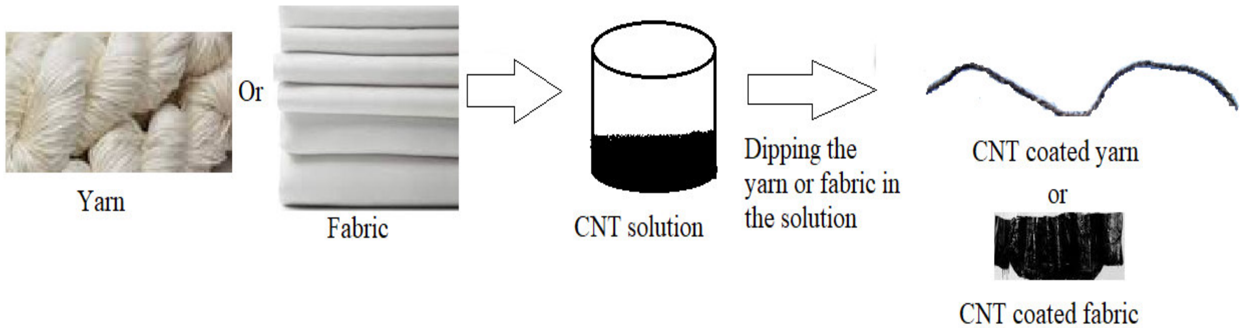


Figure 4.7: Dipping and drying process

The dipping and drying process is not difficult, but the process of making yarns is time-consuming, difficult, and heavy on the pocket most of the time. A comparison table is presented in Table 4.8.

Table 4.8: Comparison between Dipped and Dried electrodes and Ag/AgCl electrodes

S. no	Ref.	Comparative evaluation	SNR
1.	[28]	-	Fabricated ECG electrodes had an SNR comparable with the metal-based electrodes
2.	[29]	ECG signals from the dry fabricated electrodes were comparable with the metal-based electrodes.	-

4.2.9 Fabrication of electrodes with CNT pillars or CNT-arrays

Carbon Nanotubes are grown as arrays or pillars on a specified substrate. Pin-shaped electrodes are fabricated and matured with mounting CNTs as forests with extremely controlled environments and conditions. The two ways in which the CNTs can be grown are either on the substrate or employed with the polymer. Hence, these electrodes don't require the application of gel on the human skin, therefore these electrodes can be termed 'capacitive' electrodes. Various fabrications have been done using pillars and arrays and are presented in Table 4.9.

Table 4.9: Comparison between Pillar/Array electrodes and Ag/AgCl electrodes

S.No	Ref	Comparative results	SNR
1.	[58]	ECG signals from the dry fabricated electrodes were comparable with the conventional electrodes.	-

2.	[56]	-	-
3.	[170]	-	-
4.	[57]	ECG signals from the dry fabricated electrodes were comparable with the conventional electrodes.	-
5.	[171]	ECG signals from the dry fabricated electrodes were comparable with the conventional electrodes.	
6.	[44]	-	Lower SNR than Ag/AgCl electrodes was observed.

The fabricated electrodes with this process are sturdy and strong. These electrodes are robust for extreme conditions. One drawback is the extremely controlled environment required for the growth of CNT pillars. The resources and equipment(s) required are indeed heavy on the pocket.

In addition to these methodologies and techniques, many other processes in which researchers have developed ECG electrodes. As these techniques were not placed with any of the discussed techniques, therefore, these various methods are discussed in brief.

4.2.10 Other Unconventional Techniques for Fabricating CNT-based Electrodes:

In [20], ECG electrodes were fabricated using **SWCNTs and two binders: DNA and Chitosan**. Two types of electrodes were fabricated: SWCNT/DNA and SWCNT/Chitosan. The developed electrode's performance was way better and much improved than the bucky paper, i.e., more conductive, water-proof, and robust. ECG was not measured, but these electrodes have the potential to measure the same along with pace-makers. Penghua Sun et al [21] fabricated a 3-in-1 sensor which included a gas sensor, measured body temperature, along with a pressure-based ECG sensor. The fabricated sensor was developed by utilizing **CMOS- technology**. SWCNTs were utilized for the fabrication process and were placed between the 'source' and the drain' of the CMOS. A decent ECG waveform was observed.

C.L. Lam et al. [60] fabricated electrodes with **MWCNT and cotton**. MWCNT was dispersed in tapioca starch. Later, with the help of a paint-brush, MWCNT/tapioca starch was painted on the cotton cloth was placed in the oven for drying. Afterward, a thin gold film was used on the cotton cloth for evading charging effects.

In [23], a fully printable **SWCNT-based Thin Film Transistor (TFT)** was fabricated using **inkjet printing**, with a top-gate structure. This sensor was developed on a stretchable substrate, and ECG could be monitored in a real-time scenario for online applications. The developed sensors were economical and displayed a good ECG waveform, and bulk manufacturing is possible.

Benyan Liua et al [41] developed CNT/PDMS-based electrodes. AgNWs and PDMS were used to create the base. MWCNT and aPDMS were used to create the adhesive layer. The AgNW-solution was dropwise added to the membrane/filter paper, and later to the PDMS layer, and consequently, aPDMS was mixed with the composite. Later, a copper wire was rolled on top of AgNW coating which was coated on CNT/aPDMS composite. The final assembly of all the composites was placed in the oven for the thermal curing of the structure. The properties of the developed sensor included being durable, excellent conformality, and robust.

Martínez et al [40] electrodes are fabricated with Deionised (DI) water, MWCNT, and Polyvinylpyrrolidone (PVP) by ultrasonication for 1 hour. Then, Graphene conductive paint and Silver conductive paint was added to the above solution. The nanocomposite was then deposited on the Xerox-bond-paper and was dried at room temperature. Lastly, the electrode was extracted. The ECG signals were comparable to the conventional electrodes.

In [45], ECG sensors utilizing MWCNT, PDMS, Polyethylenimine (PEIE), and Polyethylene terephthalate (PET) substrate were fabricated. PEIE and PDMS were mixed in the ratio of 1:200wt%, deposited on the patterned adhesive tape, and cured @ 100ºC. 10wt%-CNT, 3wt% PEIE, and PDMS were mixed. This nanocomposite was poured over the PET substrate. ECG could be monitored in a real-time scenario for online applications. The developed electrodes were flexible and comfortable. These self-adhesive electrodes worked well for up to 100 cycles of attachments and detachments. The ECG electrodes were flexible and comfortable. Decent ECG waveforms were achieved in three scenarios: before exercise and after exercise, and after cleaning the sweat.

Jeong Hun Kim et al [47] electrodes were developed using IPA and MWCNT by sonication process. Subsequently, the PDMS (component A) and Monoethyl Phthalate (MEP) are sonicated together. Then, the cross-linking (PDMS-B) was added to the nanocomposite and thermal curing was done for approximately 2 hours, and a silicone rubber was the end product, which was flexible and conductive. The fabrication procedure was simple and pocket-friendly, and ECG results were of appreciable quality.

In [172], paper-based, and flexible physiological sensors using Graphene (chemically modified) and MWCNT-COOH were fabricated. The prepared composites were **dropped-casted** on the

0.2µm nylon filter paper, and the vacuum process was used for filtration. Then the nylon-membrane-filter paper was placed in the oven for drying, and 25wt% (concentration) portrayed the greatest amplitude and was comparable to the conventional ECG electrodes.

Youngpyo Ko et al [173] fabricated **Stretchable Conductive Adhesives (SCA)** utilizing Silver/Silicone adhesives and MWCNT. MWCNT and Ag particles were mixed, and later the silicone rubber was mixed with the above composite. The conductivity of the developed SCA was in the range of 1050-6450 S/cm. Later, the SCAs were printed on the PDMS for ECG monitoring. The electrodes displayed outstanding ECG results while the person was in motion and many cycles of attachments and detachments.

In [174], electrodes were developed using SWCNT and using Polyvinylidene-fluoride hexafluoropropylene (PVDF-HFP). PVDF-HFP and acetone were mixed and later functionalized SWCNTs functionalized (2-ureido-4[1H] pyrimidinone) were mixed with acetone in a bath sonicator. Subsequently, 5mm of this composite was deposited into the **air-brush** (Falcon, FD-116A). Later Blow spinning technique was used for electrode fabrication. The developed electrodes were, stretchable, flexible, and displayed decent ECG waveforms.

In [175], electrodes were fabricated with '**water-based-transient-resistive screen-printing using** MWCNT, distilled watermethylcellulose, and Sodium DodecynBenzeneSulfonate (SDBS). The screen-printing paste was made using methylcellulose resin and MWCNT and was printed on the 100 µm PET substrate. Good ECG waveforms were observed. But one drawback is that some skin irritation is observed due to SDBS.

A comparison table of all the works has been presented in Table 4.10.

Table 4.10: Comparison between other electrodes and Ag/AgCl electrodes

S.No	Ref	Comparative results	SNR
1.	[20]	-	-
2.	[21]	-	-
3.	[60]	ECG results were better than the conventional electrodes.	-
4.	[23]	-	-
5.	[41]	Fabricated dry electrodes were observed to have lower signal amplitudes when compared with the Ag/AgCl electrodes.	-
6.	[40]	ECG results were comparable to the conventional electrodes.	-
7.	[45]	-	-
8.	[47]	The power of the fabricated electrodes was lower than Ag/AgCl electrodes	-
9.	[172]	ECG results of the fabricated electrodes were comparable to the	

		metal-based electrodes	
10.	[173]	-	-
11.	[174]	-	-
12.	[175]	-	-

4.3 Comparison of Different Fabrication Techniques

Recent decades have observed excessive development in the sphere of stretchable and flexible electronics. The advances in this turf have certainly given birth to an impressive change in the field of biomedical applications. Numerous electrodes for ECG monitoring have been developed with these processes and methodologies. This unit of the chapter recapitulates the techniques and processes used for the fabrication of electrodes are specified in Table 4.11.

Table 4.11: Fabrication Methods in a Nutshell

S.No	Process name	Method in brief	Advantages	Limitations
1.	Spin Coating	The composite/mixture is deposited on the substrate and the spin-coater is rotated at the required speed to form a uniform film.	Easy process of fabrication, saves time, bulk production possible	Expensive equipment
2.	Metal Patterning	The mounts are formed with metals and cavities are formed for depositing the mixture and are later removed from the cavity	Bulk production possible	The process is time-consuming and a bit tedious
3.	Mold Casting	A mold is cast with a polymer deposited on the substrate and then thermally cured and extracted.	Mass production is possible, not too expensive, simple process	A bit time-consuming
4.	Screen Printing	The nanocomposite/mixture is deposited on the screen-printer. The image is transferred to the screen. The squeegee then smears the nanocomposite/mixture through the screen of the unblocked segments.	Time-saving and mass production are possible with high throughput	Expensive equipment
5.	Doctor Blade	The mixture is deposited on the glass slide and then smeared via another glass slide. The thickness of the	Pocket-friendly, easy process, materials are easily obtainable,	Occasionally rough films can be produced with

		electrode depends on how many scotch tapes are wounded on both sides	bulky equipment(s) not required	inappropriate smearing of the composite.
6.	Aqueous Gel	Sensors are fabricated by either depositing the CNT on the hydrogel/printing method	Bulk production possible	Tedious fabrication process
7.	Bar Coating	The nanocomposite is deposited on the substrate and later placed on the bar coater. Later, a drawdown bar is utilized for pushing the mixture and making it into a flat, uniform film.	Uniform-film, bulk production is possible, and the thickness of the film can be regulated.	The equipment is expensive and bulky.
8.	Dipping and drying	Fabric/yarn are dipped into the CNT solution and dried at room temperature	Simple fabrication process	Yarn production can be difficult
9.	CNT pillars or CNT arrays	CNT forests are either grown on the substrate/polymer or the polymer and are equally spaced to enhance breathability	Good electrical conductivity and decent ECG waveforms were obtained	Growing CNT pillars in a controlled environment are difficult
10.	Other unconventional methods [20]	SWCNT/DNA and SWCNT/Chitosan electrodes were fabricated	Other binders can also be utilized	The fabrication process is difficult
11.	[21]	CMOS technology	Software simulation was done	The process of fabrication is difficult and decent ECG signals were not observed
12.	[60]	Paintbrush technique	Cheap materials, easy fabrication process.	Non-conformable to the skin
13.	[23]	Inkjet Printing and TFT	Real-time monitoring for online applications	Ink-jet printing is an expensive technique
14.	[41]	Filter-paper-membrane technique	Easy disposal of the materials	Time, and effort-consuming process
15.	[40]	Xerox bond-paper technique	Easy disposal of the materials	A lengthy process of

				fabrication
16.	[45]	Laser cutter tool technique	The easy fabrication process and materials are easily available with real-time ECG monitoring.	The equipment is expensive
17.	[47]	In-situ polymerization	The cost-effective, easy fabrication process and materials are easily available	The process is time-consuming
18.	[172]	Drop casting technique	Materials are available easily and good ECG waveforms are observed	The fabrication process is a bit tedious
19.	[173]	SCAs using MWCNT and Silver/Silicone adhesives	Easy availability of materials and easy process	Expensive process
20.	[174]	Blow Spinning technique	Decent ECG waveforms were observed	Expensive equipment(s)
21.	[175]	Water based-transient resistive screen-printing	Materials were easily available	Expensive equipment(s), in addition to skin irritations.

4.4 Conclusion

In this chapter, various methods and techniques for the fabrication of ECG sensors and electrodes have been discussed. The fabrication processes are represented pictorially, along with their advantages, disadvantages, and feasibility of manufacturing. According to the processes presented above, the most refined and simple process for fabricating the electrodes is the 'mold casting' process. The materials required such as petri-dish, substrates like Teflon, glass, or acrylic sheets can be obtained easily and at economical costs. The equipment(s) required are not voluminous. Also, the fabrication process is not difficult. After fabricating the molds, the person is only required to pour the composite into the mold and after thermal curing the end product is ready. Once the molds are cast, several electrodes can be developed via the 'replication' process.

CHAPTER 5

Fabrication of ECG Electrodes - Fabrication Technique I

5.1 Introduction

In this chapter, MWCNT-COOH/PDMS and SWCNT-COOH/PDMS ECG electrodes are fabricated using Fabrication Technique I. This technique includes mold casting, dispersion of CNTs with solvent, dispersion of CNTs with PDMS, and placing the solvents in the molds. The types of equipment included a magnetic stirrer, probe sonicator, and oven. The ECG was measured using the system discussed in Chapter 3.

5.2 Fabrication of MWCNT-COOH/PDMS Electrodes

5.2.1 Materials

MWCNT-COOH were acquired from **NanoResearch Elements**. It possesses better dispersing abilities than unfunctionalized CNTs. As functional groups are attached, it creates spaces between the CNTs so that it causes fewer agglomerations. Functionalized CNTs provide better interfacial bonding between the PDMS and the CNT. This interfacial bonding also enhances the conductivity. The polymer for this research was **PDMS Sylgard 184 Silicone Elastomer from Dow Corning** due to its impressive properties including flexibility, non-toxicity, and biocompatibility. A good solvent is required for dispersing the CNTs as the materials agglomerate in a short span of time. Therefore, **IPA** was selected as both CNTs and PDMS are soluble in IPA. Figure 5.1 presents the materials used in the fabrication process.

Figure 5.1: Materials used for fabrication

5.2.2 Mold Casting

Molds were devised for the development of the CNT/PDMS-based ECG electrodes. The process of fabricating these molds is very simple. The materials required for fabrication are Sylgard 184 Silicone Elastomer from Dow Corning which includes 2 components base: Sylgard184A and curing agent Sylgard 184B, acrylic sheet, petri-dish, and oven. The process began by sticking the acrylic sheet at the bottom of the petri dish using double tape. The diameter of the acrylic sheet is 50mm, and the thickness is 15mm. Then a mixture of PDMS cross-linking agent and PDMS base in a 10:1 ratio was prepared and poured into the petri-dishes. Then, the petri-dishes were thermally-cured at 80ºC for 30 mins in the oven. Then the molds were extracted from the Petri-dishes. The molds were developed with a cavity equivalent to the thickness of the acrylic sheet. The cavity is used to hold the CNT/PDMS composite for electrode fabrication as depicted in Figure 5.2 and Figure 5.3.

5.2.3 MWCNT-COOH/PDMS Composite Preparation

(a) Distribution of MWCNT-COOH in IPA

To attain excellent conductivity, CNTs must be dispersed efficiently. Appropriate dispersion leads to the proper formation of conductive networks, which leads to decent ECG results. Before dispersing the MWCNT-COOH with the help of an ‘ultra-sonicator’, it is first distributed using a ‘magnetic stirrer’. MWCNT-COOH and IPA were stirred with the help of a REMI 5MLH magnetic stirrer @ 300 RPM for 10 minutes. Thirteen electrodes were developed with varied concentrations as follows: 0.1wt%, 0.25wt%, 0.5wt%, 0.75wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt%, 3wt%, 3.5wt%, 4wt%, 4.5wt%, and 5wt% as depicted in Figure 5.4.

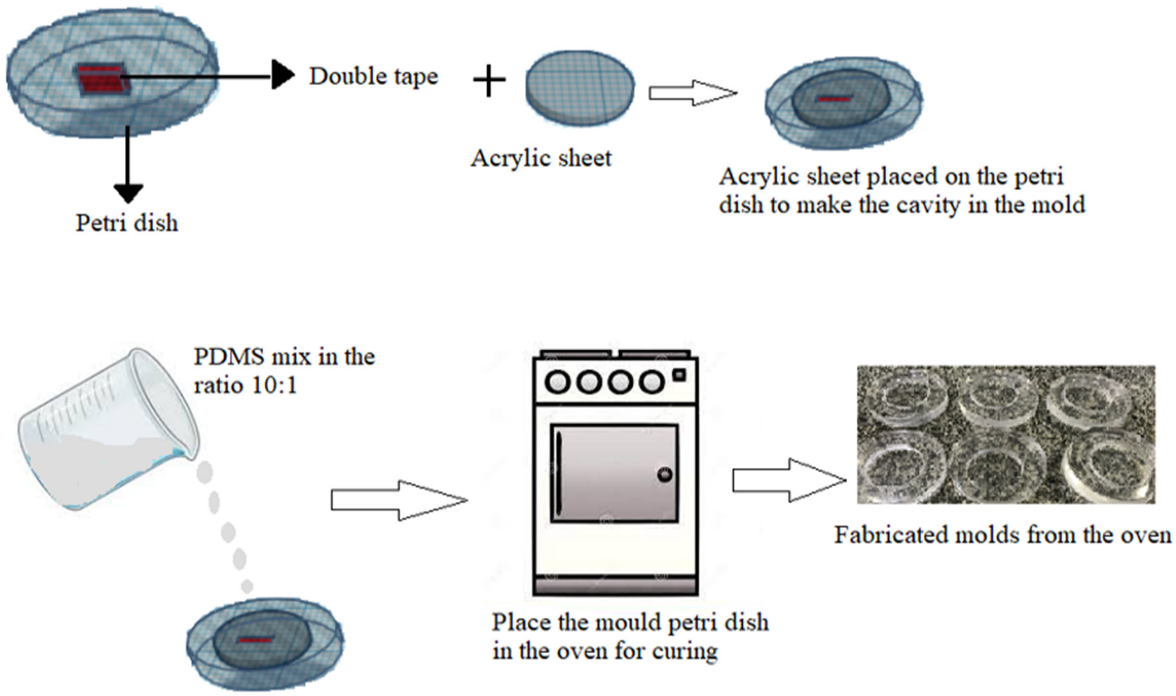


Figure 5.2: Mold Casting process

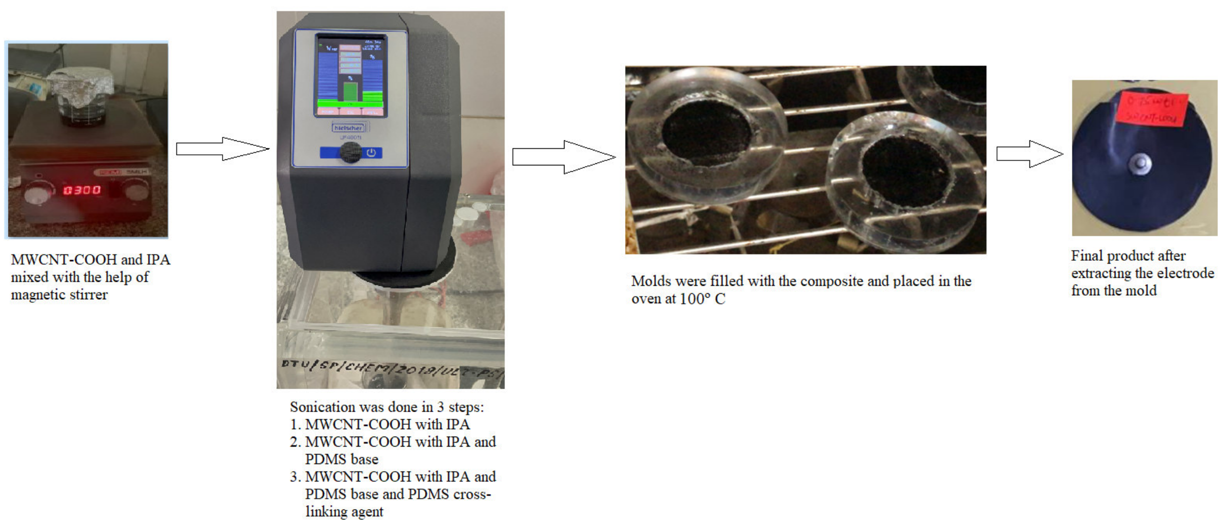


Figure 5.3: Process of fabrication

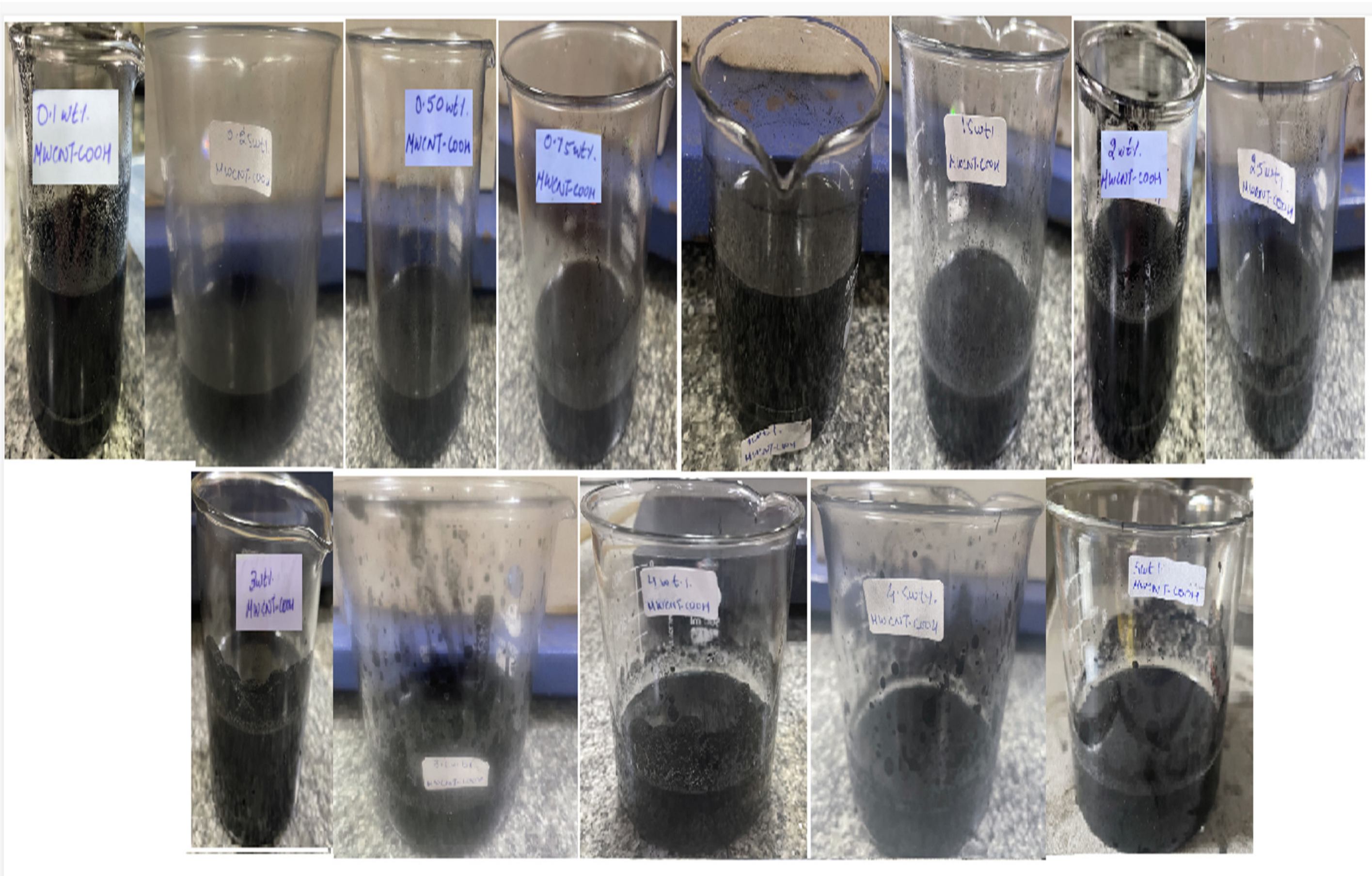


Figure 5.4: Various composite concentrations of MWCNT-COOH/PDMS solutions

(b) Dispersion of MWCNT-COOH with IPA

The big chunks (if any) are broken down into smaller particles, but not fully untangled. Therefore, to fully break down the MWCNTs and bring it into a clear solution form, it is necessary to sonicate the above solution. This is because the stirrer mixes the solution, but the sonicator with rigorous agitation blends the solution. The various compositions of MWCNT-COOH with IPA were sonicated using a probe sonicator, Hielscher UP400St. The sonication was done for 30 minutes with 34W, and the amplitude was 44%. The sonication was done only for 30 mins, to conserve the properties and characteristics of the material. With the help of this powerful sonicator, no aggregates were seen.

