CHAPTER IV
MATERIALS AND METHODS

4.1 Introduction

The details about the materials taken and chemicals used for the preparation of chitosan-ZnO and its graphene oxide hybrid composites were given in this chapter. The experimental procedure for chitin extraction and preparation of chitosan-ZnO complex, chitosan-ZnO nanostructures and chitin/chitosan based graphene oxide-ZnO hybrid composites were given in detail.

Also the subsequent sections have represent the various characterizations were used to characterize the prepared samples.

4.2 Materials

4.2.1 Chemicals

The analytical grade reagents such as zinc chloride salt, hydrochloric acid, sulfuric acid and acetic acid, sodium hydroxide, potassium permanganate and hydrogen peroxide, solvents acetone and ethanol were used for the preparation of all the samples. Graphite powder was used for preparation of graphene oxide solution. Doubly deionized distilled water was used throughout the experiment.

4.2.2 Commercial chitosan

Chitosan was purchased from M/s South India Sea Foods, Kochi, Kerala, India. It was extracted from crustacean exoskeletons, had an average molecular
weight of 180 kDa and was 90% deacetylated. The chitosan is designated as CCS.

4.2.3 Chitin extraction

The collected crab shells were washed in seawater, drained off and subsequently dried in sunlight for one day. The specimens were then shade dried, crushed and kept in airtight containers. The 50g of crushed powder was weighed and chitin was extracted from the shells using hydrochloric acid solution. The extracted chitin was subjected to heat maintained at 40ºC with constant stirring up to 24 h. Then the sample was washed distilled water for removing acid and filtered till the washed water pH has reached at neutral. The filtrate was then dried in oven at 60ºC by oven. Deproteinization was carried out using NaOH solution. The flasks were kept on a shaker at 110 rpm for 24 h at 37ºC for deproteinization. The sample was filtered and washed with distilled water; the filtrate was washed till it showed neutral pH. The precipitate was then dried in oven at 60ºC and the resultant product of chitin was collected for analysis [183].

4.2.4 Preparation of chitosan

The chitosan was prepared from extracted chitin by deacetylation. First, the extracted chitin was treated with NaOH 45% (w/v) at 70ºC for 2 h and the formed chitosan samples were purified by dissolving in 2% acetic acid. The supernatant solution was discarded, and the remaining formed precipitate was allowed to settle for 24 h by adding 200 parts