(c) Dispersion of MWCNT-COOH with PDMS

To bring this solution to the final form of an electrode and to induce flexibility, PDMS is mixed with it. The PDMS base was dispersed to the above solution with the probe sonicator for 10 minutes, with an amplitude of 58% and 34W. Later, PDMS curing agent was dispersed to the above solution. This is used to cure the solution and provide hardness to the solution. The last dispersion was done for 5 minutes with an amplitude of 56% within 31W power.

(d) Final Fabrication of Electrodes

The final step is to pour the prepared nanocomposites into the molds and place them in the oven for drying and curing purposes. The temperature for curing was at 100ºC and was kept constant throughout the process. Different concentrations of MWCNT-COOH/PDMS composite solutions needed dissimilar durations in the oven, ranging from 2 to 10 hours. When the nano-composite dried, the circular films were demoulded from the molds. Finally, the aluminum conductive snaps were pasted on top of the electrodes with the help of conductive Ten20 paste as shown in Figure 5.5.

Figure 5.5: Electrodes of various concentrations of MWCNT-COOH/PDMS

5.2.4 ECG Measurements and Testing

(a) Physical Characterization

MWCNT-COOH was examined as a solid residue with the help of Raman Spectroscopy, TEM, and FTIR. TEM provides indications of interior assemblies of the material. The concentration of -COOH in MWCNT is in the range of 2-4wt%. The -COOH functional groups in the MWCNT separate the CNT bundles, which further improved the disintegration and solubility. As observed in Figure 5.6, naturally the MWCNTs are packaged in bundles, but when the -COOH group was added, the MWCNTs were isolated from one another. Functionalized CNTs create spaces between the CNTs and hence dispersion abilities are improved. Functionalization also helps in the improvement of the surficial activities of the CNTs. FITR was carried out to identify the chemical bonds, and to provide evidence of the covalent bonding of the CNTs. FITR is achieved in the range of 400-4000cm^{-1}. Figure 5.6 (graph) offers evidence of the wave absorption by the -COOH group. The dual peaks can be seen at 3700 cm^{-1} and 1500cm^{-1} which implements the strongest bonds at these two values. Raman spectroscopy offers comprehensive information on the composition and molecular structure.

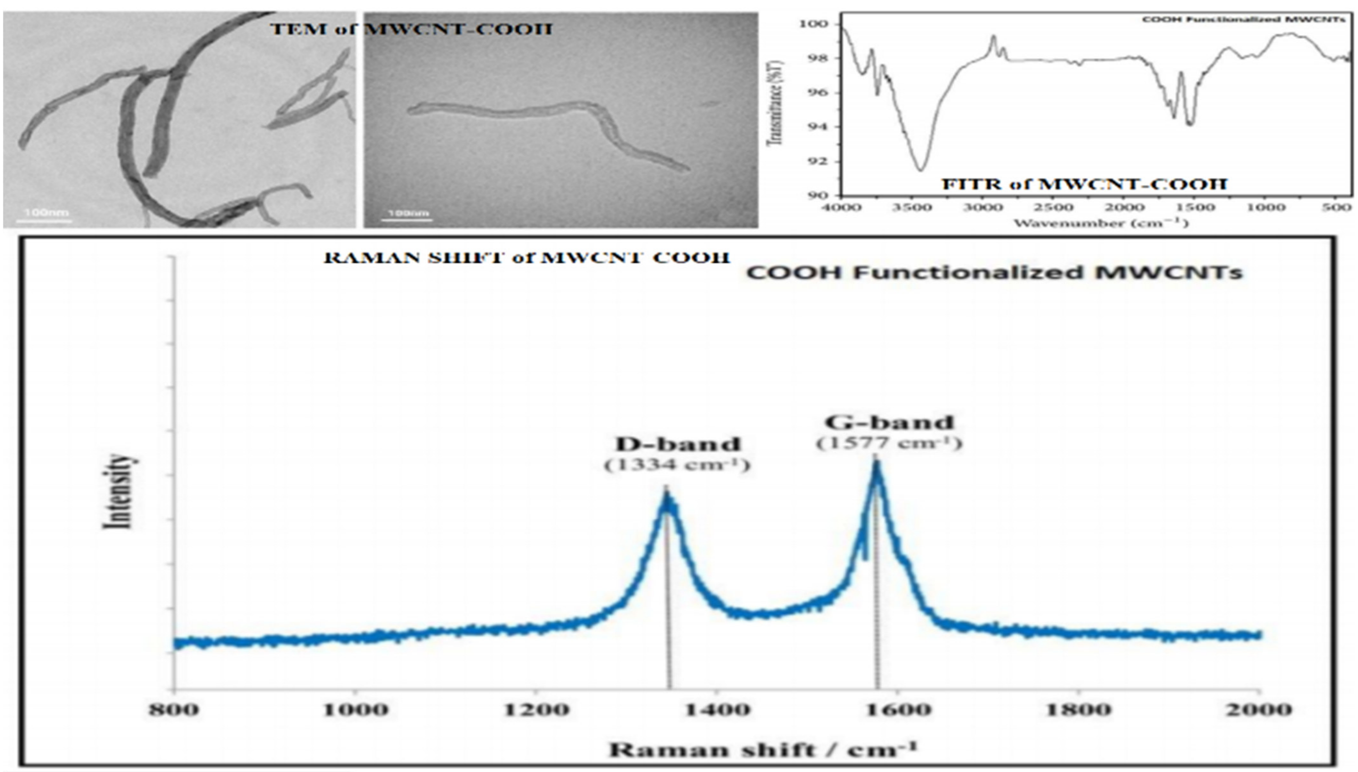


Figure 5.6: Physical Characterization of MWCNT-COOH

(b) Long-Term ECG Monitoring

All the electrodes were worn for 5 days for checking their long-term compatibility of the electrodes. The results on the 3rd and 5th day were a little degraded but after cleansing with methanol, the results revived. The electrodes were safe to wear, and no skin irritation or allergy was identified.

(c) Capacitance Measurements

Capacitance can be defined as an electrical property that offers information on the flow of electrons in a specific conductor. As observed in Figure 5.7, there is an increase in the value of capacitance as there is an increase in the concentration of MWCNT-COOH in the nanocomposite, which is a good trend. As there will be more flow of electrons, collisions will be less and will enhance the conductivity in the electrodes. It was measured using ESCORT ELC-133A.

(d) Resistance Measurements

Resistance is the characteristic of the substance to repel the current flow. Thus, when the resistance increases, the signal might have a distorted amplitude, or the signals might not display defined ECG peaks. As observed in Figure 5.7 the values of resistances are decreasing as the concentrations of nanocomposites are increasing. This graph depicts an appropriate trend as the resistance decreases with a decrease in concentration. It was measured using ESCORT ELC-133A.

(e) Impedance of the Fabricated Electrodes

In the dry electrodes, there are capacitive elements present that play an important role when conductors are considered in the AC circuit. The impedance should be low as high values of impedance leads to weak ECG signals and signal distortion. The same trend can be seen in Figure 5.7, which is a positive sign. It was measured using ESCORT ELC-133A.

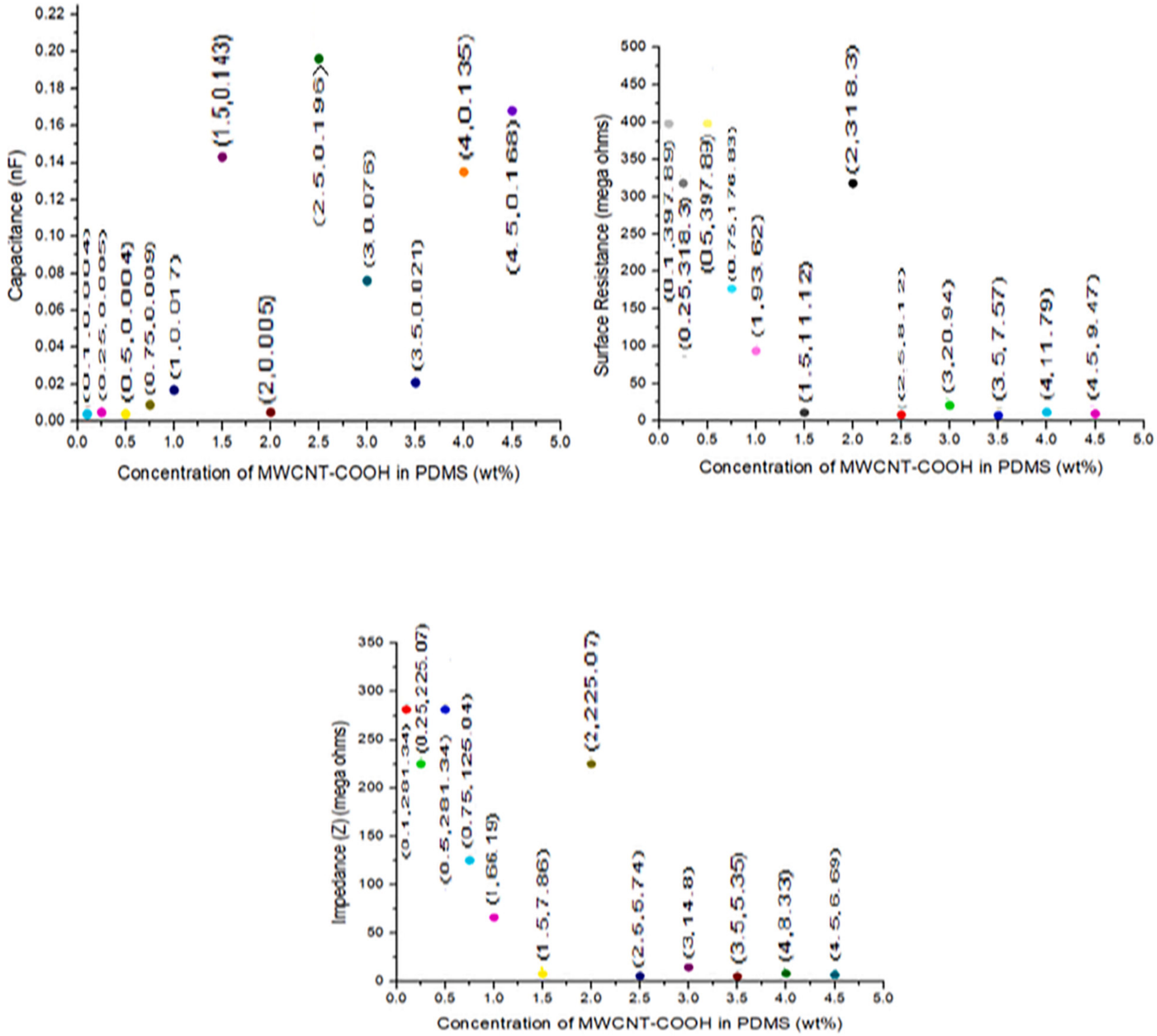


Figure 5.7: Capacitance, Surface resistance, and Impedance vs concentration

(f) Comparison with Ag/AgCl Electrodes

Figure 5.8 shows the ECG waveforms of the fabricated thirteen electrodes. The ECG waveforms obtained from the electrodes with lower concentrations of MWCNT-COOH are not satisfactory. Good ECG waveforms were depicted from a concentration of 2.5wt% (capacitance value: 0.196 nF). At 3wt% concentration, the ECG peaks are not defined and the ECG wave is shifted from the axis. The best ECG waveform is seen at 4wt%, and is comparable to conventional Ag/AgCl electrodes, despite the values of resistance, capacitance, and impedance values of 4.5wt% concentration of MWCNT-COOH being better.

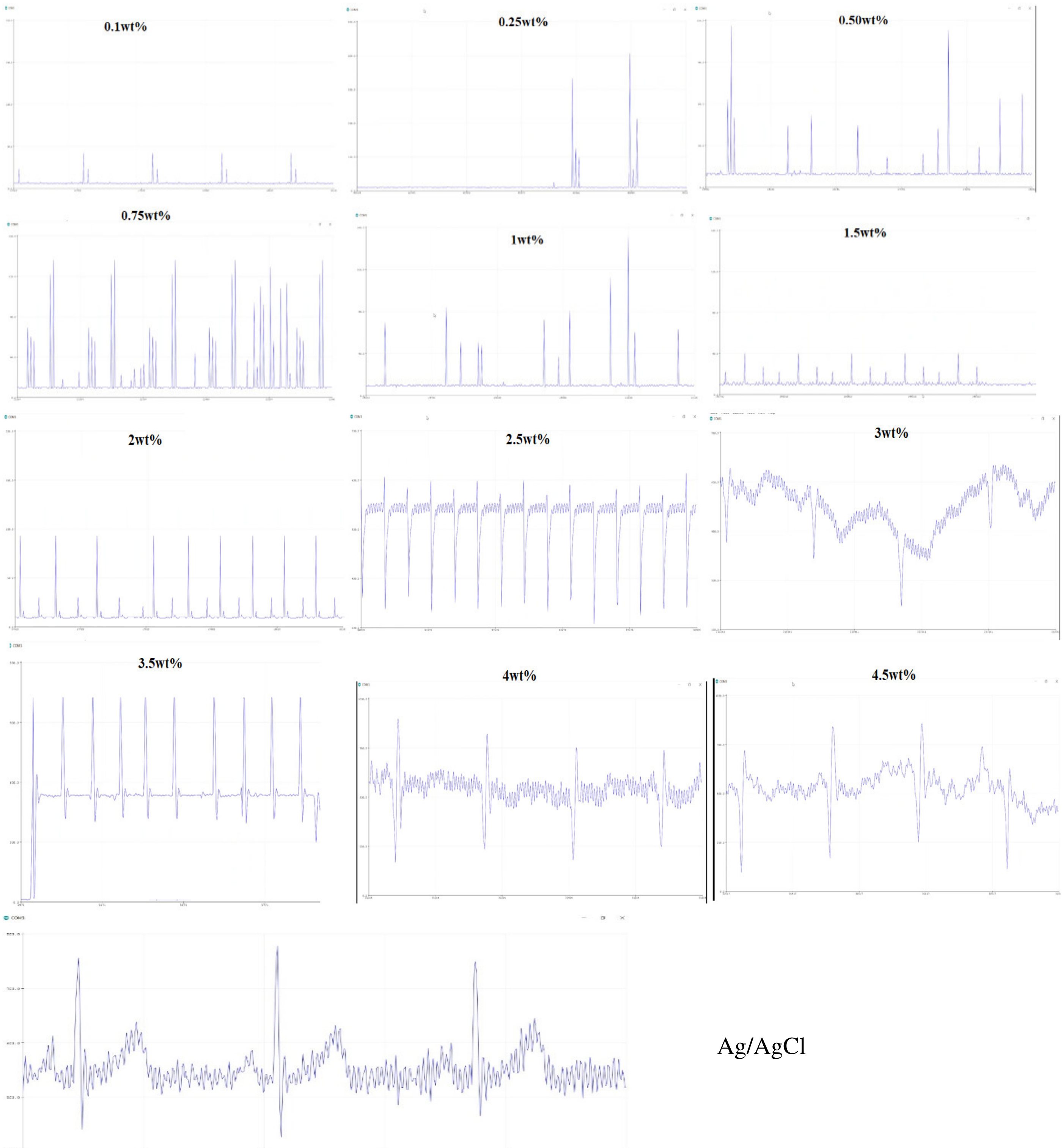


Figure 5.8 ECG waveform of various concentrations of electrodes

5.3 Comparison with Other Works

Various ECG sensors/electrodes have been designed and developed using pure MWCNTs, but less work has been done using the functionalized MWCNTs (-COOH group). Various works are described in Table 5.1 and compared with the present work done.

Table 5.1. Comparative analysis of the current work with other works

S.No	Ref.	Materials	Fabrication methodology/ technique	Nature of the sensor	Comparative analysis	IoT-enabled
1.	[52]	MWCNT-COOH/ PDMS	Metal-patterning (laser cutter)	Patch	-	No
2.	[33]	MWCNT, PDMS	Mold casting	Electrode	The obtained ECG waveform at 5wt% and 12h of ultrasonication had results comparable with the conventional ECG electrodes	No
3.	[47]	MWCNT, PDMS, MEP	Ultra-sonication and vacuum desiccation	Electrodes	ECG signals obtained were of good quality	No
4.	[58]	MWCNT	Micro-Electro-Mechanical Systems technique	Micro-pillars or Array electrodes	ECG signals were comparable to the conventional electrodes	No
5.	[166]	MWCNT, PDMS	Mold casting	Electrode	The electrode with 4.5wt% provided comparable results to the Ag/AgCl electrodes	No
6.	[40]	MWCNT, DI, PVP, Graphene and Silver conductive paint, and Xerox bond-paper	MWCNT electrodes were prepared with durapore membrane filters	Electrode	Comparable results with $AgCl_3$ electrodes	No
7.	[172]	Chemically-modified	Drop cast technique	Electrode	CG/f@MWCNTs-25% portrayed better	No

		graphene (CG) and Carboxylic-functionalized MWCNTs			results	
8.	[176]	MWCNT modified depletion type n-channel MOSFET-based electrophysiological active dry electrode	CMOS technology	Electrode	Good quality ECG signals were obtained	No
9.	[61]	MWCNT, Stainless Steel Thread, 3D Printed Fiber, Carbon Nanotube Fiber	Stitching into the fabric	Textile-based electrodes	Best results obtained from Carbon Nanotube Fiber	Bluetooth enabled transmission
10.	[44]	MWCNT, elastomeric sheets	Placing the CNT sheets on the surface of the elastomer	Elastomeric sheets	Decent ECG signals achieved	No
11.	[48]	MWCNT, PDMS	Spin coating	Electrodes	Provided an accuracy of 94% and the signal amplitude was better than PDMS and nickel-based electrodes	No
12.	[52]	MWCNT, PDMS	Mold casting	Electrodes	Electrodes above 4wt% CNT concentration had results comparable to Ag/AgCl electrodes	No
13.	[49]	MWCNT, Silbione, and Polytetrafluoroethylene	Spin coating	Electrodes	Good ECG results are observed	Bluetooth enabled transmission
14.	[35]	MWCNT, Polyaniline, acrylic resin, and Silver	Polymerization	Electrode	-	No
15.	[164]	MWCNT,	Metal	Patch	ECG results obtained	No

		PDMS	patterning		are comparable with Ag/AgCl electrodes	
16.	[36]	MWCNT, PDMS, PET substrate, Ag ink	Bar coating and screen printing	Electrode	No significant differences in signal amplitudes of both the electrodes	No
17.	[42]	MWCNT, PDMS	Metal patterning	Patch	ECG waveform was better than the conventional electrodes	No
18.	[41]	MWCNT, PDMS, aPDMS, AgNWs	Using filter membrane	Electrode	Good ECG waveforms are obtained	Radio-Frequency enabled transmission
19.	[46]	MWCNT, Ag ink, PET substrate, PDMS	Bar coating	Electrode	Better results were obtained when compared with the Ag/AgCl electrodes	No
20.	[33]	MWCNT, PDMS, Ag ink, PET substrate	Bar coating	Electrode	Electrode with the largest surface area exhibited the best results	No
21.	[34]	MWCNT, Cotton, tapioca starch	A paste of MWCNT and tapioca starch was applied to the cotton fabric	Textile based	ECG results were comparable to the commercial electrodes	No
22.	[35]	MWCNT, acrylic paint	Screen printing	Electrode	-	No
23.	[39]	MWCNT, Conductive Dry Adhesives	Spin coating	Pin - shaped Electrode	The signal obtained is almost similar to the 3M ECG electrode systems	No
24.	[50]	MWCNT, PDMS, Graphene, DMF	Spin coating	Electrode	From all the electrodes, PDMS with MWCNT depicted better results	No
25.	[43]	MWCNT, PDMS, AgNPs	Doctor blade technique	Electrode	Signal amplitude with 0.6mm thickness is higher than 0.4mm electrodes. The electrodes with a higher concentration of CNTs has better	No

					results	
26.	[45]	MWCNT, PDMS, PEIE, PET (substrate)	Printing techniques	A device with an ECG sensor, temperature sensor	Most clear peaks were observed for 7.5wt%	No
27.	[32]	MWCNT, aPDMS	Spin coating	Electrode	ECG waveform is comparable to the conventional ECG electrodes	Bluetooth enabled transmission
28.	[31]	MWCNT, PDMS,	Spin coating	Electrode	Fabricated electrodes with 1.5wt% concentration were comparable with the Ag/AgCl electrodes	No
29.	[51]	MWCNT, aPDMS	Metal patterning	Electrode	ECG waveform was comparable to the Ag/AgCl electrodes	No
30.	[38]	MWCNT, PDMS, aPDMS and AgNPs	Doctor blade technique	Electrode	The amplitude of the fabricated electrodes was slightly lower than Ag/AgCl electrodes	Radio-Frequency enabled transmission
31.	[57]	MWCNT, pvCNT, stainless steel foil substrate, polypyrrole	CNT/Pillars Arrays	Pin shaped Electrode	-	No
32.	[56]	MWCNT, pvCNT, stainless steel foil substrate	CNT/Pillars Arrays	Pin shaped Electrode	-	No
33.	This work	MWCNT-COOH/PDMS	Mould-formation	Film (circular)	4wt% MWCNT-COOH in PDMS had results comparable to the Ag/AgCl electrodes	Yes

5.4 Fabrication of SWCNT-COOH/PDMS Electrodes

5.4.1 Materials

Figure 5.9 depicts the various materials used for fabrication. **SWCNT-COOH** were acquired from **NanoResearch Elements**. It has better-dispersing abilities than unfunctionalized CNTs.

Figure 5.9: Materials used for fabrication

As functional groups are attached it creates spaces between the CNTs, which causes fewer agglomerations. Functionalized CNTs improve better interfacial bonding between the PDMS and the CNT. This bonding also enhances the conductivity because of better interfacial bonding. The polymer for this research was **PDMS Sylgard 184 Silicone Elastomer from Dow Corning** due to its impressive properties including flexibility, non-toxicity, and biocompatibility. A good solvent is required for dispersing the CNTs as the materials agglomerates very soon. Therefore, **IPA** was selected as both CNTs and PDMS are soluble in IPA. The specifications are mentioned in Table 5.2.

Table 5.2: Specifications of SWCNT-COOH

S.No	**Parameter**	**Value**
1.	Outer diameter	1-4 nm
2.	Inner diameter	0.8-1.6 nm
3.	Length	5-30 µm
4.	Specific Surface Area	690 m^2/g
5.	Electrical conductivity	>100 S/cm

5.4.2 SWCNT-COOH/PDMS Composite Formation

(a) Distribution of SWCNT-COOH in IPA

To attain excellent conductivity, CNTs must be dispersed efficiently. Appropriate dispersion leads to the proper formation of conductive networks, which leads to satisfactory ECG results. Before dispersing the SWCNT-COOH with the help of an 'ultra-sonicator', it is first distributed using a 'magnetic stirrer'. SWCNT-COOH and IPA were stirred with the help of a REMI 5MLH magnetic stirrer @ 300 RPM for 10 minutes. Thirteen electrodes were developed with varied concentrations as follows: 0.1wt%, 0.25wt%, 0.5wt%, 0.75wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt%, 3wt%, 3.5wt%, 4wt%, 4.5wt%, and 5wt%.

(b) Dispersion of SWCNT-COOH with IPA

The big chunks (if any) are broken down into smaller particles, but not fully untangled. Therefore, to fully break down the SWCNTs and brings them into a clear solution form, it is necessary to sonicate the above solution. This is because the stirrer mixes the solution, but the sonicator with rigorous agitation blends the solution. The various compositions of SWCNT-COOH with IPA were sonicated using a probe sonicator, Hielscher UP400St. The sonication was done for 30 minutes with 34W with an amplitude of 44%. The sonication was done only for 30 mins, as to conserve the properties and characteristics of the material. With the help of this sonication process, no aggregates were seen.

(c) Dispersion of SWCNT-COOH with PDMS

To bring this solution to the final form of an electrode and to induce flexibility, PDMS is mixed with it. The PDMS base was dispersed to the above solution with the probe sonicator for 10 minutes, with an amplitude of 58% and 34W. Later, PDMS curing agent was dispersed to the above solution. This is used to cure the solution and provide hardness to the solution. The last dispersion was done for 5 minutes with an amplitude of 56% within 31W power as shown in Figure 5.10.

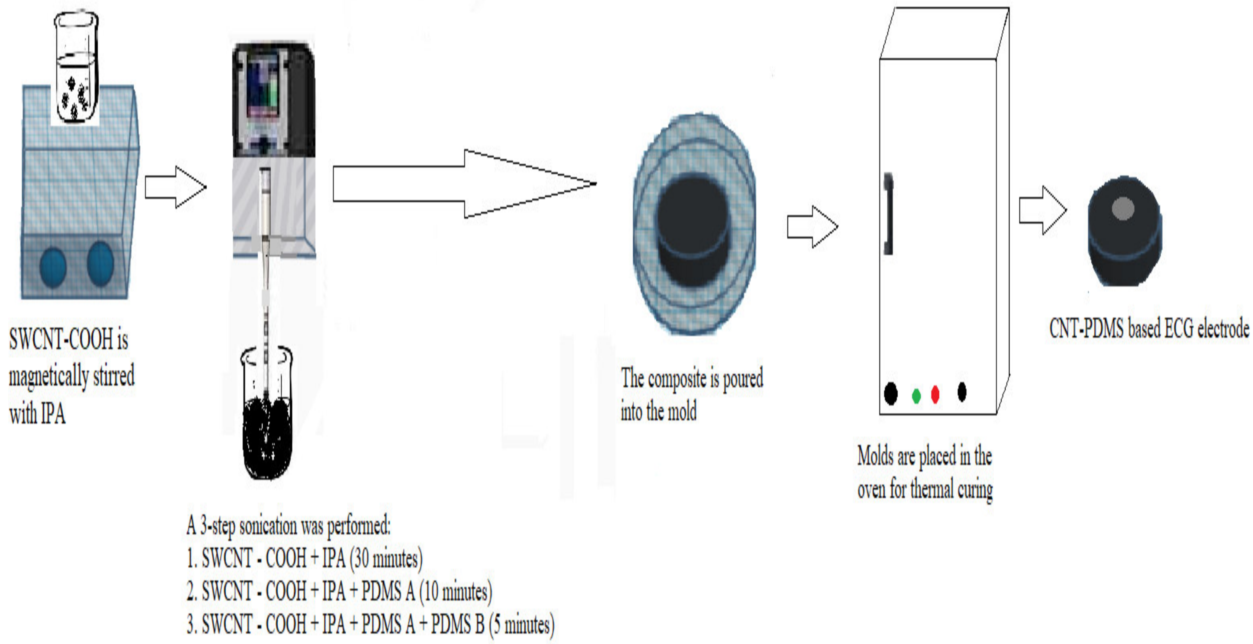


Figure 5.10: Electrode Fabrication process

(d) Final Fabrication of Electrodes

The final step is to pour the prepared nanocomposites as depicted in Figure 5.11 into the molds and place them in the oven for drying and curing purposes.

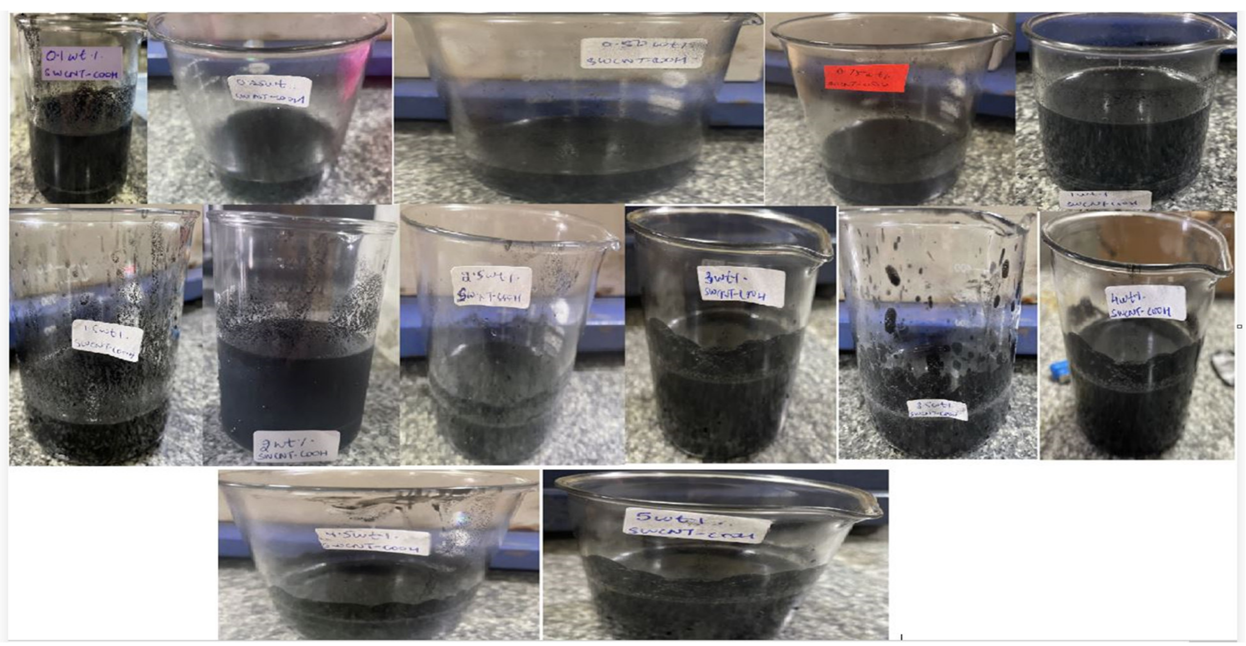


Figure 5.11: Various composite concentrations of SWCNT-COOH/PDMS

The temperature for curing was 100ºC and was kept constant throughout the process. Different concentrations of SWCNT-COOH/PDMS composite solutions needed dissimilar durations in the oven, ranging from 2-10 hours. When the nano-composite dried, the circular films were demoulded from the molds. Finally, the aluminum conductive snaps were pasted on top of the electrodes with the help of conductive Ten20 paste as shown in Figure 5.12.

Figure 5.12: Electrodes of various concentrations of SWCNT-COOH/PDM

5.5 Results

(a) Physical Characterization

SWCNT-COOH was examined as a solid residue with the help of SEM in Figure 5.13, TEM in Figure 5.14, and FTIR in Figure 5.15. TEM provides indications of interior assemblies of the material. The concentration of -COOH in SWCNT is in the range of 2-4wt%. The -COOH functional groups in the SWCNT separate the CNT bundles, which further improved the disintegration and solubility. SEM as seen in Figure 5.13 is done for information on composition, morphology, and topography. It is used to observe any sort of surface contaminations and fractures. TEM gives information on the internal structure of the substance. FTIR was carried out to identify the chemical bonds, and to provide evidence of the covalent bonding of the CNTs. FITR is achieved in the range of 500-3500cm^{-1}. Figure 5.15 (graph) offers evidence of the wave-absorption by the -COOH group. One sharp peak (the dip in the graph is called a peak) can be seen at 3426cm^{-1} which implements the strongest bond at this point.

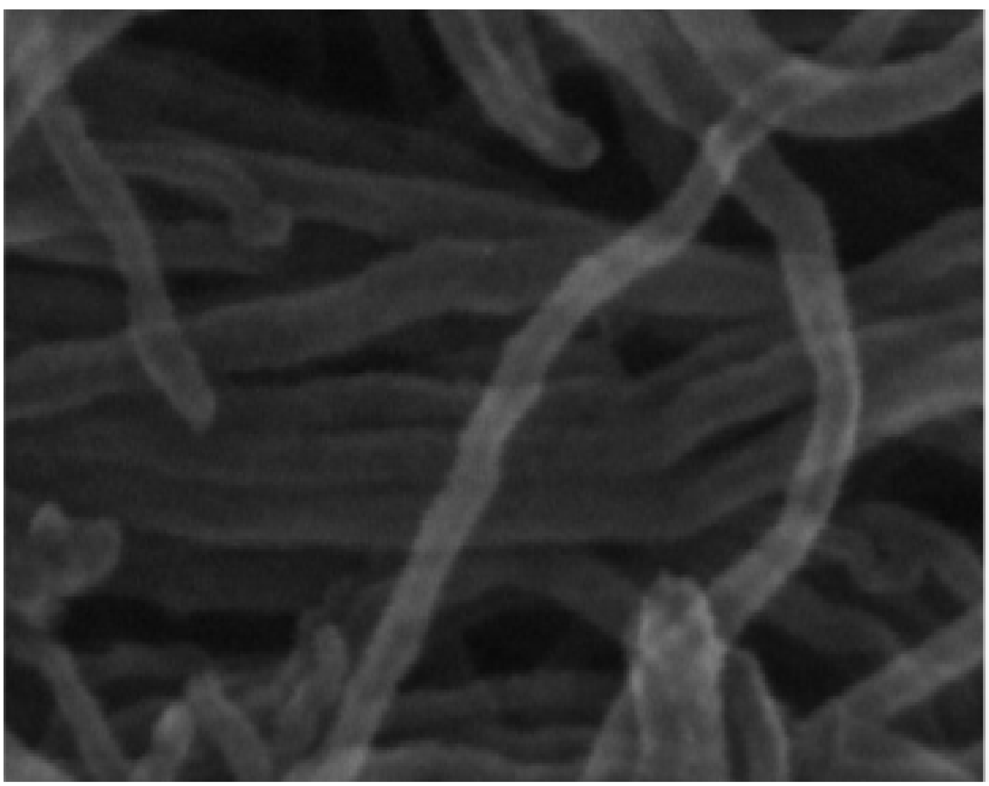


Figure 5.13: SEM of SWCNT-COOH

Figure 5.14: TEM of SWCNT-COOH

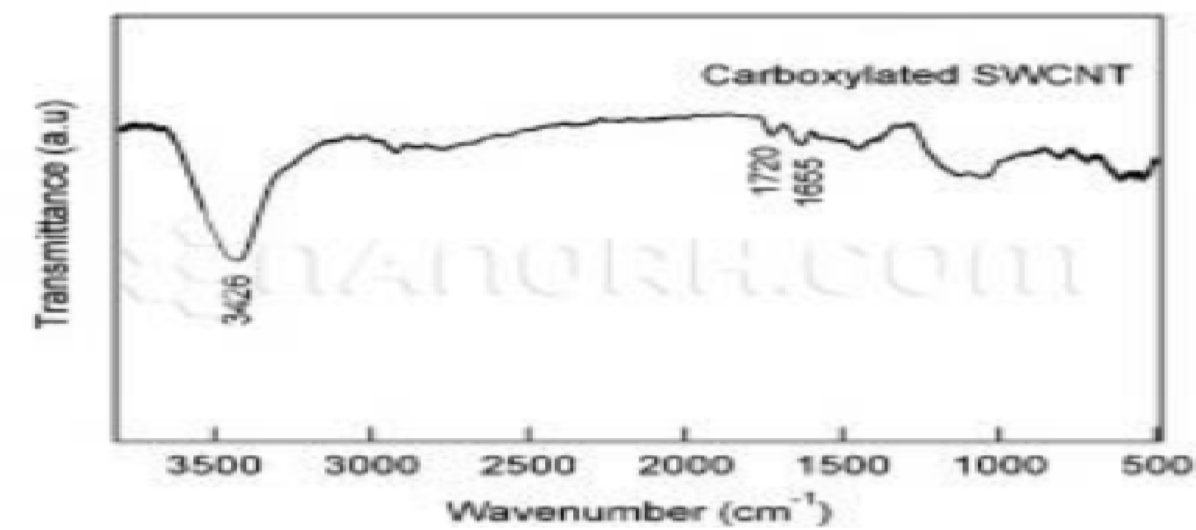


Figure 5.15: FTIR of SWCNT-COOH

5.6 ECG Measurements and Analysis

(a) Long-Term ECG Monitoring

All the electrodes were worn for 5 days for checking their long-term compatibility of the electrodes. The results on the 3rd and 5th day were a little degraded but after cleansing with methanol, the results revived. The electrodes were safe to wear, and no skin irritation or allergy was identified.

(b) Capacitance Measurements

Capacitance can be defined as an electrical property that offers information on the flow of electrons in a specific conductor. As observed in Figure 5.16, there is an increase in the value of capacitance with an increase in the concentration of SWCNT-COOH in the nanocomposite, which is a good trend. As there will be more flow of electrons, collisions will be less and will enhance the conductivity in the electrodes. It was measured using ESCORT ELC-133A.

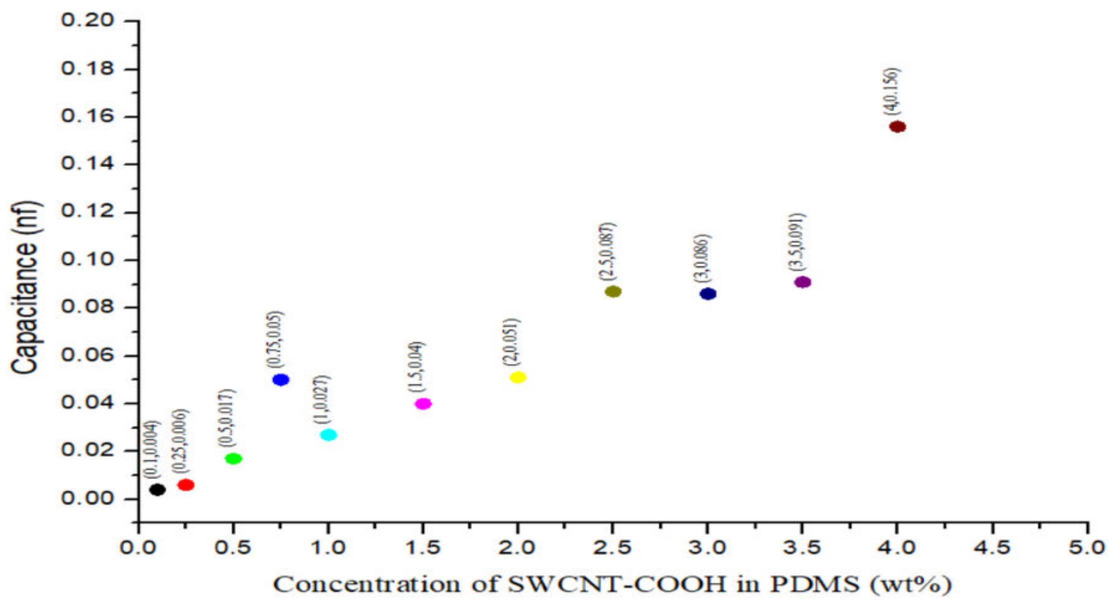


Figure 5.16: Capacitance vs concentration of SWCNT-COOH in PDMS

(c) Resistance Measurements

Resistance is the characteristic of the substance to repel the current flow. Thus, when the resistance increases, the signal might have a distorted amplitude, or the signals might not display defined ECG peaks. As observed in Figure 5.17, the values of resistances are decreasing as the concentrations of nanocomposites are increasing. This graph depicts an appropriate trend as the resistance decreases while the concentration decreases. It was measured using ESCORT ELC-133A.

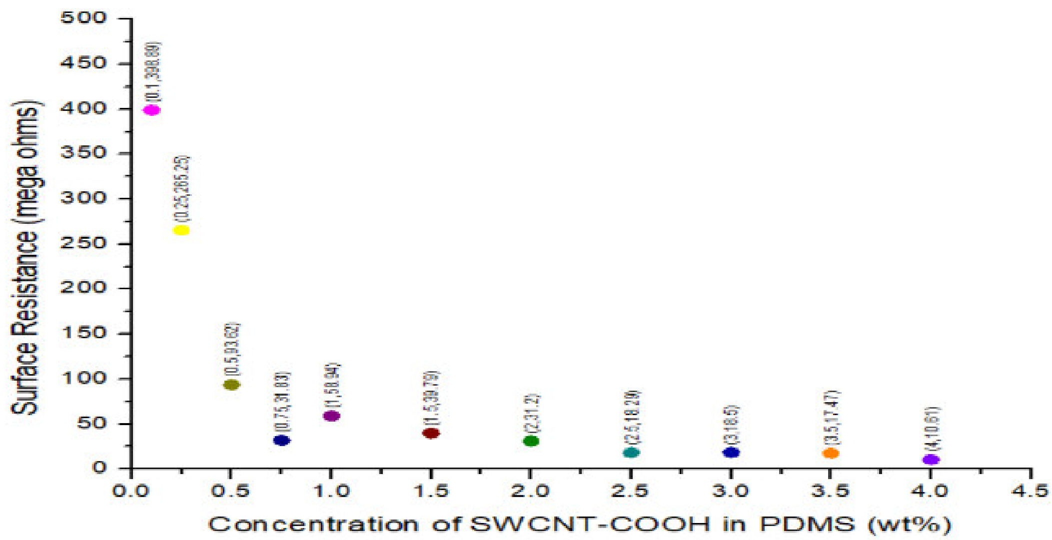


Figure 5.17: Resistance vs concentration of SWCNT-COOH in PDMS

(d) Impedance of the Fabricated Electrodes

In the dry electrodes, there are capacitive elements present that play an important role when conductors are considered in the AC circuit. The impedance should be low as high values of impedance leads to weak ECG signals and signal distortion. The same trend can be seen in Figure 5.18, which is a positive sign. It was measured using ESCORT ELC-133A.

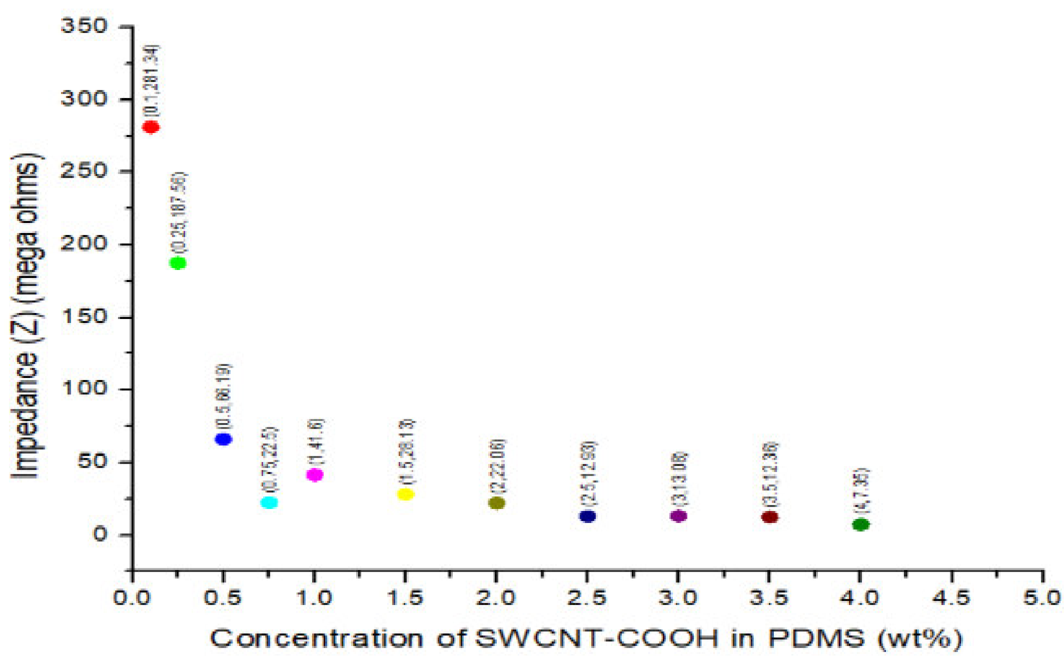


Figure 5.18: Impedance vs concentration of SWCNT-COOH in PDMS

(e) Comparison with Ag/AgCl Electrodes

Figure 5.19 shows the ECG waveforms of the fabricated thirteen electrodes. The ECG waveforms obtained from the electrodes with lower concentrations of SWCNT-COOH do not offer satisfactory ECG waveforms. Good ECG waveforms were depicted from a concentration of 3.5wt%. The best ECG waveform is seen at 3.5wt% and is comparable to conventional Ag/AgCl electrodes.

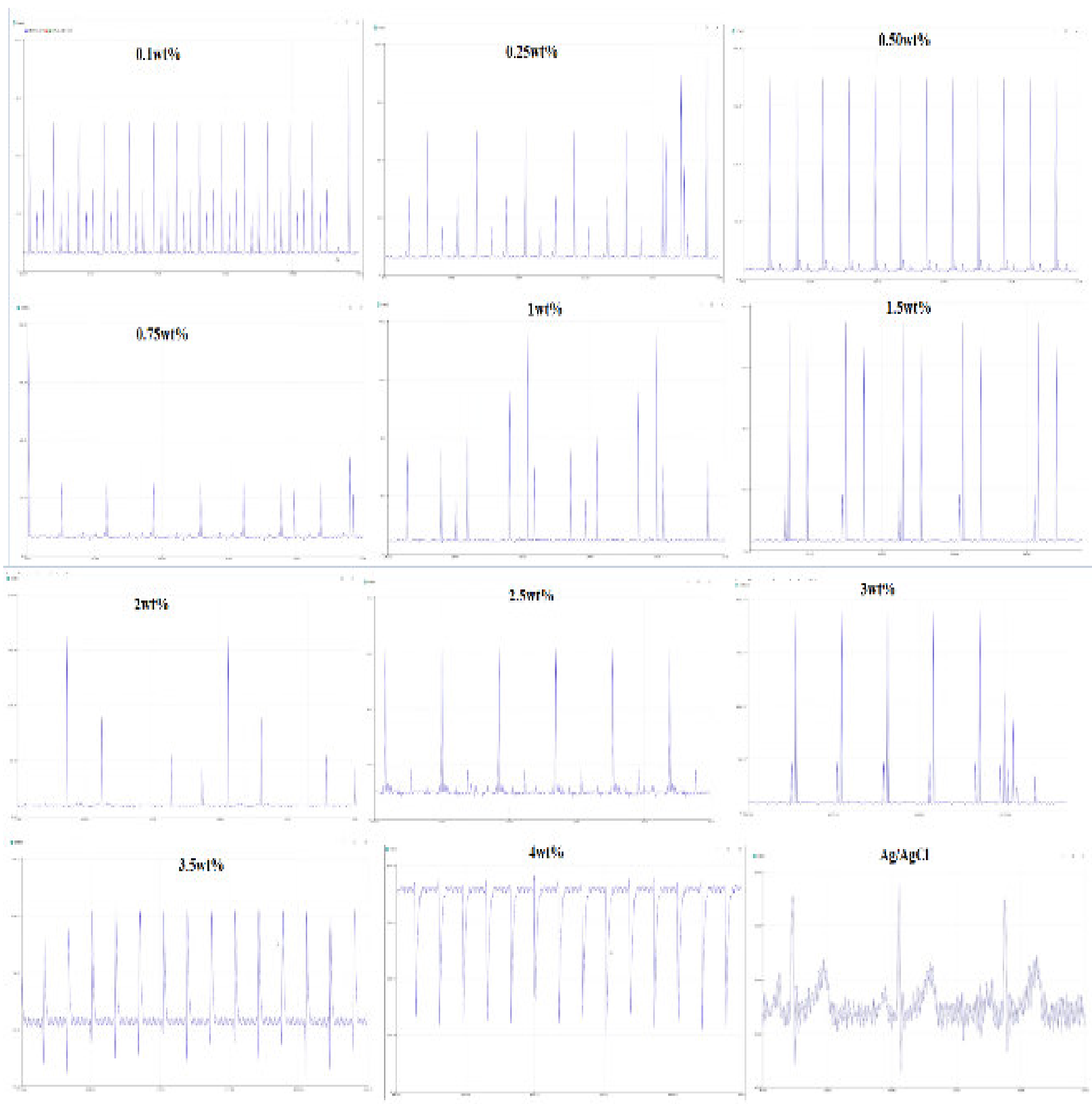


Figure 5.19: ECG results obtained from the ECG tracking system

5.7 Comparison with Other Works

In this segment of the thesis, a comparison is presented with other works in Table 5.3.

Table 5.3: Comparative analysis of the current work with other works

S. No	Ref.	Materials used	Fabrication technique	Nature of sensor	Comparative results	IoT-enabled
1.	[177]	SWCNT, Poly (pyrrole-co-pyrrolepropylic acid) with pendant carboxyl groups	Electrochemical process	Electrode	ECG was not measured	No
2.	[20]	SWCNT, DNA, and Chitosan	Membrane filtering technique	Freestanding films	ECG was not measured but it was mentioned that these will work well as ECG pads.	No
3.	[28]	SWCNT, Electrically Conductive Fibers, regioregular poly(3hexylthiophene)	Dipping and drying	Electrode	Moderate ECG was achieved	No
4.	[27]	SWCNT, Hydrogel	Doctor blade technique	Film	ECG results were better than the conventional electrodes	No
5.	[29]	SWCNT, Cotton Yarn (CY)	Dipping and drying	Textile-based electrode	ECG waveforms measured by 4-strand, and 6-strand SWNT-CYs are comparable to the standard ECG waveforms measured by the conventional lead wires.	No
6.	[30]	SWCNT, Silver Nanowires, and Polyutherene nanoweb	Bar coating	Film	There was no substantial alteration in the quality of the ECG signal	No
7.	[23]	SWCNT, and	Inkjet	Film	ECG was not measured,	No

		TFT (flexible substrate)	printing		but the authors mentioned there is a scope for online ECG measurements.	
8.	[21]	SWCNT, SSDNA	CMOS technology	Chip	Appropriate ECG signals were not visible.	No
9.	[174]	SWCNT, Poly (vinylidene fluoride-cohexafluoro propylene)	Blow spinning technique, inkjet printing	Fabric-based sensor	Decent ECG signals were achieved	No
10.	This work	SWCNT-COOH, PDMS	Mold casting	Film (circular)	3.5wt% has the closest peaks as Ag/AgCl electrodes	Yes

5.8 Conclusion

v. MWCNT-COOH/PDMS

1. The percolation threshold was attained at 4.5wt%.
2. The finest ECG results were seen at 4wt% and the ECG waveform was comparable to the conventional Ag/AgCl electrodes.
3. The fabricated ECG electrodes proved to be good for remote, continuous, and long-term patient monitoring. As there were no signs of allergy or irritations on the skin even after 5 days of wearing the electrodes.

vi. SWCNT-COOH/PDMS

1. The percolation threshold was achieved at 4wt%, also the electrodes were a bit brittle.
2. The finest results were witnessed at 3.5wt% and were comparable to the conventional electrodes.
3. The developed electrodes were appropriate for longstanding and distant ECG monitoring since the electrodes were bio-compatible and no skin allergy or irritation was observed even after 5 days of wearing the electrodes.

Table 5.4. Comparison between MWCNT-COOH/PDMS and SWCNT-COOH/PDMS-based ECG electrodes

S.No	Parameter	MWCNT-COOH/PDMS	SWCNT-COOH/PDMS
1.	Best ECG waveform	4wt%	3.5wt%
2.	Capacitance (nF)	0.135	0.091
3.	Surface Resistance (MΩ)	11.79	17.47
4.	Impedance (MΩ)	8.33	5.35

As per Table 5.4, the electrode which depicted the best result and is closer to the conventional electrodes has a 3.5wt% concentration with SWCNT-COOH/PDMS. The peaks were well defined when compared with 4wt% MWCNT-COOH/PDMS electrodes. Better waveforms are achieved with less concentration of the nanomaterial.

CHAPTER 6

Fabrication of ECG Electrodes - Fabrication Technique II

6.1 INTRODUCTION

In this chapter, Graphene/PDMS, Graphene/SWCNT-COOH/PDMS, Graphene/MWCNT-COOH/PDMS, Graphene/SWCNT-COOH/MWCNT-COOH/PDMS, and SWCNT-COOH/MWCNT-COOH/PDMS ECG electrodes are fabricated using Fabrication Technique II. This technique includes mold-casting, dispersion of nanomaterials in IPA, dispersion of nanomaterials with PDMS, and placing the solvents in the molds. The types of equipment included a bath sonicator and an oven. The ECG was measured using the system discussed in Chapter 3.

6.2 Fabrication Process of Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS (G-II), and SWCNT-COOH/MWCNT-COOH/PDMS (G-III)

6.2.1 Materials

Graphene was purchased from Thermo-Fischer Scientific. SWCNT-COOH, and MWCNT-COOH from purchased from NanoResearch Elements. PDMS was purchased from Dow Corning, and IPA was purchased from Loba Chemie as shown in Figure 6.1.

Figure 6.1: Materials used for fabrication

6.2.2 Preparation of Nanocomposites

(a) Dispersion of Graphene /SWCNT-COOH/ MWCNT-COOH with IPA

It is of utmost importance that there is proper dispersion of nanomaterials for achieving excellent conductivity. First, equal parts of the three nanomaterials with IPA were sonicated using the ‘bath-sonicator’ from PCI analytics for 30 minutes @ 50Hz with 230 volts. Thirteen solutions from 0.1wt%-5wt% were prepared as shown in Figure 6.2.

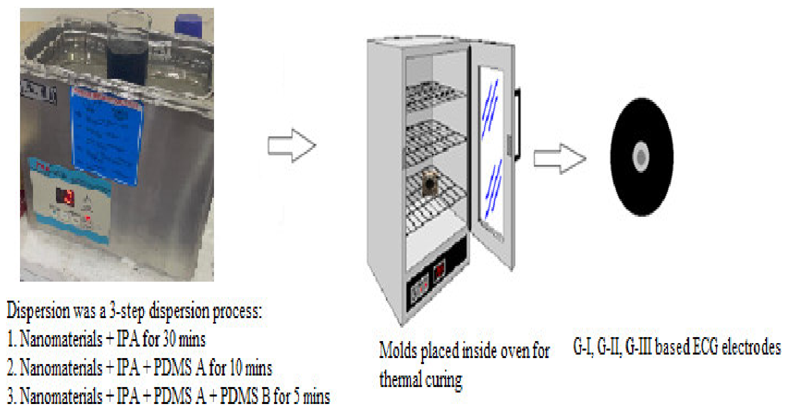


Figure 6.2: Electrode fabrication process

(b) Dispersion of Graphene in IPA

Thirteen solutions from 0.1wt%-5wt% of Graphene and IPA as described in section 6.2.2 (a).

(c) Dispersion of SWCNT-COOH/MWCNT-COOH in IPA

Equal parts of the two nanomaterials with IPA were sonicated using the ‘bath-sonicator’ from PCI analytics for 30 minutes @ 50Hz with 230 volts. Thirteen solutions from 0.1wt%-5wt% were prepared.

(d) Dispersion of Nanomaterials/IPA Solutions Prepared in 6.2.2 (a), 6.2.2 (b), and 6.2.2 (c), with PDMS

After the nanomaterials were sonicated properly with IPA, PDMS was added. Initially, PDMS-base was added first and sonicated for 10 mins @ 50Hz with 230V. Later, PDMS cross-linking agent is added to harden the nanocomposite @ 50Hz with 230 volts. This is depicted in Figure

6.3, Figure 6.4, and Figure 6.5. Lastly, the nanocomposites were poured into the molds and thermally cured in the oven for 2-6 hours, depending on the various concentrations of the nanomaterials at 100°C. This is depicted in Figure 6.6, Figure 6.7, and Figure 6.8.

Figure 6.3: Graphene/MWCNT-COOH/SWCNT-COOH/PDMS nanocomposites

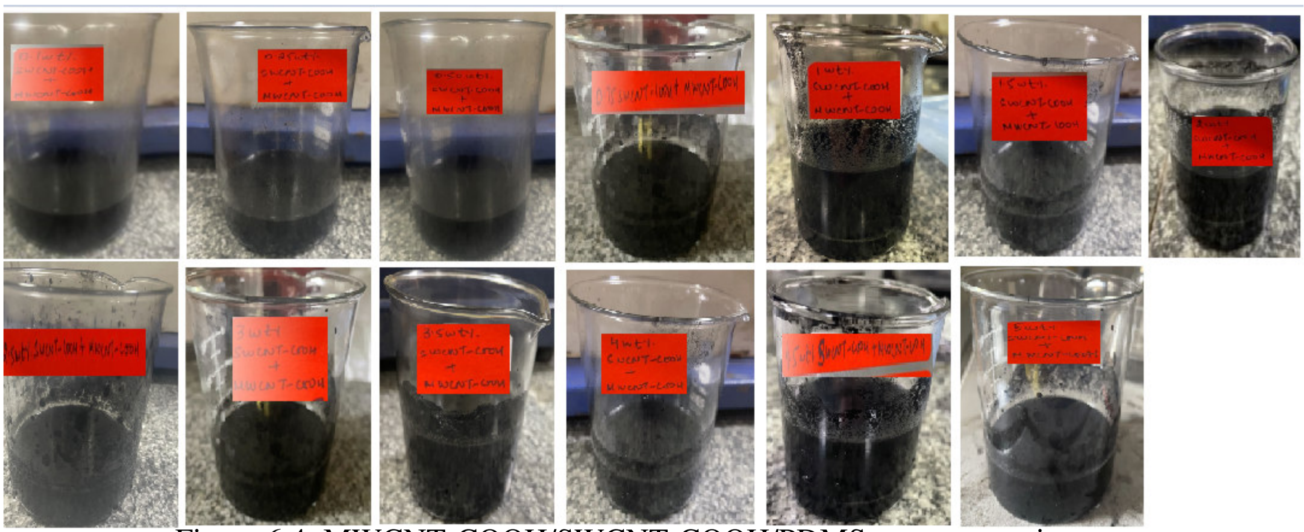


Figure 6.4: MWCNT-COOH/SWCNT-COOH/PDMS nanocomposites

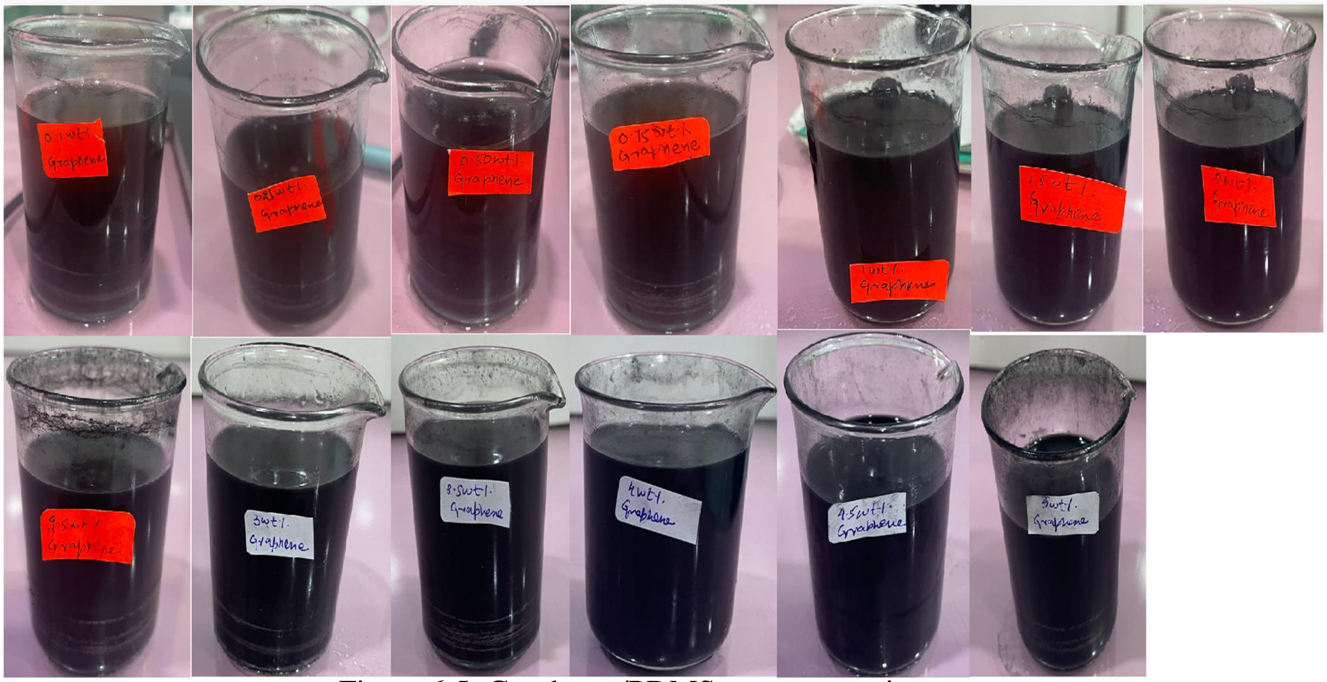


Figure 6.5: Graphene /PDMS nanocomposite

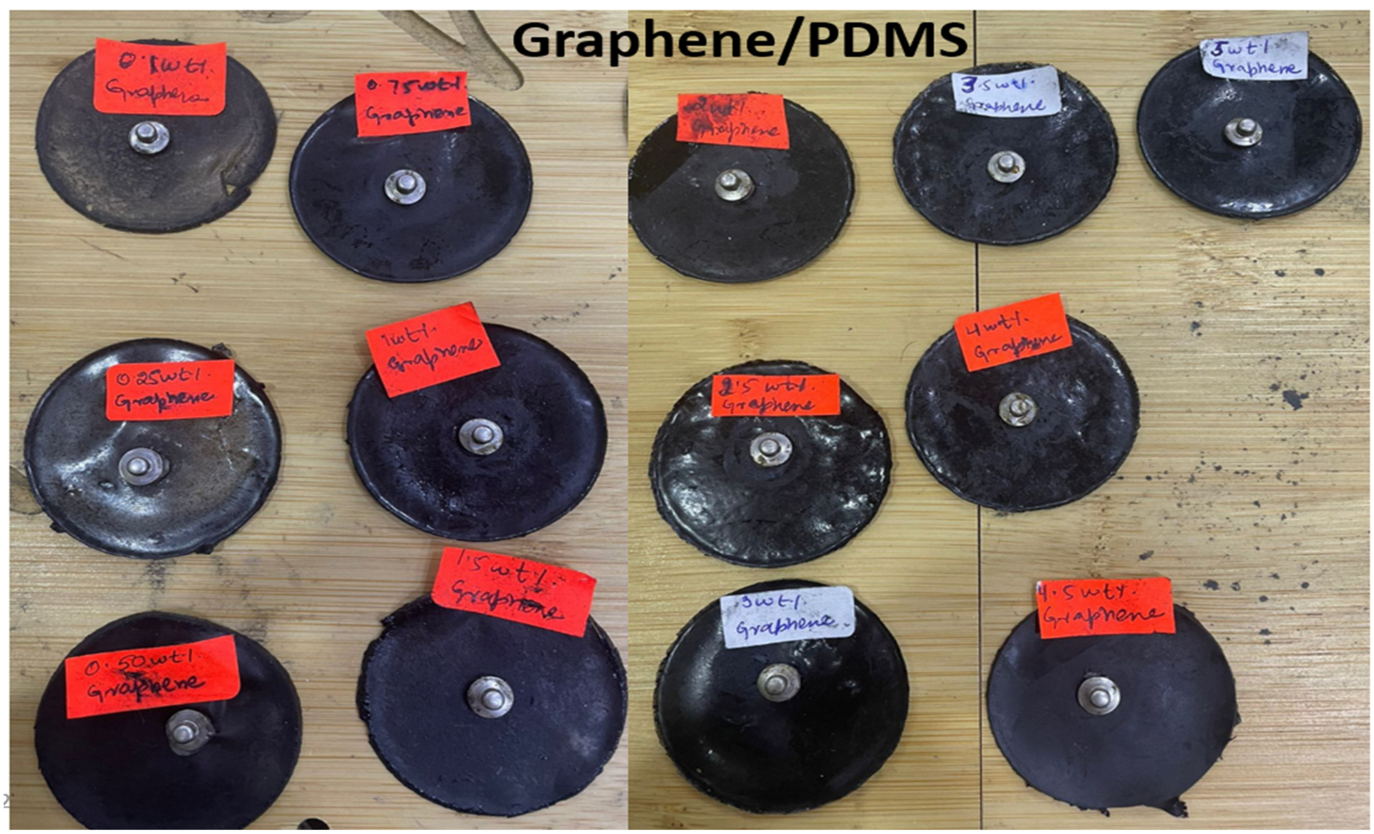


Figure 6.6: Fabricated Electrodes: Graphene/PDMS

Figure 6.7: Fabricated Electrodes: Graphene/SWCNT-COOH/MWCNT-COOH/PDMS

Figure 6.8: Fabricated Electrodes: SWCNT-COOH/MWCNT-COOH/PDMS

6.2.3 ECG Results and Evaluation

(a) Long-term ECG Monitoring

All the electrodes were worn for 5 days for checking their long-term compatibility of the electrodes. The results on the 3rd and 5th day were a little degraded but after cleansing with methanol, the results revived. The electrodes were safe to wear, and no skin irritation or allergy was identified.

(b) Capacitance of the ECG Electrodes

ESCORT ELC-133A was used to measure the capacitance. A graph has been plotted between concentrations of all the thirty-nine electrodes versus capacitances. The capacitances of Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS (G-II), and SWCNT-COOH/MWCNT-COOH/PDMS (G-III) is depicted in Figure 6.9 (a). For G-I the capacitance is in the range 0.012-0.0176nF and the trend is that the capacitance is increasing but not at a faster rate, the largest value is seen at 5wt%. For G-II, the capacitance is increasing from 0.042-1.509nF for different concentrations and the largest value is seen at 4wt%. For G-III, the capacitance is increasing in the range 0.004-0.341nF, and 4wt% had the highest value. When all the electrodes were compared, the best value was achieved at 1.509nF for G-II electrodes at 4wt%.

(c) Resistance of the ECG Electrodes

ESCORT ELC-133A was used to measure the resistance. A graph has been plotted between concentrations of all the thirty-nine electrodes versus resistance. The resistance of Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS (G-II), and SWCNT-COOH/MWCNT-COOH/PDMS (G-III) are depicted in Figure 6.9 (b). For G-I the resistance is in the range 5.6-0.0162MΩ and the trend is that the resistance is decreasing but it is almost constant, ~2MΩ, and the lowest value is seen at 2.5wt%. For G-II the resistance is decreasing from 3.485-0.026MΩ for different concentrations and the lowest value is seen at 3wt%. For G-III, the resistance is in the range 397.89-4.67MΩ, and 4.5wt% had the lowest value, but G-III had the highest value of resistances.

(d) Impedance of the ECG Electrodes

ESCORT ELC-133A was used to measure the impedance. A graph has been plotted between concentrations of all the thirty-nine electrodes versus impedance. The impedance of Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS (G-II), and SWCNT-COOH/MWCNT-COOH/PDMS (G-III) are depicted in Figure 6.9 (c). For G-I the impedance is in the range 17-0.161MΩ and the trend is that the impedance is decreasing but it is almost constant, and the lowest value is seen at 2.5wt%. For G-II the impedance is decreasing from 3.4709-0.026 MΩ for different concentrations and the lowest value is seen at 3wt%. For G-III, the impedance is in the range 281.34-3.3MΩ, and 4.5wt% had the lowest value.

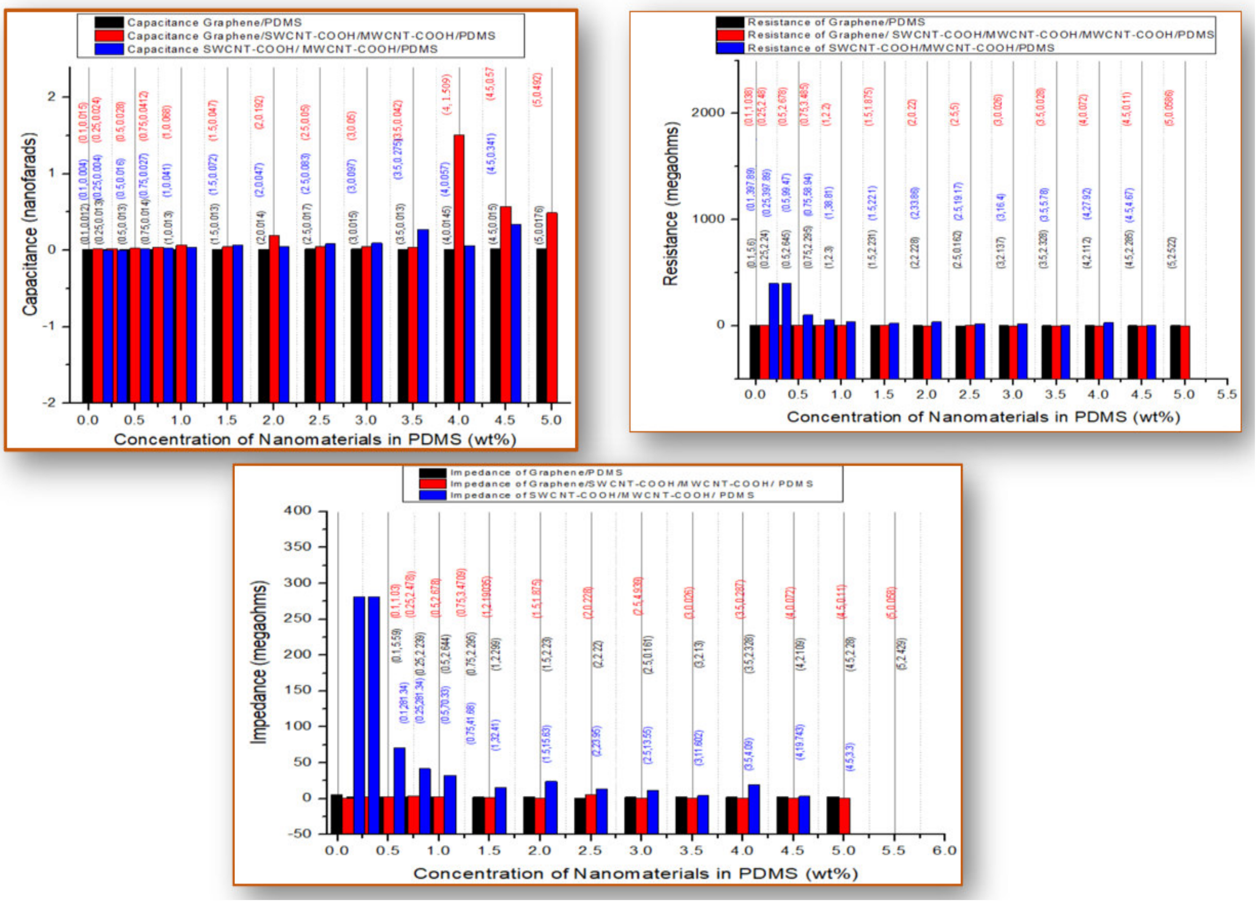
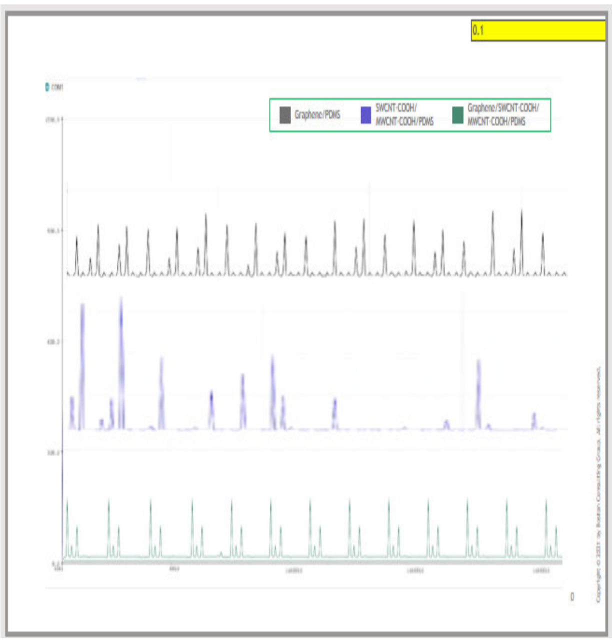
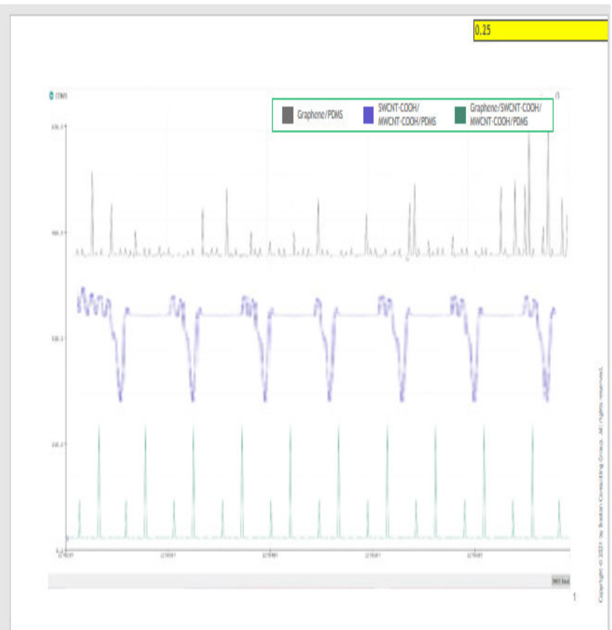


Figure 6.9: (a) Capacitance vs concentration of G -I, G-II, and G-III electrodes; (b) Resistance vs concentration of G-I, G-II, and G-III electrodes; (c) Impedance vs concentration of G-I, G-II, and G-III electrodes

(e) **Comparison of ECG Results of G-I, G-II, and G-III Electrodes**

Figure 6.10 shows the ECG waveforms of the fabricated thirty-nine electrodes as shown in Figure 6.6, Figure 6.7, and Figure 6.8. The ECG waveforms obtained from the electrodes with lower concentrations of nanomaterials do not offer satisfactory ECG signals for all the groups. Defined peaks were seen improving from 1wt%. For G-I electrodes, the ECG waveforms obtained were not up to the mark. The ECG waveforms were not even resembling the basic ECG waveform. For G-II electrodes, 0.1wt%, 0.25wt%, and 0.5wt% concentrations showed unacceptable ECG results. From 0.75wt% some improved ECG peaks were visible. For 1.5wt% and 2wt%, the ECG waveforms were clear and sharp. From 2.5wt%, the ECG waveforms started deviating from the baseline. Therefore, the best ECG waveform was observed at 2wt%. For G-III electrodes, good ECG results were seen from 1wt%, and the best ECG waveform was obtained at 3wt%.





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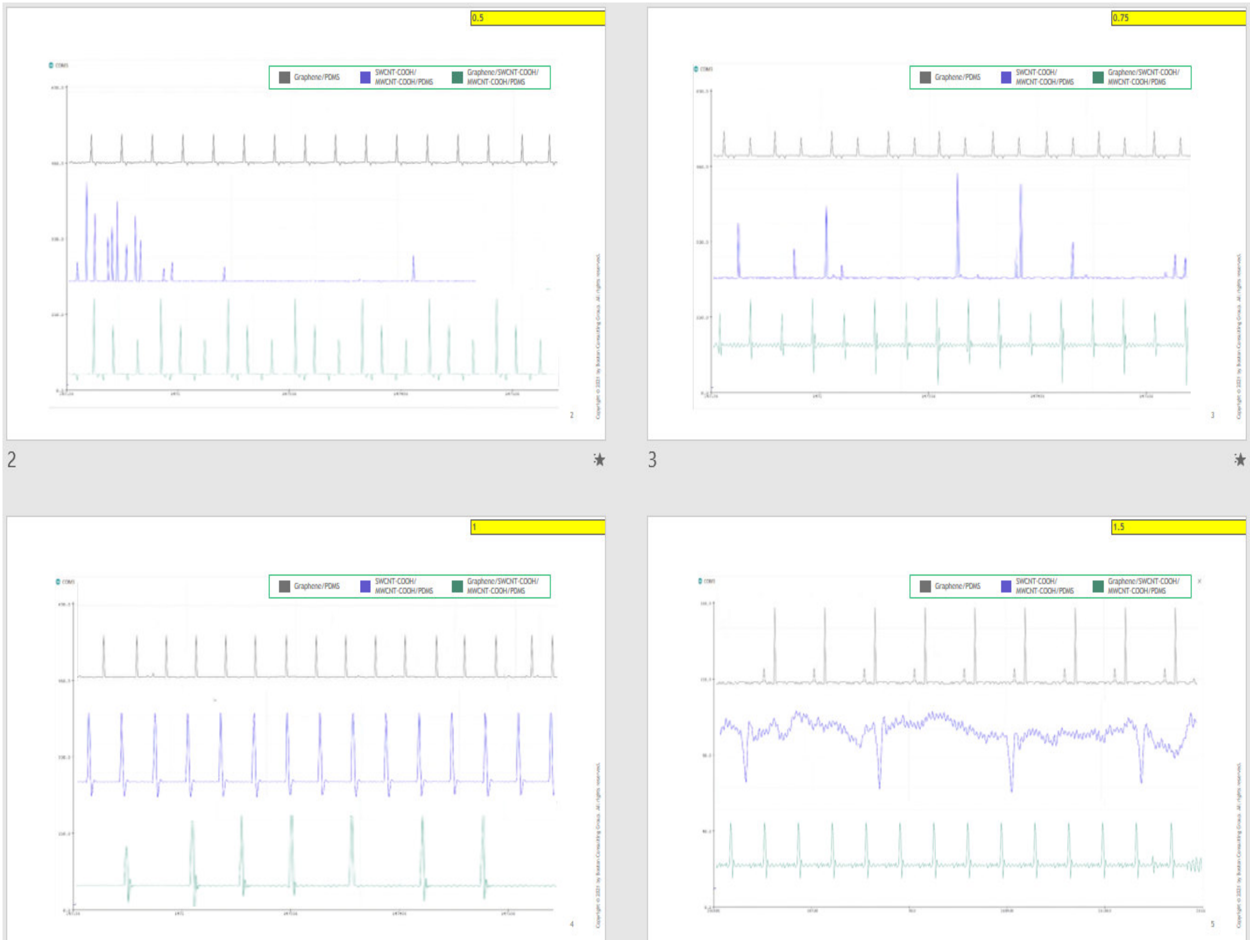

0.5
0.75
1
1.5
Graphene/PDMS
SWCNT-COOH/
MWCNT-COOH/PDMS
Graphene/SWCNT-COOH/
MWCNT-COOH/PDMS
2
3

Figure 6.10: ECG waveforms of fabricated electrode

6.2.4 Discussion

Table 6.1 represents a comparison with other similar works. In [133], textile-based sensors were fabricated with the help of acrylic ink, with varied formulations and sizes, using the Screen Printing technique, which is an expensive piece of equipment. Also, the system is not wirelessly enabled.

In this work, -COOH-based CNTs and Graphene have been used for fabricating the electrodes, which have not been reported yet. The fabrication method is economical and user-friendly. No bulky and expensive equipment is required since only a bath sonicator and oven are used for readily available fabrication. Also, the system is IoT-enabled.

Table 6.1 Comparison with other works

S.no	Ref	Materials	Fabrication technique/ methodology	Nature of the sensor	Comparative evaluation	IoT enabled
1.	[133]	SWCNT, MWCNT, acrylic composite	Screen-printing	Film (rectangular), textile-based	Best ECG results received for 3cm x 3cm electrode of the 1:1 formulation CNT-ACC electrodes	No
2.	This work	G-I	Mold casting	Film (circular)	No definite ECG waveform was observed	Yes
		G-II			2wt% had better results than Ag/AgCl electrodes G-I and G-III	
		G-III			3wt% electrodes had results comparable to Ag/AgCl electrodes	

6.3 Graphene/MWCNT-COOH/PDMS (G-IV), and Graphene/SWCNT-COOH/PDMS (G-V)

6.3.1 Materials

Graphene was purchased from Thermo-Fischer Scientific. SWCNT-COOH, and MWCNT-COOH were purchased from NanoResearch Elements. PDMS was purchased from Dow Corning, and IPA was purchased from Loba Chemie.

6.3.2 Preparation of Nanocomposites

(a) Dispersion of Graphene /MWCNT-COOH with IPA

Equal parts of the two nanomaterials with IPA were sonicated using the 'bath-sonicator' from PCI analytics for 30 minutes @ 50Hz with 230V. Thirteen solutions from 0.1wt%-5wt% were prepared.

(b) Dispersion of Graphene in IPA

Thirteen solutions from 0.1wt%-5wt% of Graphene and IPA as described in section 6.2.2 (a).

(c) Dispersion of SWCNT-COOH/MWCNT-COOH in IPA

Equal parts of the two nanomaterials with IPA were sonicated using the 'bath-sonicator' from PCI analytics for 30 minutes @ 50Hz with 230 volts. Thirteen solutions from 0.1wt%-5wt% were prepared.

(d) Dispersion of Nanomaterials/IPA Solutions Prepared in 6.3.2 (a), 6.3.2 (b), and 6.3.2 (c), with PDMS

After the nanomaterials were sonicated properly with IPA, PDMS was added. Initially, PDMS-base was added first added to the above solutions and sonicated for 10 mins @ 50Hz with 230 volts. Later, PDMS cross-linking agent is added to harden the nanocomposite @ 50Hz with 230 volts. The solutions can be seen in Figure 6.11, and Figure 6.12.

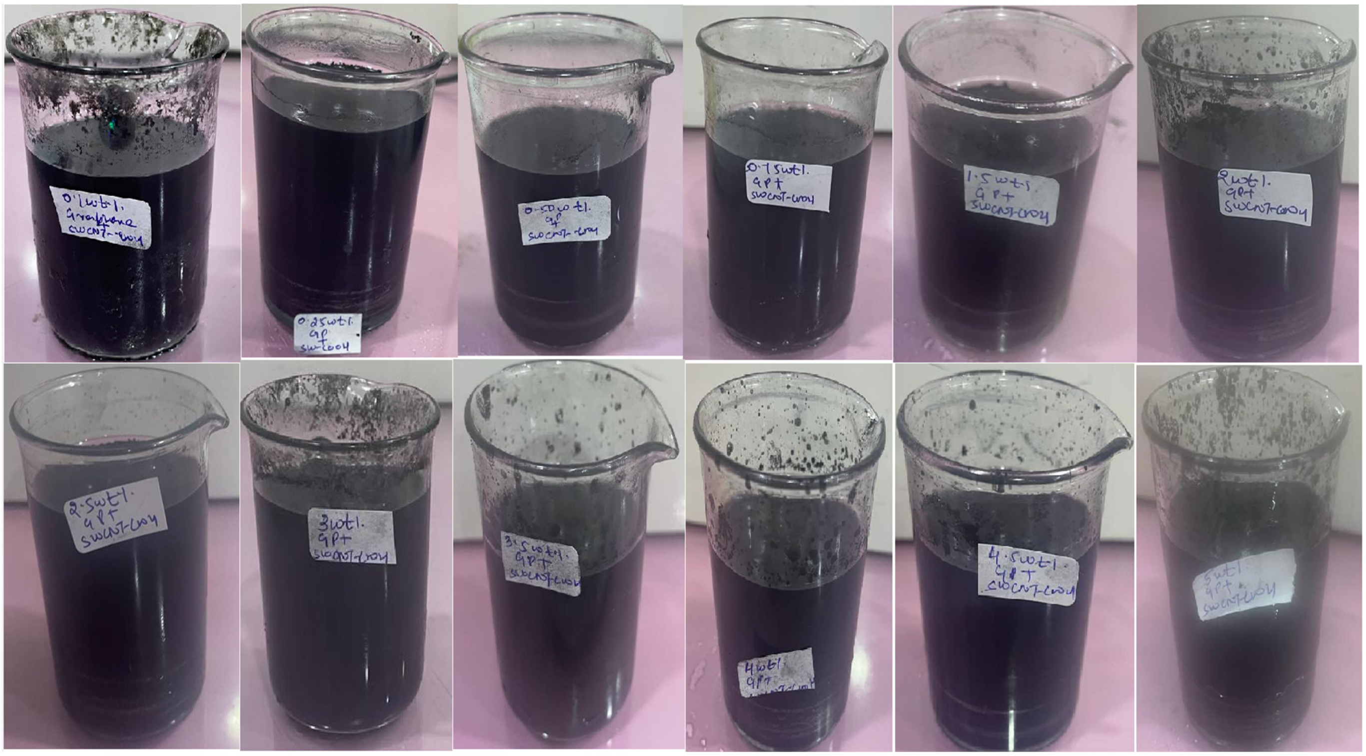


Figure 6.11: Solutions of various concentrations of Graphene/SWCNT-COOH/PDMS

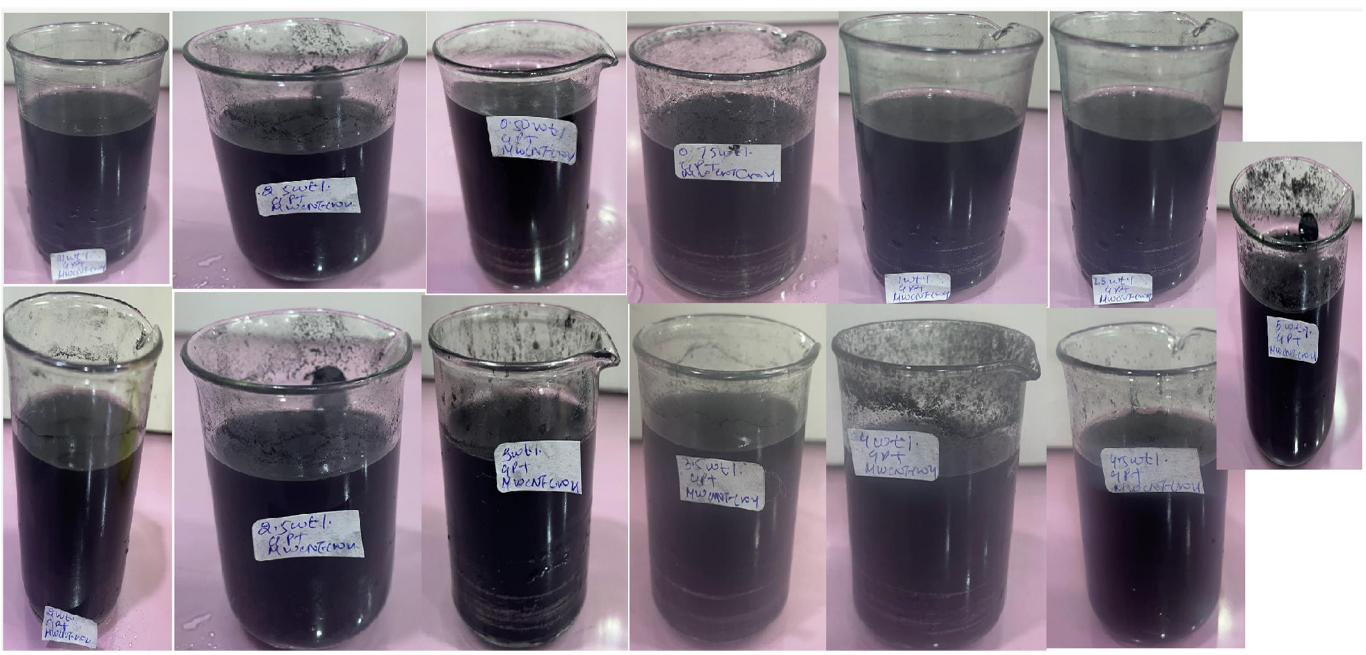


Figure 6.12: Solutions of various concentrations of Graphene/MWCNT-COOH/PDMS

Lastly, the nanocomposites were poured into the molds and thermally cured in the oven for 2-6 hours, depending on the various concentrations of the nanomaterials at 100°C. The final electrodes are obtained as shown in Figure 6.13 and Figure 6.14.

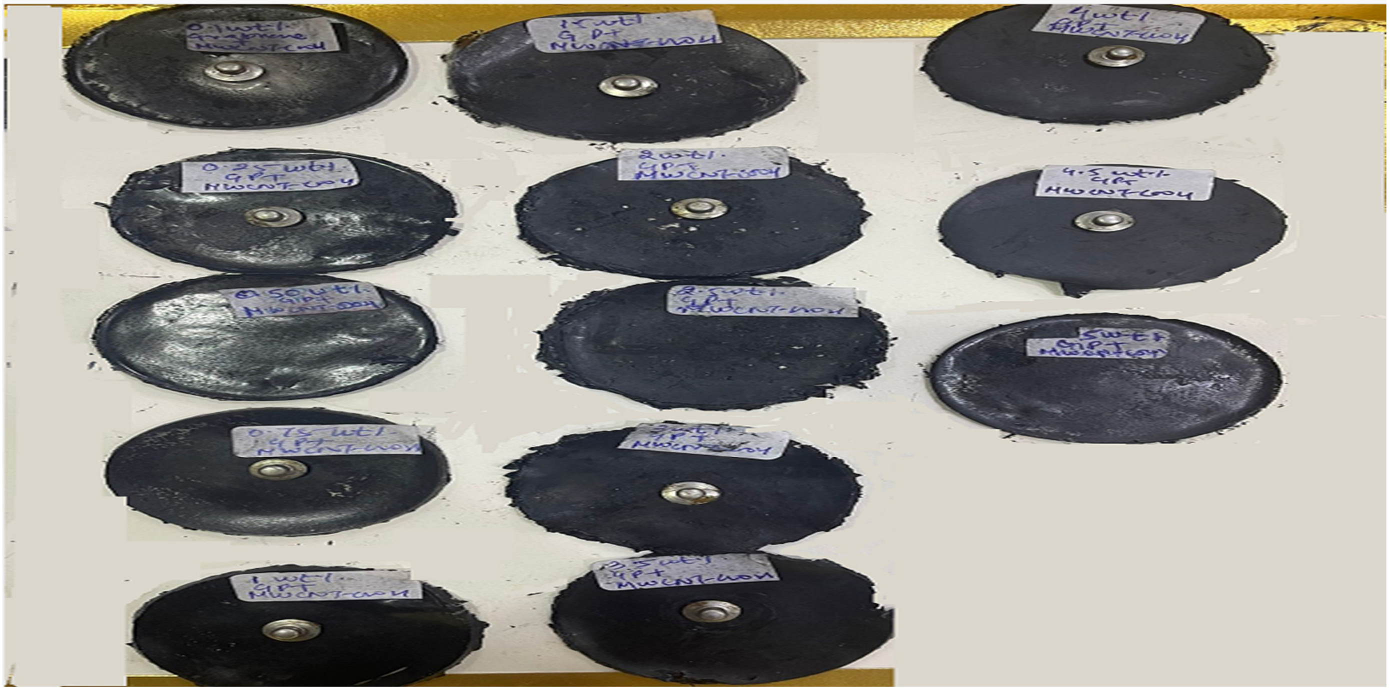


Figure 6.13: Fabricated Electrodes of Graphene/MWCNT-COOH/PDMS

Figure 6.14: Fabricated Electrodes of Graphene/SWCNT-COOH/PDMS

6.3.3 Results

(a) Long-term ECG Monitoring

All the electrodes were worn for 5 days for checking their long-term compatibility of the electrodes. The results on the 3rd and 5th day were a little degraded but after cleansing with methanol, the results revived. The electrodes were safe to wear, and no skin irritation or allergy was identified.

(b) Capacitance of the ECG Electrodes

ESCORT ELC-133A was used to measure the capacitance. A graph has been plotted between concentrations of all the thirty-nine electrodes versus capacitances. The capacitances of Graphene/ MWCNT-COOH/PDMS (G-IV), and Graphene/SWCNT-COOH (G-V) is depicted in Figure 6.15. For G-I, the capacitance is in the range 0.012-0.0176nF and the trend is that the capacitance is increasing but not at a faster rate, the largest value is seen at 5wt%. For G-IV, the capacitance is increasing for different concentrations and the largest value of 0.061nF is seen at 5wt%. For G-V, the capacitance is increasing but only till 3wt% had the highest value of 0.172nF, and later the values tend to decrease, due to the percolation threshold, also this value is greater than G-IV electrodes at 5wt%. The highest value of capacitance is observed at 3wt% (0.172nF) of G-V electrodes. Therefore, with G-V good capacitance values can be seen at lower concentrations of nanomaterials when compared with G-IV and G-I electrodes.

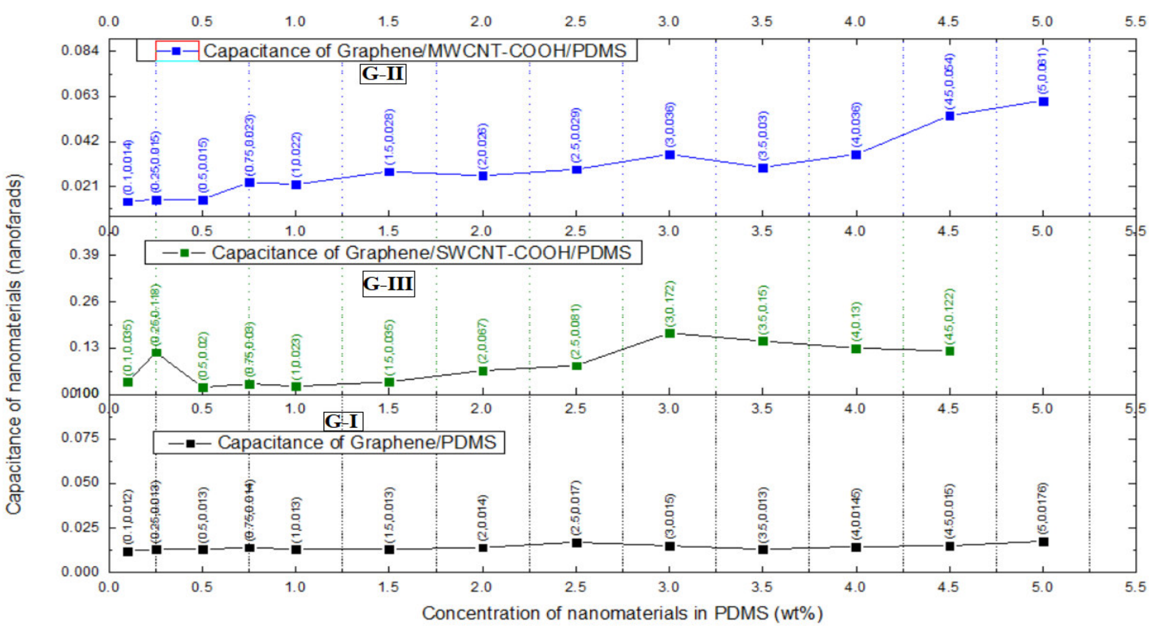


Figure 6.15: Capacitance of G-I, G-II, and G-III

(c) Resistance of the ECG electrodes

ESCORT ELC-133A was used to measure the resistance. A graph has been plotted between concentrations of all the thirty-nine electrodes versus resistance. Graphene/MWCNT-COOH/PDMS (G-IV), and Graphene/SWCNT-COOH (G-V) are depicted in Figure 6.16. For G-IV the resistance is decreasing and the peak was achieved at 8.426 MΩ at 2.5wt%. At 4.5wt%, the least value of resistance is observed which is 1.138 MΩ. For G-V electrodes the resistance values are consistently decreasing with increasing concentrations at 4wt% 7.78 MΩ.

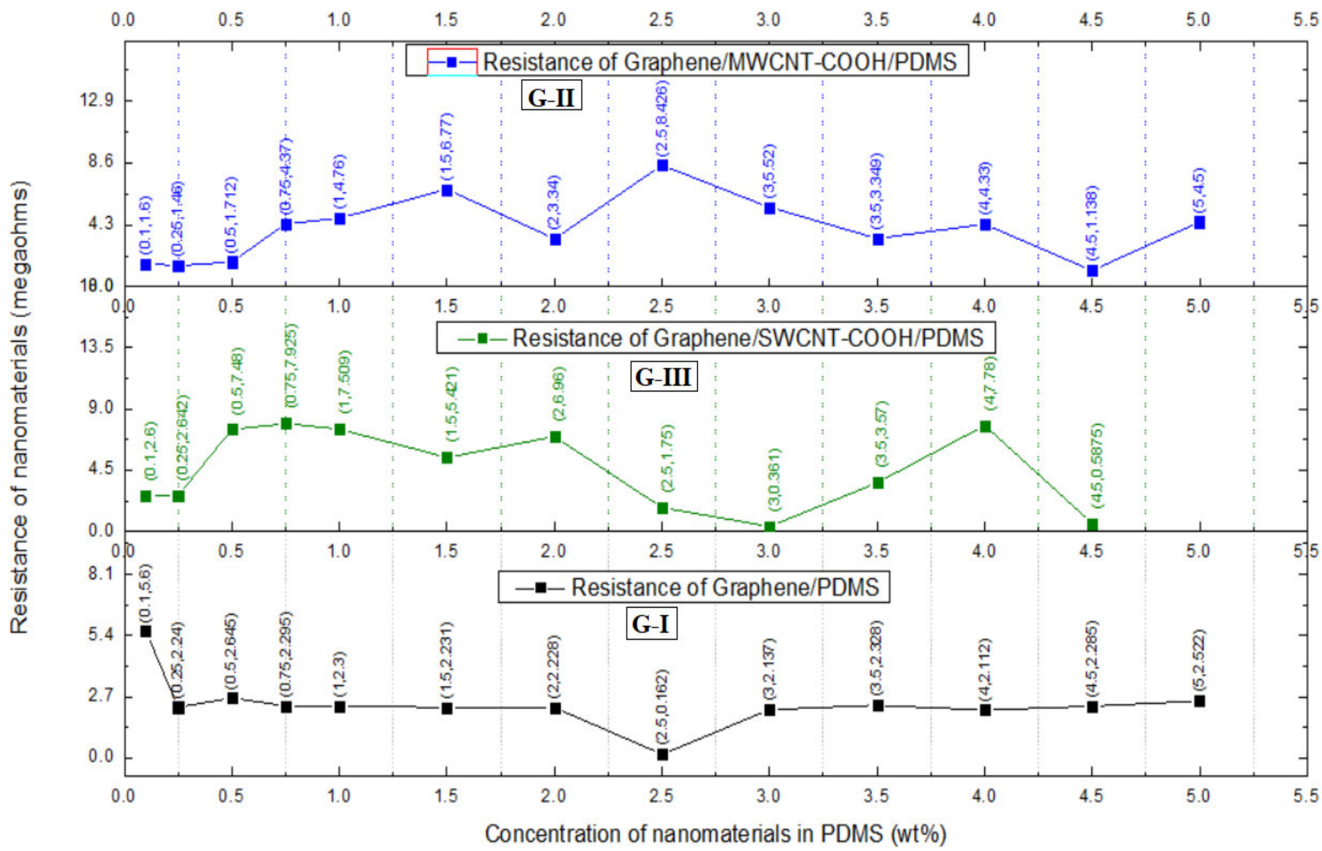


Figure 6.16: Resistance of G-I, G-II, and G-III

(d) Impedance of the ECG Electrodes

ESCORT ELC-133A was used to measure the impedance. A graph has been plotted between concentrations of all the thirty-nine electrodes versus impedance. The impedance of Graphene/MWCNT-COOH/PDMS(G-IV), and Graphene/SWCNT-COOH (G-V) are depicted in Figure 6.17. For G-I, the impedance is in the range 17-0.161MΩ and the trend is that the impedance is decreasing but it is almost constant, and the lowest value is seen at 2.5wt%. For G-IV, the impedance is decreasing for different concentrations and the lowest value of 1.138 MΩ is seen at 4.5wt%. For G-V, the impedance is decreasing with the increasing concentration of nanomaterials, a sharp peak was seen at 4wt% with 7.78 MΩ.

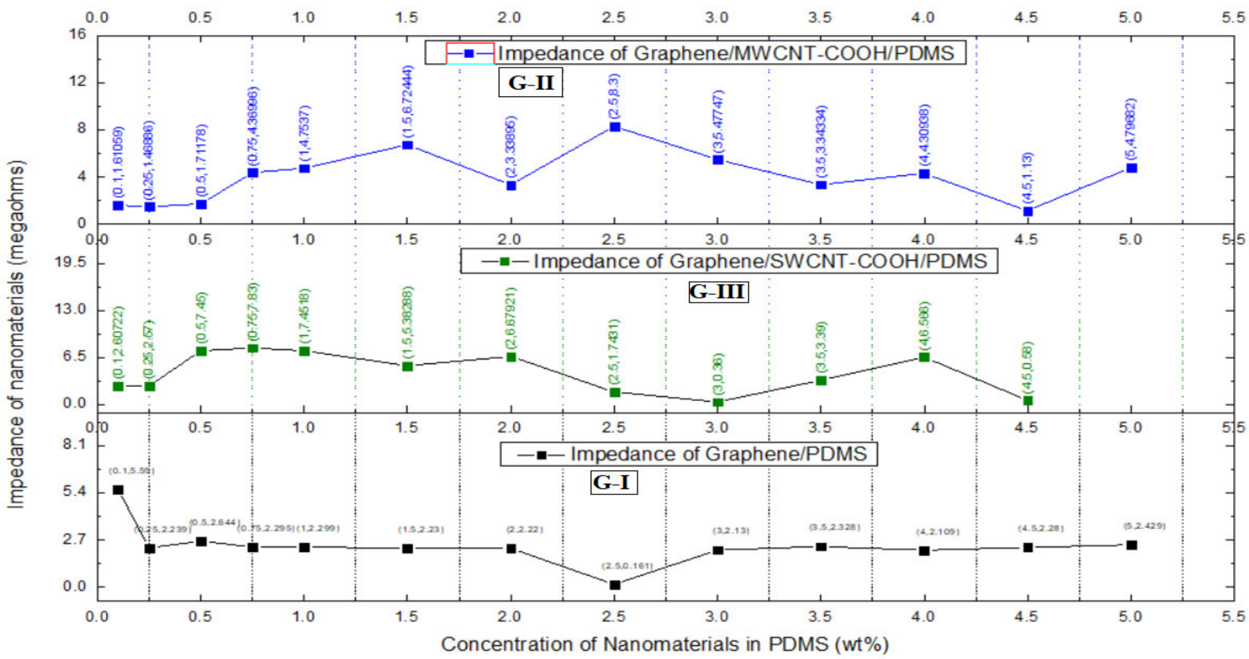
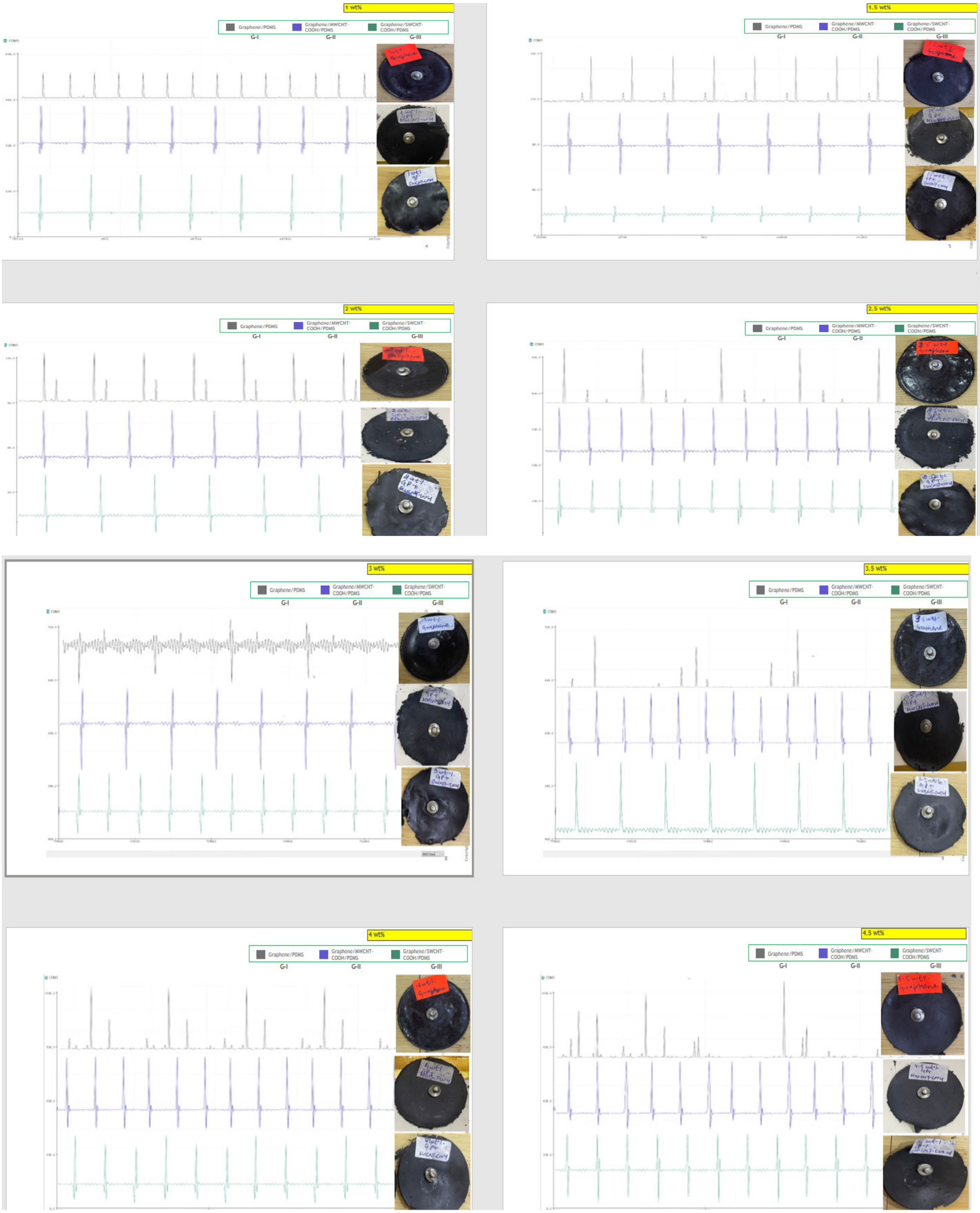


Figure 6.17: Impedance of G-I, G-II, and G-III

(e) Comparison with Ag/AgCl Electrodes

Figure 6.18 shows the ECG waveforms of the fabricated thirty-nine electrodes. The ECG waveforms obtained from the electrodes with lower concentrations of nanomaterials do not offer satisfactory ECG signals for all the groups. Defined peaks were seen improving from 1wt%. For G-I electrodes, the ECG waveforms obtained were not up to the mark. The ECG waveforms were not even resembling the basic ECG waveform. For G-IV electrodes, the best ECG waveform is observed at 4wt% with clear peaks. For G-V electrodes, good ECG results were seen at 3wt%.


0.1wt%
Graphene/PDMS
Graphene/MWCNT-COOH/PDMS
Graphene/SWCNT-COOH/PDMS
G-I
G-II
G-III
0.25wt%
0.5wt%
0.75 wt%


1 wt%
1.5 wt%
2 wt%
2.5 wt%
3 wt%
3.5 wt%
4 wt%
4.5 wt%
Graphene/PDMS
G-I
Graphene/MWCNT-COOH/PDMS
G-II
Graphene/SWCNT-COOH/PDMS
G-III

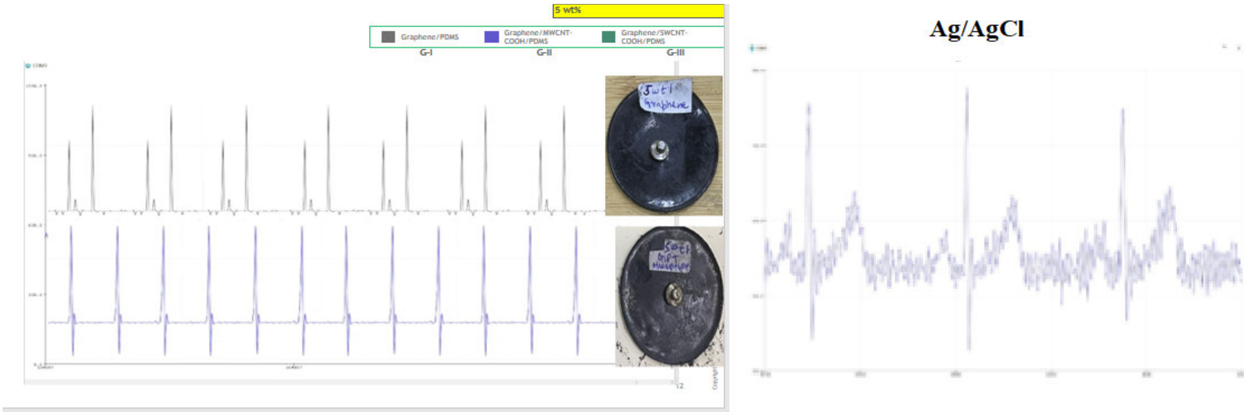


Figure 6.18. ECG waveforms of fabricated electrodes

6.3.4 Discussion

Table 6.2 represents a comparison with other similar works.

Table 6.2: Comparative results of various works

S.No	Ref	Materials	Fabrication technique/methodology	Nature of the sensor	Comparative evaluation	IoT enabled
1.	[172]	Chemically -modified graphene/ MWCNT-COOH	Drop cast	Film (circular)	CG/f@MWCNTs-25% portrayed the best ECG results among others	No
2.	[59]	Graphene/ MWCNT	Spin coating	Film (circular)	Results were comparable to Ag/AgCl electrodes	No
3.	This work	G-I	Mold casting	Film (circular)	No definite ECG waveform was observed	Yes
		G-II			2wt% had better results than Ag/AgCl electrodes G-I and G-III	
		G-III			3wt% electrodes had results comparable to Ag/AgCl electrodes	

6.4 Conclusion

6.4.1 Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS (G-II), and SWCNT-COOH/MWCNT-COOH/PDMS (G-III)

Three types of electrodes have been fabricated; G-I, G-II, and G-III. G-I electrodes did not portray good waveforms, although their capacitive and resistive values were better than G-III electrodes.

G-II electrodes with 2wt% displayed the best ECG waveform with well-defined peaks and no baseline wander was observed. Also, this group of electrodes was economical as Graphene is more economical than CNTs. G-III electrodes had extremely high values of resistance and impedance and the best results were seen at 4.5wt% as depicted in Table 6.3.

Table 6.3: Comparison of Graphene/PDMS (G-I), Graphene/SWCNT-COOH/MWCNT-COOH/PDMS (G-II), and SWCNT-COOH/MWCNT-COOH/PDMS (G-III)

S.No	Parameter	G-I	G-II	G-III
1.	Best ECG waveform	No definite ECG waveform was observed	2wt% had better results than Ag/AgCl electrodes G-I and G-III	4.5wt% electrodes had results comparable to Ag/AgCl electrodes
2.	Capacitance (nF)	-	0.192	0.341
3.	Surface Resistance (MΩ)	-	0.22	4.67
4.	Impedance (MΩ)	-	1.875	19.743

6.4.2 Graphene/PDMS (G-I), Graphene/MWCNT-COOH/PDMS (G-IV), and Graphene/SWCNT-COOH/PDMS (G-V)

For G-I electrodes the ECG results were not comparable to the conventional Ag/AgCl electrodes, also the values of capacitance did not show any significant improvements as the values were constant. For G-IV electrodes, 4wt% were comparable with the conventional electrodes. The best ECG waveform with the sharpest peaks was seen at a 3wt% concentration of G-V ECG electrodes. With 3wt% concentration, the ECG results with the best results were received out of the three groups as seen in Table 6.4. Also, electrodes are fabricated with less concentration of nanomaterials which would help fabricate economical electrodes, with the best peaks and great conductivity it offers. Also, these electrodes offer the least resistance, which again is an important parameter for acquiring the weak ECG signal.

Table 6.4: Comparison of Graphene/PDMS (G-I), Graphene/MWCNT-COOH /PDMS(G-IV), and Graphene/SWCNT-COOHPDMS (G-V)

S.No	Parameter	G-I	G-IV	G-V
1.	Best ECG waveform	No definite ECG waveform was observed	4wt% had better results than Ag/AgCl electrodes G-I and G-III	3wt% electrodes had results comparable to Ag/AgCl electrodes
2.	Capacitance (nF)	**-**	0.036	0.172
3.	Surface Resistance (MΩ)	**-**	4.433	0.361
4.	Impedance (MΩ)	**-**	4.309	0.36

The best results were achieved at 2wt% GP/MWCNT-COOH/SWCNT-COOH/ PDMS, the peaks were well-defined, and the values are great for capacitance, resistance, and impedance as seen in Table 6.5. The electrodes were cost-effective even at low concentrations, and great results are achieved.

Table 6.5: Electrodes Fabrication-Conclusion-Technique I and Technique II

S.No	Parameter	GP/ PDMS	GP/MWCNT-COOH/SWCNT-COOH/ PDMS	MWCNT-COOH/ SWCNT-COOH/ PDMS	GP/MWCNT-COOH	GP/SWCNT-COOH
1.	Best ECG waveform	No definite ECG waveform was observed	2wt% had better results than Ag/AgCl electrodes G-I and G-III	4.5wt% electrodes had results comparable to Ag/AgCl electrodes	4wt% had better results than Ag/AgCl electrodes G-I and G-III	3wt% electrodes had results comparable to Ag/AgCl electrodes
2.	Capacitance (nF)	-	0.192	0.341	0.036	0.172
3.	Surface Resistance (MΩ)	-	0.22	4.67	4.433	0.361
4.	Impedance (MΩ)	-	1.875	19.743	4.309	0.36

CHAPTER 7

Conclusion and Future Scope

This chapter is discussed in two segments: (i) the conclusion of this comprehensive work, which has been presented in this thesis, and (ii) the future scope of the presented work. The presented thesis mainly focuses on the fabrication of ECG electrodes which are reusable, cost-effective, and provide long-term and continuous ECG monitoring in a real-time scenario.

7.1 Conclusion of the Presented Work

In the first chapter, a brief introduction to the evolution of nanomaterials has been discussed along with the various synthesis methods. Following this, Graphene and CNTs have been discussed along with their characteristics. The importance of Graphene and CNTs as an integral part of the technological advancement in the medical field, especially in the sensor industry has been discussed. Later, the importance of Nanomaterial-based ECG sensors has been explained along with ECG basics and the importance of wireless communication (IoT) with nanotechnology has been briefly described.

In the second chapter, various cardiovascular diseases have been discussed. Along with this ECG measuring techniques have been described in detail, their patterns, and the electrode placements for various lead systems of the ECG systems have been discussed. Following this, the evolution of ECG electrodes has been discussed including Pre-gelled Electrodes, Polymer-based Electrodes, Fabric Electrodes, Bulb Electrodes/Clamp Electrodes, Polymer, and Fabric-based Electrodes, Pin-shaped Electrodes, and Bulk Sensor. In this work, a 3-lead system has been employed because this work aims to deploy a system where remote, long-term, and continuous ECG monitoring is required, as it makes the system concise and portable.

In the third chapter, the overview of flexible electronics has been described. A detailed description has been done on CNTs and Graphene taken into account for fabricating ECG electrodes for this work. Carboxylic-acid functionalized CNTs, which include both MWCNT-COOH, SWCNT-COOH, and Graphene have been employed to fabricate ECG electrodes of various concentrations. The importance of IoT has been discussed for providing a remote ECG monitoring system. In addition to this, a discussion on software and hardware components required for this system has been discussed. In this research, the framework of an intelligent ECG tracking system which is based on the IoT cloud is anticipated and implemented. The accumulated ECG data via the patient will be communicated to the IoT cloud platform with the help of Wi-Fi. Also, for convenient and on-time access to the data, HTTP protocol has been deployed in the IoT-cloud platform. An API was created for implementing the GUI. The ECG data was stored in the API using a database MongoDB. The system performed effectively, and the results were obtained successfully.

In the fourth chapter, various fabrication techniques like Spin Coating, Bar Coating, Doctor blade technique, Metal Patterning, Screen Printing, Mold Casting, and other unconventional techniques have been discussed in detail along with their advantages and disadvantages. In this research, the mold-formation technique has been employed as it is economical and easy to employ. The reasons include materials required are available easily and are cost-effective. The equipment(s) required are not bulky. Also, the fabricating process is not difficult. After fabricating the molds, the person is only required to pour the composite into the mold and after thermal curing the electrodes are ready. Once the molds are cast, several electrodes can be developed via the 'replication' process.

In the fifth chapter, two groups of electrodes are fabricated MWCNT-COOH/PDMS and SWCNT-COOH/PDMS using Fabrication Technique I. In this technique magnetic stirrer, probe-sonicator and oven are used. First, the molds are fabricated, then CNT-COOH with IPA is first distributed using the magnetic stirrer, then probe sonicated with IPA, and later with PDMS. The final composite is poured into the molds and thermally cured, and lastly, the electrodes are demoulded from the mold. Thirteen different concentrations 0.1wt%, 0.25wt%, 0.5wt%, 0.75wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt%, 3wt%, 3.5wt%, 4wt%, 4.5wt%, and 5wt% for both the groups were fabricated. Physical characterizations like TEM, SEM, FITR, and Raman Spectroscopy were performed. Tests like capacitance, resistance, and impedance were also carried out. The ECG was monitored using the system developed in Chapter 3. The ECG results were compared with the Ag/AgCl electrodes. Of the two groups, the best results were achieved from 3.5wt% concentration with SWCNT-COOH/PDMS. The peaks were well defined when compared with 4wt% MWCNT-COOH/PDMS electrodes. Also, better waveforms are achieved with less concentration of the nanomaterial.

In the sixth chapter, two groups of electrodes are fabricated Graphene/PDMS, Graphene/SWCNT-COOH/MWCNT-COOH/PDMS, SWCNT-COOH/MWCNT-COOH/PDMS, Graphene/MWCNT-COOH/PDMS and Graphene/SWCNT-COOH/PDMS using Fabrication Technique II. In this technique, only a bath-sonicator and oven are used. First, the molds are fabricated, then Nanomaterials with IPA are first distributed using the bath-sonicator, and later with PDMS. The final composite is poured into the molds and thermally cured, and lastly, the electrodes are demoulded from the mold. Thirteen different concentrations 0.1wt%, 0.25wt%, 0.5wt%, 0.75wt%, 1wt%, 1.5wt%, 2wt%, 2.5wt%, 3wt%, 3.5wt%, 4wt%, 4.5wt%, and 5wt% for both the groups were fabricated. Physical characterizations like TEM, SEM, FTIR, and Raman Spectroscopy were performed. Tests like capacitance, resistance, and impedance were also carried out. The ECG was monitored using the system developed in Chapter 3. The ECG results were compared with the Ag/AgCl electrodes. Of the two groups, the best results were achieved from 2wt% concentration with Graphene/SWCNT-COOH/MWCNT-COOH/PDMS. The peaks were well defined when compared with all the fabricated electrodes. Also, better waveforms are achieved with less concentration of the nanomaterial.

7.2 Future scope of the Presented Work

This thesis provides the results of various fabricated electrodes using Graphene, MWCNT-COOH, and SWCNT-COOH with PDMS as the polymer. The fabricated electrodes are non-toxic, biocompatible, and suitable for long-term, continuous, and remote ECG monitoring.

This thesis mostly concentrated on measuring the ECG signals, many other physiological signals like EEG, EMG, pH, etc. can be monitored too.

Many other nanomaterials can also be combined and electrodes can be fabricated using different permutations and combinations. As in this work, a 3-lead system is used, to make the system more portable, a 1-lead system can be incorporated. Also, the fabricated process can be made more time-efficient. Also, the response time and sensitivity of the electrode can be taken into consideration.

List of Publications

1. Journals

i. Bani Gandhi and N.S. Raghava, "Fabrication Techniques for Carbon Nanotubes Based ECG Electrodes: A Review," *IETE Journal of Research*, DOI: DOI: 10.1080/03772063.2020.1768909, May 2020, SCIE (2.333).

ii. Bani Gandhi and N. S. Raghava, "MWCNT-COOH/PDMS based dry ECG electrodes for ambulatory ECG recordings", *Materials Letters*, vol.307, pp.1-5, Oct 2021, SCI (3.423)

iii. Bani Gandhi, N. S. Raghava, "Fabrication and Analysis of Carboxylic acid-functionalized SWCNT/PDMS based electrodes for ECG monitoring via IoT", Under Review: *Microsystem Technologies.*

iv. Bani Gandhi, N. S. Raghava, "Graphene and Graphene Nanohybrid Composites-based Electrodes for Physiological Sensing Applications", Accepted: *Biomedical Microdevices.*

v. Bani Gandhi, N. S. Raghava, "Fabrication and analysis of Polymer Nanocomposite based dry ECG Electrodes for Wireless Healthcare Applications", Under Review: *IETE Journal of Research.*

2. Conferences

i. Bani Gandhi and N.S. Raghava, "SWCNT-COOH based Electrodes for Smart and Real-Time ECG Monitoring," in 2021 2nd International Conference on Future Communication & Computing Technology (ICFCCT), 2021.

ii. Bani Gandhi, N.S. Raghava, "Dry and Flexible MWCNT-COOH Nanocomposites for Continuous ECG Tracking System," in 2021 2nd International Conference on Future Communication & Computing Technology (ICFCCT), 2021.

iii. Bani Gandhi, N.S. Raghava, "Smart ECG Monitoring System based on IoT," in 2022 5th Edition of International Conference on Future Communication & Computing Technology (ICFCCT), 2021.

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APPENDIX 1: MATERIALS DATASHEET

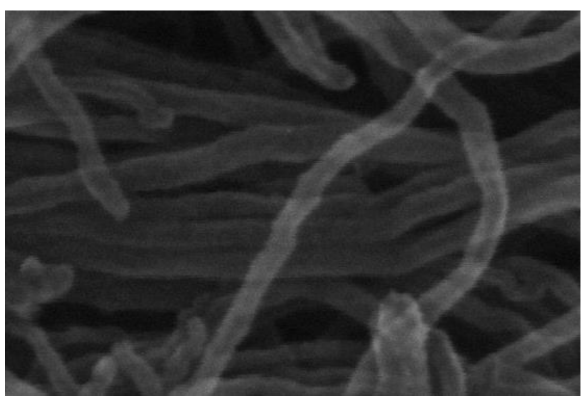
Product Specification Sheet

COOH Functionalized High Purified SWCNTs							
Product No.	**CAS No.**	**Outer Diameter**	**Inner Diameter**	**Average Length**	**Special Surface Area(SSA)**	**Tap Density**	**True Density**
NRE-32003	308068-56-6	1-4 nm	0.8-1.6 nm	5-30 um	690m^2/g	0.09g/cm^3	2.0g/cm^3
Electric Conductivity	> 100 S/cm						
Thermal Conductivity	50-200 W/m.K						
Ignited temperature	630 °C						

Certificate Of Analysis				
C	O	Mg	S	Ni
>98%	1.3	0.4	0.08	0.04

All above mentioned figures are determined by ICPMS. Above values are in % unless and other wise specified

Characterization of COOH Functionalized High Purified SWCNTs



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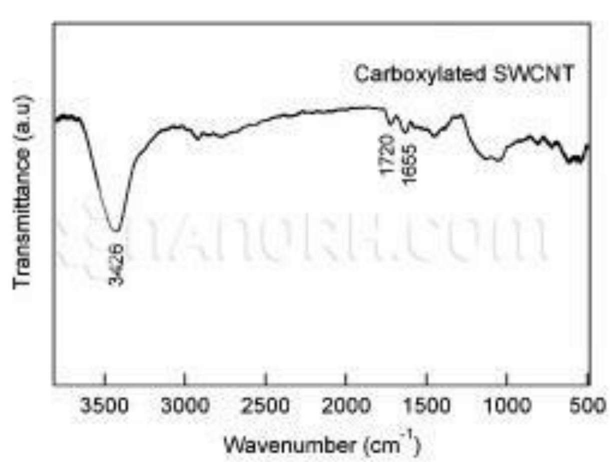
SEM of COOH Functionalized High Purified SWCNTs

TEM of COOH Functionalized High Purified SWCNTs



FTIR of SEM of COOH Functionalized High Purified SWCNTs

Note: Product Specification are subject to amendment and may change over time

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Product Specification Sheet

COOH Functionalized MWCNTs

Description: Sample purity of **COOH Functionalized MWCNTs** is 95-98 Vol%, as determined by Raman Spectrophotometer and SEM Analysis. Nano Research Elements Nanomaterials contains no residual catalyst impurities. Tubes occur in bundles of length ~10-30 µm. (±1.5µm).

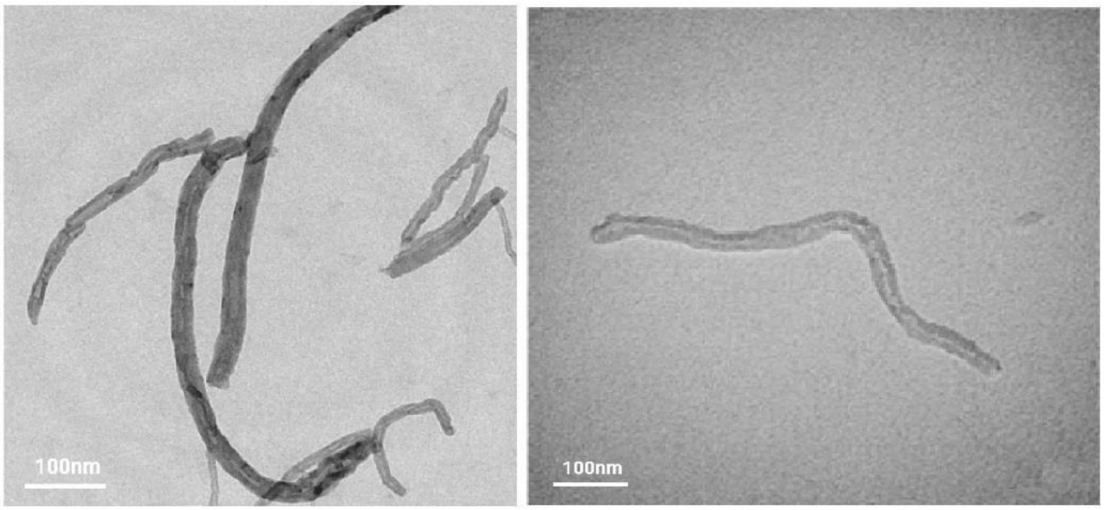
Quality Control: Each lot of **COOH Functionalized MWCNTs** was tested successfully.

COOH Functionalized MWCNTs							
Product No.	**CAS No.**	**Purity**	**Average Diameter**	**Average Length**	**Special Surface Area(SSA)**	**Tap Density**	**True Density**
NRE-5022	308068-56-6	> 98wt%	10-30 nm	10-30 µm (TEM)	>180 m^2/g(BET)	0.27g/cm^3	2.1g/cm^3
Electric Conductivity			> 100 S/cm				
COOH Content %			2-4Wt%				
Ignited temperature			NA				

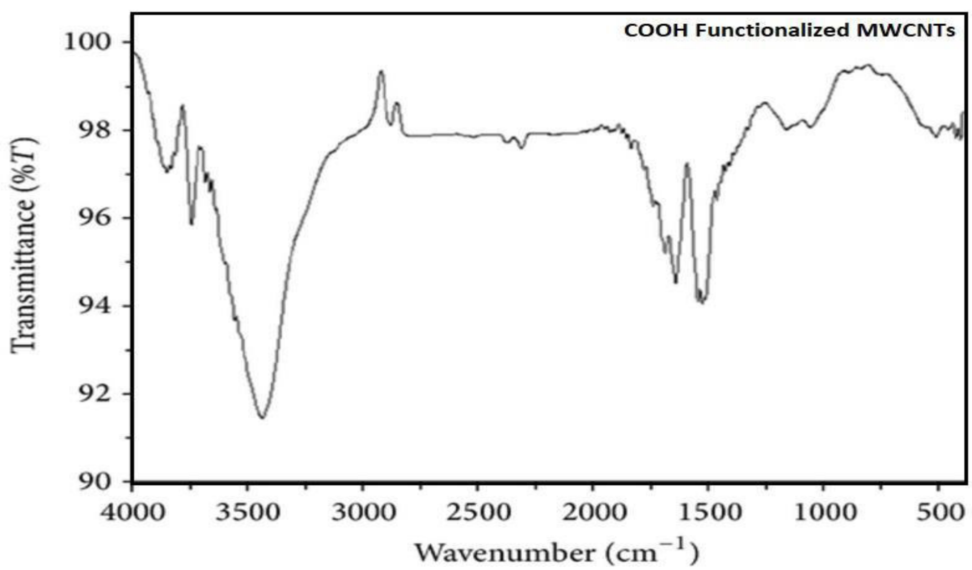
Certificate Of Analysis				
C	O	Fe	Ni	S
>98%	1.4	0.5	0.09	0.22

All above mentioned figures are determined by ICPMS. Above values are in % unless and other wise specified

Characterization of COOH Functionalized MWCNTs



TEM of COOH Functionalized MWCNTs



FTIR of COOH Functionalized MWCNTs

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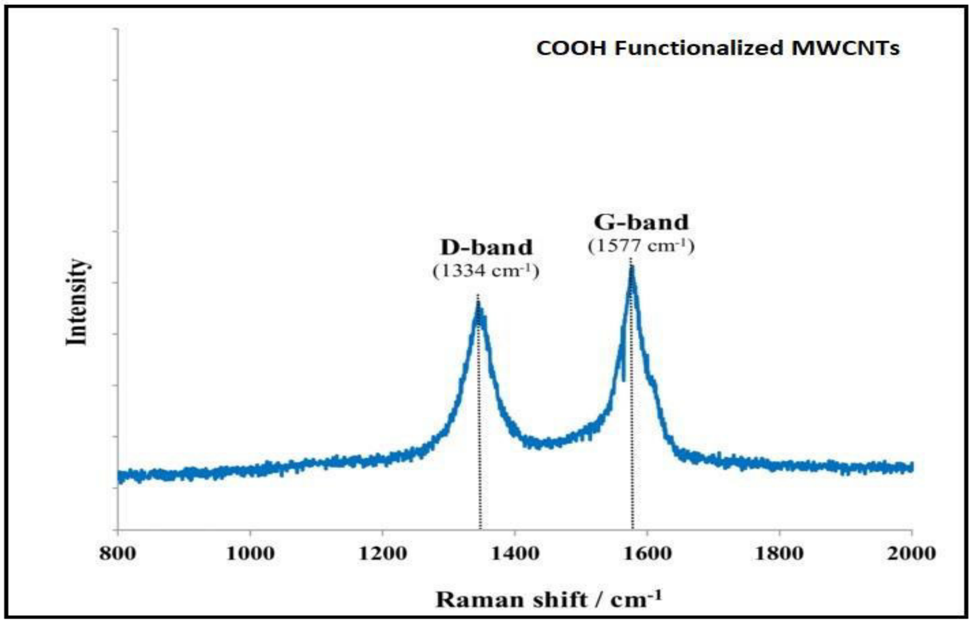

Raman of COOH Functionalized MWCNTs

Note: Product Specification are subject to amendment and may change over time

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SAFETY DATA SHEET

Creation Date 09-Apr-2018 **Revision Date** 19-Feb-2021 **Revision Number** 2

SECTION 1: IDENTIFICATION OF THE SUBSTANCE/MIXTURE AND OF THE COMPANY/UNDERTAKING

1.1. Product identifier

Product Description:	**Graphene nanoplatelet aggregates**
Cat No. :	**47132**
CAS-No	7782-42-5

1.2. Relevant identified uses of the substance or mixture and uses advised against

Recommended Use	Laboratory chemicals.
Uses advised against	No Information available

1.3. Details of the supplier of the safety data sheet

Company

Alfa Aesar .
Avocado Research Chemicals, Ltd.
Shore Road
Port of Heysham Industrial Park
Heysham, Lancashire LA3 2XY
United Kingdom
Office Tel: +44 (0) 1524 850506
Office Fax: +44 (0) 1524 850608

E-mail address

uktech@alfa.com
www.alfa.com
Product Safety Department

1.4. Emergency telephone number

Call Carechem 24 at
+44 (0) 1865 407333 (English only);
+44 (0) 1235 239670 (Multi-language)

SECTION 2: HAZARDS IDENTIFICATION

2.1. Classification of the substance or mixture

CLP Classification - Regulation (EC) No 1272/2008

Physical hazards

Based on available data, the classification criteria are not met

Health hazards

Serious Eye Damage/Eye Irritation	Category 2 (H319)

Specific target organ toxicity - (single exposure)	Category 3 (H335)

Environmental hazards
Based on available data, the classification criteria are not met

Full text of Hazard Statements: see section 16

2.2. Label elements

Signal Word **Warning**

Hazard Statements
H319 - Causes serious eye irritation
H335 - May cause respiratory irritation

Precautionary Statements
P280 - Wear eye protection/ face protection
P337 + P313 - If eye irritation persists: Get medical advice/attention
P304 + P340 - IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing
P312 - Call a POISON CENTER or doctor/physician if you feel unwell

2.3. Other hazards

Substance is not considered persistent, bioaccumulative and toxic (PBT) / very persistent and very bioaccumulative (vPvB)

SECTION 3: COMPOSITION/INFORMATION ON INGREDIENTS

3.1. Substances

Component	CAS-No	EC-No.	Weight %	CLP Classification - Regulation (EC) No 1272/2008
Graphite	7782-42-5	EEC No. 231-955-3	<=100	Eye Irrit. 2 (H319) STOT SE 3 (H335)

Full text of Hazard Statements: see section 16

SECTION 4: FIRST AID MEASURES

4.1. Description of first aid measures

General Advice	If symptoms persist, call a physician.
Eye Contact	Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes. Get medical attention.
Skin Contact	Wash off immediately with plenty of water for at least 15 minutes. If skin irritation persists, call a physician.
Ingestion	Clean mouth with water and drink afterwards plenty of water. Get medical attention if symptoms occur.
Inhalation	Remove to fresh air. If not breathing, give artificial respiration. Get medical attention if symptoms occur.
Self-Protection of the First Aider	No special precautions required.

4.2. Most important symptoms and effects, both acute and delayed

None reasonably foreseeable.

4.3. Indication of any immediate medical attention and special treatment needed

Notes to Physician Treat symptomatically.

SECTION 5: FIREFIGHTING MEASURES

5.1. Extinguishing media

Suitable Extinguishing Media
Carbon dioxide (CO_2). Powder. Water spray. In case of major fire and large quantities: Evacuate area. Fight fire remotely due to the risk of explosion.

Extinguishing media which must not be used for safety reasons
No information available.

5.2. Special hazards arising from the substance or mixture

Thermal decomposition can lead to release of irritating gases and vapors.

Hazardous Combustion Products
Carbon monoxide (CO), Carbon dioxide (CO_2).

5.3. Advice for firefighters

As in any fire, wear self-contained breathing apparatus pressure-demand, MSHA/NIOSH (approved or equivalent) and full protective gear.

SECTION 6: ACCIDENTAL RELEASE MEASURES

6.1. Personal precautions, protective equipment and emergency procedures

Ensure adequate ventilation. Use personal protective equipment as required. Avoid dust formation.

6.2. Environmental precautions

Should not be released into the environment. See Section 12 for additional Ecological Information.

6.3. Methods and material for containment and cleaning up

Sweep up and shovel into suitable containers for disposal. Keep in suitable, closed containers for disposal.

6.4. Reference to other sections

Refer to protective measures listed in Sections 8 and 13.

SECTION 7: HANDLING AND STORAGE

7.1. Precautions for safe handling

Wear personal protective equipment/face protection. Ensure adequate ventilation. Avoid ingestion and inhalation. Avoid dust formation. Do not get in eyes, on skin, or on clothing.

Hygiene Measures
Handle in accordance with good industrial hygiene and safety practice. Keep away from food, drink and animal feeding stuffs. Do not eat, drink or smoke when using this product. Remove and wash contaminated clothing and gloves, including the inside, before re-use. Wash hands before breaks and after work.

7.2. Conditions for safe storage, including any incompatibilities

Keep container tightly closed in a dry and well-ventilated place.

Technical Rules for Hazardous Substances (TRGS) 510 Storage Class (LGK) (Germany) Class 11

7.3. Specific end use(s)

Use in laboratories

SECTION 8: EXPOSURE CONTROLS/PERSONAL PROTECTION

8.1. Control parameters

Exposure limits
List source(s): **UK** - EH40/2005 Work Exposure Limits, Third edition. Published 2018. **IRE -** 2018 Code of Practice for the Chemical Agents Regulations, Schedule 1. Published by the Health and Safety Authority

Component	The United Kingdom	European Union	Ireland
Graphite	STEL: 30 mg/m³ 15 min STEL: 12 mg/m³ 15 min TWA: 10 mg/m³ 8 hr TWA: 4 mg/m³ 8 hr		TWA: 2 mg/m³ 8 hr. all forms except fibres; respirable fraction STEL: 6 mg/m³ 15 min

Biological limit values
This product, as supplied, does not contain any hazardous materials with biological limits established by the region specific regulatory bodies

Monitoring methods
BS EN 14042:2003 Title Identifier: Workplace atmospheres. Guide for the application and use of procedures for the assessment of exposure to chemical and biological agents.
MDHS14/3 General methods for sampling and gravimetric analysis of respirable and inhalable dust

Derived No Effect Level (DNEL) No information available

Route of exposure	Acute effects (local)	Acute effects (systemic)	Chronic effects (local)	Chronic effects (systemic)
Oral				

Dermal
Inhalation

Predicted No Effect Concentration (PNEC) No information available.

8.2. Exposure controls

Engineering Measures
Ensure that eyewash stations and safety showers are close to the workstation location.
Wherever possible, engineering control measures such as the isolation or enclosure of the process, the introduction of process or equipment changes to minimise release or contact, and the use of properly designed ventilation systems, should be adopted to control hazardous materials at source

Personal protective equipment

Eye Protection Goggles (European standard - EN 166)

Hand Protection Protective gloves

Glove material	Breakthrough time	Glove thickness	EU standard	Glove comments
Nitrile rubber	See manufacturers recommendations	-	EN 374	(minimum requirement)

Skin and body protection Long sleeved clothing.

Inspect gloves before use.
Please observe the instructions regarding permeability and breakthrough time which are provided by the supplier of the gloves. (Refer to manufacturer/supplier for information)
Ensure gloves are suitable for the task: Chemical compatability, Dexterity, Operational conditions, User susceptibility, e.g. sensitisation effects, also take into consideration the specific local conditions under which the product is used, such as the danger of cuts, abrasion.
Remove gloves with care avoiding skin contamination.

Respiratory Protection When workers are facing concentrations above the exposure limit they must use appropriate certified respirators.
To protect the wearer, respiratory protective equipment must be the correct fit and be used and maintained properly

Large scale/emergency use In case of insufficient ventilation, wear suitable respiratory equipment
Recommended Filter type: Particle filter 2

Small scale/Laboratory use Use a NIOSH/MSHA or European Standard EN 149:2001 approved respirator if exposure limits are exceeded or if irritation or other symptoms are experienced.
When RPE is used a face piece Fit Test should be conducted

Environmental exposure controls No information available.

SECTION 9: PHYSICAL AND CHEMICAL PROPERTIES

9.1. Information on basic physical and chemical properties

Physical State Solid

Appearance Black
Odor No information available
Odor Threshold No data available
Melting Point/Range > 2760 °C / 5000 °F
Softening Point No data available
Boiling Point/Range No information available

Flammability (liquid)	Not applicable	Solid
Flammability (solid,gas)	No information available	
Explosion Limits	No data available	
Flash Point	No information available	**Method -** No information available
Autoignition Temperature	No data available	
Decomposition Temperature	No data available	
pH	No information available	
Viscosity	Not applicable	Solid
Water Solubility	Insoluble in water	
Solubility in other solvents	No information available	
Partition Coefficient (n-octanol/water)		
Vapor Pressure	No data available	
Density / Specific Gravity	2 g/cm3	@ 20 °C
Bulk Density	No data available	
Vapor Density	Not applicable	Solid
Particle characteristics	No data available	

9.2. Other information

Evaporation Rate Not applicable - Solid

SECTION 10: STABILITY AND REACTIVITY

10.1. Reactivity

None known, based on information available

10.2. Chemical stability

Stable under normal conditions.

10.3. Possibility of hazardous reactions

Hazardous Polymerization No information available.
Hazardous Reactions None under normal processing.

10.4. Conditions to avoid

Incompatible products. Excess heat.

10.5. Incompatible materials

Oxidizing agent.

10.6. Hazardous decomposition products

Carbon monoxide (CO). Carbon dioxide (CO_2).

SECTION 11: TOXICOLOGICAL INFORMATION

11.1. Information on hazard classes as defined in Regulation (EC) No 1272/2008

Product Information

(a) acute toxicity;

Oral	No data available
Dermal	No data available
Inhalation	No data available

Component	LD50 Oral	LD50 Dermal	LC50 Inhalation

Graphite	-	-	LC50 > 2000 mg/m³ (Rat) 4 h

(b) skin corrosion/irritation; No data available

(c) serious eye damage/irritation; Category 2

(d) respiratory or skin sensitization;
Respiratory No data available
Skin No data available

(e) germ cell mutagenicity; No data available

(f) carcinogenicity; No data available

There are no known carcinogenic chemicals in this product

(g) reproductive toxicity; No data available

(h) STOT-single exposure; Category 3

Results / Target organs Respiratory system.

(i) STOT-repeated exposure; No data available

Target Organs No information available.

(j) aspiration hazard; Not applicable
Solid

Symptoms / effects,both acute and delayed No information available.

11.2. Information on other hazards

Endocrine Disrupting Properties Assess endocrine disrupting properties for human health. This product does not contain any known or suspected endocrine disruptors.

SECTION 12: ECOLOGICAL INFORMATION

12.1. Toxicity
Ecotoxicity effects .

Component	Freshwater Fish	Water Flea	Freshwater Algae
Graphite	LC50: > 100 mg/L, 96h semi-static (Danio rerio)		

12.2. Persistence and degradability
Persistence Insoluble in water.

12.3. Bioaccumulative potential	May have some potential to bioaccumulate
12.4. Mobility in soil	Spillage unlikely to penetrate soil The product is insoluble and sinks in water Is not likely mobile in the environment due its low water solubility.
12.5. Results of PBT and vPvB assessment	Substance is not considered persistent, bioaccumulative and toxic (PBT) / very persistent and very bioaccumulative (vPvB).
12.6. Endocrine disrupting properties	
Endocrine Disruptor Information	This product does not contain any known or suspected endocrine disruptors
12.7. Other adverse effects	
Persistent Organic Pollutant	This product does not contain any known or suspected substance
Ozone Depletion Potential	This product does not contain any known or suspected substance

SECTION 13: DISPOSAL CONSIDERATIONS

13.1. Waste treatment methods

Waste from Residues/Unused Products	Waste is classified as hazardous. Dispose of in accordance with the European Directives on waste and hazardous waste. Dispose of in accordance with local regulations.
Contaminated Packaging	Dispose of this container to hazardous or special waste collection point. Empty containers retain product residue, (liquid and/or vapor), and can be dangerous. Keep product and empty container away from heat and sources of ignition.
European Waste Catalogue (EWC)	According to the European Waste Catalog, Waste Codes are not product specific, but application specific.
Other Information	Waste codes should be assigned by the user based on the application for which the product was used. Do not flush to sewer. Can be landfilled or incinerated, when in compliance with local regulations. Do not empty into drains.

SECTION 14: TRANSPORT INFORMATION

IMDG/IMO Not regulated

14.1. UN number
14.2. UN proper shipping name
14.3. Transport hazard class(es)
14.4. Packing group

ADR Not regulated

14.1. UN number
14.2. UN proper shipping name
14.3. Transport hazard class(es)
14.4. Packing group

IATA Not regulated

14.1. UN number
14.2. UN proper shipping name
14.3. Transport hazard class(es)
14.4. Packing group

14.5. Environmental hazards No hazards identified

14.6. Special precautions for user No special precautions required

14.7. Maritime transport in bulk according to IMO instruments Not applicable, packaged goods

SECTION 15: REGULATORY INFORMATION

15.1. Safety, health and environmental regulations/legislation specific for the substance or mixture

International Inventories
X = listed, Europe (EINECS/ELINCS/NLP), U.S.A. (TSCA), Canada (DSL/NDSL), Philippines (PICCS), China (IECSC), Japan (ENCS), Australia (AICS), Korea (ECL).

Component	EINECS	ELINCS	NLP	TSCA	DSL	NDSL	PICCS	ENCS	IECSC	AICS	KECL
Graphite	231-955-3	-		X	X	-	X	-	X	X	KE-18101

Regulation (EC) No 649/2012 of the European Parliament and of the Council of 4 July 2012 concerning the export and import of dangerous chemicals
Not applicable

National Regulations

WGK Classification See table for values

Component	Germany - Water Classification (VwVwS)	Germany - TA-Luft Class
Graphite	nwg	

Component	France - INRS (Tables of occupational diseases)
Graphite	Tableaux des maladies professionnelles (TMP) - RG 16 Tableaux des maladies professionnelles (TMP) - RG 25

UK - Take note of Control of Substances Hazardous to Health Regulations (COSHH) 2002 and 2005 Amendment

15.2. Chemical safety assessment

A Chemical Safety Assessment/Report (CSA/CSR) has not been conducted

SECTION 16: OTHER INFORMATION

Full text of H-Statements referred to under sections 2 and 3
H319 **-** Causes serious eye irritation
H335 **-** May cause respiratory irritation

Legend

CAS - Chemical Abstracts Service
EINECS/ELINCS - European Inventory of Existing Commercial Chemical Substances/EU List of Notified Chemical Substances
PICCS - Philippines Inventory of Chemicals and Chemical Substances
IECSC - Chinese Inventory of Existing Chemical Substances
KECL - Korean Existing and Evaluated Chemical Substances
TSCA - United States Toxic Substances Control Act Section 8(b) Inventory
DSL/NDSL - Canadian Domestic Substances List/Non-Domestic Substances List
ENCS - Japanese Existing and New Chemical Substances
AICS - Australian Inventory of Chemical Substances
NZIoC - New Zealand Inventory of Chemicals

WEL - Workplace Exposure Limit
ACGIH - American Conference of Governmental Industrial Hygienists
DNEL - Derived No Effect Level
RPE - Respiratory Protective Equipment
LC50 - Lethal Concentration 50%
NOEC - No Observed Effect Concentration
PBT - Persistent, Bioaccumulative, Toxic

TWA - Time Weighted Average
IARC - International Agency for Research on Cancer
Predicted No Effect Concentration (PNEC)
LD50 - Lethal Dose 50%
EC50 - Effective Concentration 50%
POW - Partition coefficient Octanol:Water
vPvB - very Persistent, very Bioaccumulative

ADR - European Agreement Concerning the International Carriage of Dangerous Goods by Road
IMO/IMDG - International Maritime Organization/International Maritime Dangerous Goods Code
OECD - Organisation for Economic Co-operation and Development
BCF - Bioconcentration factor

ICAO/IATA - International Civil Aviation Organization/International Air Transport Association
MARPOL - International Convention for the Prevention of Pollution from Ships
ATE - Acute Toxicity Estimate
VOC - (Volatile Organic Compound)

Key literature references and sources for data
https://echa.europa.eu/information-on-chemicals
Suppliers safety data sheet, Chemadvisor - LOLI, Merck index, RTECS

Training Advice
Chemical hazard awareness training, incorporating labelling, Safety Data Sheets (SDS), Personal Protective Equipment (PPE) and hygiene.

Prepared By	Health, Safety and Environmental Department
Creation Date	09-Apr-2018
Revision Date	19-Feb-2021
Revision Summary	Update to CLP Format.

This safety data sheet complies with the requirements of Regulation (EC) No. 1907/2006 COMMISSION REGULATION (EU) 2020/878 amending Annex II to Regulation (EC) No 1907/2006

Disclaimer
The information provided in this Safety Data Sheet is correct to the best of our knowledge, information and belief at the date of its publication. The information given is designed only as a guidance for safe handling, use, processing, storage, transportation, disposal and release and is not to be considered a warranty or quality specification. The information relates only to the specific material designated and may not be valid for such material used in combination with any other materials or in any process, unless specified in the text

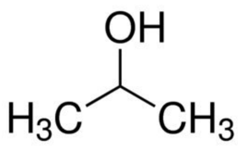
End of Safety Data Sheet

LABORATORY REAGENTS & FINE CHEMICALS

MATERIAL SAFETY DATA SHEET (MSDS)

SECTION 1: Identification of the substance/mixture and of the company/undertaking

1.1. Product identifier

Product form	:	Substance
	:	
EC Index-No.	:	603-117-00-0
EC-No.	:	200-661-7
CAS-No.	:	67-63-0
Product code	:	00270
Formula	:	C3H8O
Chemical structure	:	
Synonyms	:	sec-Propyl alcohol / 2-propanol / Isopropanol

1.2. Relevant identified uses of the substance or mixture and uses advised against

1.2.1. Relevant identified uses

Industrial/Professional use spec : Industrial
For professional use only

1.2.2. Uses advised against

No additional information available

1.3. Details of the supplier of the safety data sheet

LOBA CHEMIE PVT.LTD.
107 Wode House Road, Jehangir Villa, Colaba
400005 Mumbai - INDIA
T +91 22 6663 6663 - F +91 22 6663 6699
info@lobachemie.com - www.lobachemie.com

1.4. Emergency telephone number

Emergency number : + 91 22 6663 6663 (9:00am - 6:00 pm)

SECTION 2: Hazards identification

2.1. Classification of the substance or mixture

Classification according to Regulation (EC) No. 1272/2008 [CLP]

Flammable liquids, Category 2	H225
Serious eye damage/eye irritation, Category 2	H319
Specific target organ toxicity — Single exposure, Category 3, Narcosis	H336

Full text of H statements : see section 16

Classification according to Directive 67/548/EEC [DSD] or 1999/45/EC [DPD]

F; R11
Xi; R36
R67

Full text of R-phrases: see section 16

Adverse physicochemical, human health and environmental effects

No additional information available

2.2. Label elements

Labelling according to Regulation (EC) No. 1272/2008 [CLP]

Hazard pictograms (CLP) :

GHS02 GHS08 GHS07

Signal word (CLP)	:	Danger
Hazard statements (CLP)	:	H225 - Highly flammable liquid and vapour. H319 - Causes serious eye irritation. H336 - May cause drowsiness or dizziness.
Precautionary statements (CLP)	:	P210 - Keep away from heat, hot surfaces, sparks, open flames and other ignition sources. No smoking. P261 - Avoid breathing vapours, dust, fume, gas. P305+P351+P338 - IF IN EYES: Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing.

2.3. Other hazards

No additional information available

SECTION 3: Composition/information on ingredients

3.1. Substances

Name	:	iso-PROPYL ALCOHOL For Synthesis
CAS-No.	:	67-63-0
EC-No.	:	200-661-7
EC Index-No.	:	603-117-00-0

Full text of R- and H-statements: see section 16

3.2. Mixtures

Not applicable

SECTION 4: First aid measures

4.1. Description of first aid measures

First-aid measures after inhalation	:	Remove person to fresh air and keep comfortable for breathing. Call a POISON CENTER/doctor if you feel unwell.
First-aid measures after skin contact	:	Get medical advice/attention. Wash with plenty of water/….
First-aid measures after eye contact	:	Rinse cautiously with water for several minutes. Remove contact lenses, if present and easy to do. Continue rinsing. Get medical advice/attention.
First-aid measures after ingestion	:	Rinse mouth. Do not induce vomiting. Get medical advice/attention.

4.2. Most important symptoms and effects, both acute and delayed

Symptoms/effects after inhalation	:	May cause drowsiness or dizziness.
Symptoms/effects after eye contact	:	Causes serious eye irritation.

4.3. Indication of any immediate medical attention and special treatment needed

Treat symptomatically.

SECTION 5: Firefighting measures

5.1. Extinguishing media

Suitable extinguishing media	:	Carbon dioxide. Dry powder. Foam. Water spray.
Unsuitable extinguishing media	:	Do not use extinguishing media containing water.

5.2. Special hazards arising from the substance or mixture

Fire hazard	:	Highly flammable liquid and vapour.
Explosion hazard	:	May form flammable/explosive vapour-air mixture.

5.3. Advice for firefighters

Protection during firefighting	:	Do not enter fire area without proper protective equipment, including respiratory protection.

SECTION 6: Accidental release measures

6.1. Personal precautions, protective equipment and emergency procedures

General measures	:	Remove ignition sources. Use special care to avoid static electric charges. No open flames. No smoking.

6.1.1. For non-emergency personnel

Emergency procedures	:	Evacuate unnecessary personnel.

6.1.2. For emergency responders

Protective equipment	:	Use personal protective equipment as required.
Emergency procedures	:	Ventilate area.

6.2. Environmental precautions

Avoid release to the environment.

6.3. Methods and material for containment and cleaning up

Methods for cleaning up	:	On land, sweep or shovel into suitable containers. Collect spillage.

6.4. Reference to other sections

No additional information available

SECTION 7: Handling and storage

7.1. Precautions for safe handling

Additional hazards when processed	:	Handle empty containers with care because residual vapours are flammable.
Precautions for safe handling	:	Keep away from sources of ignition - No smoking. Use only non-sparking tools. Avoid contact with skin and eyes. Use only outdoors or in a well-ventilated area. Do not breathe vapours.
Hygiene measures	:	Do not eat, drink or smoke when using this product. Wash hands and other exposed areas with mild soap and water before eating, drinking or smoking and when leaving work.

7.2. Conditions for safe storage, including any incompatibilities

Technical measures	:	Proper grounding procedures to avoid static electricity should be followed. Ground/bond container and receiving equipment.
Storage conditions	:	Keep in fireproof place. Keep container tightly closed.
Incompatible materials	:	Heat sources.

7.3. Specific end use(s)

No additional information available

SECTION 8: Exposure controls/personal protection

8.1. Control parameters

No additional information available

8.2. Exposure controls

Hand protection	:	Protective gloves
Eye protection	:	Chemical goggles or safety glasses
Skin and body protection	:	Wear suitable protective clothing
Respiratory protection	:	Where exposure through inhalation may occur from use, respiratory protection equipment is recommended

SECTION 9: Physical and chemical properties

9.1. Information on basic physical and chemical properties

Physical state	:	Liquid
Molecular mass	:	60.1 g/mol
Colour	:	Clear Colorless.
Odour	:	alcoholic odour.
Odour threshold	:	No data available
pH	:	No data available
Relative evaporation rate (butylacetate=1)	:	2.83
Melting point	:	-89 °C
Freezing point	:	No data available
Boiling point	:	82 °C

Flash point	:	12 °C
Auto-ignition temperature	:	399 °C
Decomposition temperature	:	No data available
Flammability (solid, gas)	:	Flammable Highly flammable liquid and vapour.
Vapour pressure	:	43.2 hPa at 20°C
Relative vapour density at 20 °C	:	2.1
Relative density	:	No data available
Density	:	0.79 g/cm³
Solubility	:	Water: Miscible in water
Log Pow	:	0.05
Viscosity, kinematic	:	No data available
Viscosity, dynamic	:	No data available
Explosive properties	:	No data available
Oxidising properties	:	No data available
Explosive limits	:	0.02 - 0.127 vol %

9.2. Other information

No additional information available

SECTION 10: Stability and reactivity

10.1. Reactivity

No additional information available

10.2. Chemical stability

Stable under normal conditions.

10.3. Possibility of hazardous reactions

No additional information available

10.4. Conditions to avoid

Open flame. Heat. Sparks.

10.5. Incompatible materials

No additional information available

10.6. Hazardous decomposition products

May release flammable gases.

SECTION 11: Toxicological information

11.1. Information on toxicological effects

Acute toxicity	:	Not classified
Skin corrosion/irritation	:	Not classified
Serious eye damage/irritation	:	Causes serious eye irritation.

Respiratory or skin sensitisation	:	Not classified
Germ cell mutagenicity	:	Not classified
Carcinogenicity	:	Not classified
Reproductive toxicity	:	Not classified
STOT-single exposure	:	May cause drowsiness or dizziness.
STOT-repeated exposure	:	Not classified
Aspiration hazard	:	Not classified

SECTION 12: Ecological information

12.1. Toxicity
No additional information available

12.2. Persistence and degradability
No additional information available

12.3. Bioaccumulative potential

iso-PROPYL ALCOHOL For Synthesis (67-63-0)	
Log Pow	0.05

12.4. Mobility in soil
No additional information available

12.5. Results of PBT and vPvB assessment
No additional information available

12.6. Other adverse effects
No additional information available

SECTION 13: Disposal considerations

13.1. Waste treatment methods

Product/Packaging disposal recommendations	:	Dispose of contents/container to hazardous or special waste collection point, in accordance with local, regional, national and/or international regulation.
Additional information	:	Handle empty containers with care because residual vapours are flammable.

SECTION 14: Transport information

In accordance with ADR / RID / IMDG / IATA / ADN

14.1. UN number

UN-No. (ADR)	:	1219

UN-No. (IMDG)	:	1219
UN-No. (IATA)	:	1219
UN-No. (ADN)	:	1219
UN-No. (RID)	:	1219

14.2. UN proper shipping name

Proper Shipping Name (ADR)	:	ISOPROPANOL (ISOPROPYL ALCOHOL)
Proper Shipping Name (IMDG)	:	ISOPROPANOL (ISOPROPYL ALCOHOL)
Proper Shipping Name (IATA)	:	Isopropanol
Proper Shipping Name (ADN)	:	ISOPROPANOL (ISOPROPYL ALCOHOL)
Proper Shipping Name (RID)	:	ISOPROPANOL (ISOPROPYL ALCOHOL)
Transport document description (ADR)	:	UN 1219 ISOPROPANOL (ISOPROPYL ALCOHOL), 3, II, (D/E)
Transport document description (IMDG)	:	UN 1219 ISOPROPANOL (ISOPROPYL ALCOHOL), 3, II (12°C c.c.)
Transport document description (IATA)	:	UN 1219 Isopropanol, 3, II
Transport document description (ADN)	:	UN 1219 ISOPROPANOL (ISOPROPYL ALCOHOL), 3, II
Transport document description (RID)	:	UN 1219 ISOPROPANOL (ISOPROPYL ALCOHOL), 3, II

14.3. Transport hazard class(es)

ADR

Transport hazard class(es) (ADR) : 3

Danger labels (ADR) : 3

:


IMDG

Transport hazard class(es) (IMDG) : 3

Danger labels (IMDG) : 3

:


IATA

Transport hazard class(es) (IATA) : 3

Hazard labels (IATA) : 3

:


ADN

Transport hazard class(es) (ADN) : 3

Danger labels (ADN) : 3

:


RID

Transport hazard class(es) (RID)	:	3
Danger labels (RID)	:	3
	:	

14.4. Packing group

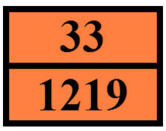
Packing group (ADR)	:	II
Packing group (IMDG)	:	II
Packing group (IATA)	:	II
Packing group (ADN)	:	II
Packing group (RID)	:	II

14.5. Environmental hazards

Dangerous for the environment	:	No
Marine pollutant	:	No
Other information	:	No supplementary information available

14.6. Special precautions for user

- Overland transport

Classification code (ADR)	:	F1
Special provisions (ADR)	:	601
Limited quantities (ADR)	:	1l
Excepted quantities (ADR)	:	E2
Packing instructions (ADR)	:	P001, IBC02, R001
Mixed packing provisions (ADR)	:	MP19
Portable tank and bulk container instructions (ADR)	:	T4
Portable tank and bulk container special provisions (ADR)	:	TP1
Tank code (ADR)	:	LGBF
Vehicle for tank carriage	:	FL
Transport category (ADR)	:	2
Special provisions for carriage - Operation (ADR)	:	S2, S20
Hazard identification number (Kemler No.)	:	33
Orange plates	:	
Tunnel restriction code (ADR)	:	D/E
EAC code	:	•2YE

- Transport by sea

Limited quantities (IMDG)	:	1 L
Excepted quantities (IMDG)	:	E2
Packing instructions (IMDG)	:	P001
IBC packing instructions (IMDG)	:	IBC02
Tank instructions (IMDG)	:	T4
Tank special provisions (IMDG)	:	TP1

EmS-No. (Fire)	:	F-E
EmS-No. (Spillage)	:	S-D
Stowage category (IMDG)	:	B
Flash point (IMDG)	:	12°C c.c.
MFAG-No	:	129

- Air transport

PCA Excepted quantities (IATA)	:	E2
PCA Limited quantities (IATA)	:	Y341
PCA limited quantity max net quantity (IATA)	:	1L
PCA packing instructions (IATA)	:	353
PCA max net quantity (IATA)	:	5L
CAO packing instructions (IATA)	:	364
CAO max net quantity (IATA)	:	60L
Special provisions (IATA)	:	A180
ERG code (IATA)	:	3L

- Inland waterway transport

Classification code (ADN)	:	F1
Special provisions (ADN)	:	601
Limited quantities (ADN)	:	1 L
Excepted quantities (ADN)	:	E2
Carriage permitted (ADN)	:	T
Equipment required (ADN)	:	PP, EX, A
Ventilation (ADN)	:	VE01
Number of blue cones/lights (ADN)	:	1

- Rail transport

Classification code (RID)	:	F1
Special provisions (RID)	:	601
Limited quantities (RID)	:	1L
Excepted quantities (RID)	:	E2
Packing instructions (RID)	:	P001, IBC02, R001
Mixed packing provisions (RID)	:	MP19
Portable tank and bulk container instructions (RID)	:	T4
Portable tank and bulk container special provisions (RID)	:	TP1
Tank codes for RID tanks (RID)	:	LGBF
Transport category (RID)	:	2
Colis express (express parcels) (RID)	:	CE7
Hazard identification number (RID)	:	33

14.7. Transport in bulk according to Annex II of MARPOL 73/78 and the IBC Code

Not applicable

SECTION 15: Regulatory information

15.1. Safety, health and environmental regulations/legislation specific for the substance or mixture

15.1.1. EU-Regulations

No REACH Annex XVII restrictions

iso-PROPYL ALCOHOL For Synthesis is not on the REACH Candidate List

iso-PROPYL ALCOHOL For Synthesis is not on the REACH Annex XIV List

15.1.2. National regulations

Germany

Reference to AwSV	: Water hazard class (WGK) 1, Slightly hazardous to water (Classification according to AwSV; ID No. 135)
12th Ordinance Implementing the Federal Immission Control Act - 12.BImSchV	: Is not subject of the 12. BlmSchV (Hazardous Incident Ordinance)

Netherlands

SZW-lijst van kankerverwekkende stoffen	: The substance is not listed
SZW-lijst van mutagene stoffen	: The substance is not listed
NIET-limitatieve lijst van voor de voortplanting giftige stoffen – Borstvoeding	: The substance is not listed
NIET-limitatieve lijst van voor de voortplanting giftige stoffen – Vruchtbaarheid	: The substance is not listed
NIET-limitatieve lijst van voor de voortplanting giftige stoffen – Ontwikkeling	: The substance is not listed

Denmark

Class for fire hazard	: Class I-1
Store unit	: 1 liter
Classification remarks	: F <Flam. Liq. 2>; Emergency management guidelines for the storage of flammable liquids must be followed
Recommendations Danish Regulation	: Young people below the age of 18 years are not allowed to use the product

15.2. Chemical safety assessment

No additional information available

SECTION 16: Other information

Full text of R-, H- and EUH-statements:

Eye Irrit. 2	Serious eye damage/eye irritation, Category 2
Flam. Liq. 2	Flammable liquids, Category 2
STOT SE 3	Specific target organ toxicity — Single exposure, Category 3, Narcosis
H225	Highly flammable liquid and vapour.
H319	Causes serious eye irritation.
H336	May cause drowsiness or dizziness.
R11	Highly flammable
R36	Irritating to eyes
R67	Vapours may cause drowsiness and dizziness
F	Highly flammable
Xi	Irritant

This information is based on our current knowledge and is intended to describe the product for the purposes of health, safety and environmental requirements only. It should not therefore be construed as guaranteeing any specific property of the product.

Technical Data Sheet

SYLGARD™ 184 Silicone Elastomer

FEATURES & BENEFITS

- Flowable
- Room temperature and heat cure
- Good dielectric properties
- Rapid, versatile cure processing controlled by temperature
- High transparency allows easy inspection of components

COMPOSITION

- Two-part
- 10 to 1 mix ratio
- Polydimethylsiloxane elastomer

Transparent encapsulant with good flame resistance

APPLICATIONS

SYLGARD™ 184 Silicone Elastomer is suitable for:

- LED Lighting encapsulation
- Power supplies
- Connectors
- Sensors
- Industrial controls
- Transformers
- Amplifiers
- High voltage resistor packs
- Relays
- Adhesive/encapsulant for solar cells
- Adhesive handling beam lead integrated circuits during processing

TYPICAL PROPERTIES

Specification Writers: These values are not intended for use in preparing specifications.

Property	Unit	Result
One or Two Part		Two
Color		Colorless
Viscosity (Base)	cP	5100
	Pa-sec	5.1
Viscosity (Mixed)	cP	3500
	Pa-sec	3.5
Thermal Conductivity	btu/hr ft °F	0.15
	W/m °K	0.27
Specific Gravity (Cured)		1.03
Working Time at 25°C (Pot Life - Hours)	hours	1.5
Cure Time at 25°C	hours	48
Heat Cure Time at 100°C	minutes	35
Heat Cure Time at 125°C	minutes	20
Heat Cure Time at 150°C	minutes	10
Durometer Shore		43
Dielectric Strength	volts/mil	500
	kV/mm	19

TYPICAL PROPERTIES (Continued)

Property	Unit	Result
Volume Resistivity	ohm*cm	2.9E+14
Dissipation Factor at 100 Hz		0.00257
Dissipation Factor at 100kHz		0.00133
Dielectric Constant at 100 Hz		2.72
Dielectric Constant at 100 kHz		2.68
Linear CTE (by DMA)	ppm/°C	340
Tensile Strength	PSI	980
	MPa	6.7
	Kg/cm^2	69
Refractive Index	@ 589 nm	1.4118
Refractive Index	@ 632.8 nm	1.4225
Refractive Index	@1321 nm	1.4028
Refractive Index	@ 1554 nm	1.3997
UL RTI Rating	°C	150

DESCRIPTION

Dow silicone 10 to 1 encapsulants are supplied as two-part liquid component kits. When liquid components are thoroughly mixed, the mixture cures to a flexible elastomer, which is well suited for the protection of electrical/PCB system assembly applications. Dow silicone encapsulants cure without exotherm at a constant rate regardless of sectional thickness or degree of confinement.

Dow silicone elastomers require no post cure and can be placed in service immediately following the completion of the cure schedule. Standard silicone encapsulants require a surface treatment with a primer in addition to good cleaning for adhesion while primerless silicone encapsulants require only good cleaning.

APPLICATION METHODS

- Automated metered mixing and dispensing
- Manual mixing

MIXING AND DE-AIRING

The 10 to 1 mix ratio these products are supplied in gives one latitude to tune the modulus and hardness for specific application needs and production lines. In most cases de-airing is not required.

PREPARING SURFACES

In applications requiring adhesion, priming will be required for many of the silicone encapsulants. For best results, the primer should be applied in a very thin, uniform coating and then wiped off after application. After application, it should be thoroughly cured prior to application of the silicone elastomer. Additional instructions for primer usage can be found in the information sheets specific to the individual primers.

PROCESSING/CURING

Thoroughly mixed Dow silicone encapsulant may be poured/dispensed directly into the container in which it is to be cured. Care should be taken to minimize air entrapment. When practical, pouring/dispensing should be done under vacuum, particularly if the component being potted or encapsulated has many small voids. If this technique cannot be used, the unit should be evacuated after the silicone encapsulant has been poured/dispensed. Dow silicone encapsulants may be either room temperature (25°C/77°F) or heat cured. Room temperature cure encapsulants may also be heat accelerated for faster cure. Ideal cure conditions for each product are given in the product selection table.

POT LIFE AND CURE RATE

Cure reaction begins with the mixing process. Initially, cure is evidenced by a gradual increase in viscosity, followed by gelation and conversion to a solid elastomer. Pot life is defined as the time required for viscosity to double after base and curing agent are mixed and is highly temperature and application dependent. Please refer to the data table.

USEFUL TEMPERATURE RANGES

For most uses, silicone elastomers should be operational over a temperature range of -45 to 200°C (-49 to 392°F) for long periods of time. However, at both the low and high temperature ends of the spectrum, behavior of the materials and performance in particular applications can become more complex and require additional considerations and should be adequately tested for the particular end-use environment. For low-temperature performance, thermal cycling to conditions such as -55°C (-67°F) may be possible, but performance should be verified for your parts or assemblies. Factors that may influence performance are configuration and stress sensitivity of components, cooling rates and hold times, and prior temperature history. At the high-temperature end, the durability of the cured silicone elastomer is time and temperature dependent. As expected, the higher the temperature, the shorter the time the material will remain useable.

COMPATIBILITY

Certain materials, chemicals, curing agents and plasticizers can inhibit the cure of addition cure gels. Most notable of these include: Organotin and other organometallic compounds, silicone rubber containing organotin catalyst, sulfur, polysulfides, polysulfones or other sulfur containing materials, unsaturated hydrocarbon plasticizers, and some solder flux residues. If a substrate or material is questionable with respect to potentially causing inhibition of cure, it is recommended that a small scale compatibility test be run to ascertain suitability in a given application. The presence of liquid or uncured product at the interface between the questionable substrate and the cured gel indicates incompatibility and inhibition of cure.

REPAIRABILITY

In the manufacture of electrical devices and PCB system assemblies it is often desirable to salvage or reclaim damaged or defective units. With most non-silicone rigid potting/ encapsulating materials, removal or entry is difficult or impossible without causing excessive damage to internal circuitry. Dow silicone encapsulants can be selectively removed with relative ease, depending on the chosen remove method and technique and repairs or changes accomplished, and the repaired area repotted in place with additional product. To remove silicone elastomers, simply cut with a sharp blade or knife and tear and remove unwanted material from the area to be repaired. Sections of the adhered elastomer are best removed from substrates and circuitry by mechanical action such as scraping or rubbing and can be assisted by applying DOWSIL™ OS Fluids to swell the elastomer. Before applying additional encapsulant to a repaired device, roughen the exposed surfaces of the cured encapsulant with an abrasive paper and rinse with a suitable solvent and dry. This will enhance adhesion and permit the repaired material to become an integral matrix with the existing encapsulant. Silicone prime coats are not recommended for adhering products to themselves.

PACKAGING INFORMATION

Multiple packaging sizes are available for this product.

USABLE LIFE AND STORAGE

Shelf life is indicated by the "Use Before" date found on the product label. Refer to the product label for storage temperature requirements. Special precautions must be taken to prevent moisture from contacting these materials. Containers should be kept tightly closed and head or air space minimized. Partially filled containers should be purged with dry air or other gases, such as nitrogen.

HANDLING PRECAUTIONS

PRODUCT SAFETY INFORMATION REQUIRED FOR SAFE USE IS NOT INCLUDED IN THIS DOCUMENT. BEFORE HANDLING, READ PRODUCT AND SAFETY DATA SHEETS AND CONTAINER LABELS FOR SAFE USE, PHYSICAL AND HEALTH HAZARD INFORMATION. THE SAFETY DATA SHEET IS AVAILABLE ON THE DOW WEBSITE AT WWW.CONSUMER.DOW.COM, OR FROM YOUR DOW SALES APPLICATION ENGINEER, OR DISTRIBUTOR, OR BY CALLING DOW CUSTOMER SERVICE.

LIMITATIONS

This product is neither tested nor represented as suitable for medical or pharmaceutical uses.

HEALTH AND ENVIRONMENTAL INFORMATION

To support customers in their product safety needs, Dow has an extensive Product Stewardship organization and a team of product safety and regulatory compliance specialists available in each area.

For further information, please see our website, www.consumer.dow.com or consult your local Dow representative.

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Your exclusive remedy for breach of such warranty is limited to refund of purchase price or replacement of any product shown to be other than as warranted.

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Form No. 11-3184-01 C

APPENDIX 2: CODE 1

```
#include <ESP8266HTTPClient.h>
#include <ESP8266WiFi.h>
#include <ArduinoJson.h>

HTTPClient http;    //Declare object of class HTTPClient
int i = 10;

void setup() {

  Serial.begin(9600);                              //Serial connection
  pinMode(0, INPUT); // Setup for leads off detection LO +
  pinMode(2, INPUT); // Setup for leads off detection LO -
  WiFi.begin("ERROR404", "24949040");   //WiFi connection

  while (WiFi.status() != WL_CONNECTED) {  //Wait for the WiFI connection completion

    delay(500);
    Serial.println("Waiting for connection");

  }

}

void loop() {

  if (WiFi.status() == WL_CONNECTED) { //Check WiFi connection status
```

```
    StaticJsonBuffer<300> JSONbuffer;   //Declaring static JSON buffer
    JsonObject& JSONencoder = JSONbuffer.createObject();

    if((digitalRead(0) == 1)||(digitalRead(2) == 1)){
      i+=1;
      if(i==20){i=15;}
      JSONencoder["key"] = i;
      }
    else{
      JSONencoder["key"] = analogRead(17);
     }
    char JSONmessageBuffer[300];
    JSONencoder.prettyPrintTo(JSONmessageBuffer, sizeof(JSONmessageBuffer));
    Serial.println(JSONmessageBuffer);
    http.begin("http://3.22.208.231/api/insert");      //Specify request destination
    http.addHeader("Content-Type", "application/json");  //Specify content-type header
    int httpCode = http.POST(JSONmessageBuffer);   //Send the request
//   String payload = http.getString();                                    //Get the response payload
//   Serial.println(httpCode);   //Print HTTP return code
//   Serial.println(payload);    //Print request response payload
    http.end();  //Close connection
  } else {
    Serial.println("Error in WiFi connection");

  }

}
```

APPENDIX 2: CODE 2

```
package com.bani.ecggraph;

import androidx.appcompat.app.AppCompatActivity;

import android.graphics.Color;
import android.os.Bundle;
import android.os.Handler;
import android.util.Log;
import android.widget.Toast;

import com.android.volley.Request;
import com.android.volley.RequestQueue;
import com.android.volley.Response;
import com.android.volley.VolleyError;
import com.android.volley.toolbox.JsonObjectRequest;
import com.android.volley.toolbox.Volley;
import com.github.mikephil.charting.charts.LineChart;
import com.github.mikephil.charting.components.Legend;
import com.github.mikephil.charting.components.XAxis;
import com.github.mikephil.charting.components.YAxis;
import com.github.mikephil.charting.data.Entry;
import com.github.mikephil.charting.data.LineData;
import com.github.mikephil.charting.data.LineDataSet;
import com.github.mikephil.charting.interfaces.datasets.ILineDataSet;
import com.github.mikephil.charting.listener.OnChartGestureListener;
import
com.github.mikephil.charting.listener.OnChartValueSelectedListener;

import org.json.JSONArray;
import org.json.JSONException;
import org.json.JSONObject;

import java.util.ArrayList;

public class MainActivity extends AppCompatActivity {

    public static final String TAG = "MainActivity";
    private LineChart lineChart;
    public static String dataY;
    public static int i;
    public static ArrayList<Entry> yValues = new ArrayList<>();
    public static boolean isDrawn;
    public static int j=0;
    public static String newApi;
    private RequestQueue requestQueue;
    @Override
    protected void onCreate(Bundle savedInstanceState) {
        super.onCreate(savedInstanceState);
```

```
        setContentView(R.layout.activity_main);
        lineChart = findViewById(R.id.chart);
//        lineChart.setOnChartGestureListener(MainActivity.this);
//
lineChart.setOnChartValueSelectedListener(MainActivity.this);

        newApi = getIntent().getExtras().getString("Value");
        Toast.makeText(this, newApi, Toast.LENGTH_SHORT).show();
        requestQueue = Volley.newRequestQueue(this);
        fetchData();
        isDrawn = true;
        final Handler handler = new Handler();
        final int delay = 1; //milliseconds

        handler.postDelayed(new Runnable(){
            int lol;
            public void run(){
                fetchData();
                lol++;
                //Log.d(TAG, "run: Fetched! "+lol+" data is:
"+yValues); //Just for logging purpose
                handler.postDelayed(this, delay);
            }
        }, delay);

    }

    public void fetchData(){
        String apiUrl;
        if (!newApi.isEmpty()){
            apiUrl = newApi;
        } else {
            apiUrl = "http://3.22.208.231/api/get/latest";
        }
        JsonObjectRequest jsonObjectRequest = new
JsonObjectRequest(Request.Method.GET, apiUrl, null, new
Response.Listener<JSONObject>() {
            @Override
            public void onResponse(JSONObject response) {
                try {
                    JSONArray jsonArray =
response.getJSONArray("data");
                    if (isDrawn){
                        lineChart.invalidate();
                        lineChart.clear();
                    }
                    for (i = 0; i<jsonArray.length(); i++){
                        JSONObject jsonObject =
jsonArray.getJSONObject(i);
                        dataY = jsonObject.getString("key");
                        yValues.add(new Entry(j,
Float.parseFloat(dataY)));
```

```
                    j=j+10;
                }
                lineChart.setDragEnabled(true);
                lineChart.setScaleEnabled(false);

                LineDataSet set1 = new LineDataSet(yValues,
"ECG");
                set1.setFillAlpha(110);
                set1.setColor(Color.RED);
                set1.setLineWidth(1f);
                set1.setDrawCircles(false);
                Legend legend = lineChart.getLegend();
                legend.setEnabled(false);
                set1.setDrawValues(false);
                lineChart.setDrawBorders(false);
                lineChart.setDrawGridBackground(false);
                lineChart.getAxisRight().setDrawLabels(false);
                lineChart.getDescription().setEnabled(false);

lineChart.getXAxis().setPosition(XAxis.XAxisPosition.BOTTOM);
                lineChart.getXAxis().setLabelCount(10);
//                float minXRange = 10;
//                float maxXRange = 0;
//                lineChart.setVisibleXRange(minXRange,
maxXRange);


                ArrayList<ILineDataSet> dataSets = new
ArrayList<>();
                dataSets.add(set1);

                LineData data = new LineData(dataSets);
                if (set1.getEntryCount() >= 50) {
                    set1.removeFirst();
                    for (int i = 0; i < set1.getEntryCount(); i++)
{
                        Entry entryToChange =
set1.getEntryForIndex(i);
                        entryToChange.setX(entryToChange.getX() -
1);
                    }
                }
                    lineChart.setData(data);

            } catch (JSONException e) {
                e.printStackTrace();
            }
        }
    }, new Response.ErrorListener() {
        @Override
        public void onErrorResponse(VolleyError error) {
            error.printStackTrace();
```

```
            }
        });
        requestQueue.add(jsonObjectRequest);
    }
}


package com.bani.ecggraph;

import org.junit.Test;

import static org.junit.Assert.*;

/**
 * Example local unit test, which will execute on the development
machine (host).
 *
 * @see <a href="http://d.android.com/tools/testing">Testing
documentation</a>
 */
public class ExampleUnitTest {
    @Test
    public void addition_isCorrect() {
        assertEquals(4, 2 + 2);
    }
```

APPENDIX 2: CODE 3

```
package com.bani.ecggraph;

import androidx.appcompat.app.AppCompatActivity;

import android.content.Intent;
import android.os.Bundle;
import android.view.View;
import android.widget.EditText;

public class GetApiActivity extends AppCompatActivity {
    public static String newApi;
    EditText editText;
    @Override
    protected void onCreate(Bundle savedInstanceState) {
        super.onCreate(savedInstanceState);
        setContentView(R.layout.activity_get_api);

        editText = findViewById(R.id.getapi);
    }

    public void getApi(View view){
        newApi = editText.getText().toString();
        Intent intent = new Intent(GetApiActivity.this,
MainActivity.class);
        intent.putExtra("Value", newApi);
        startActivity(intent);
    }
}
```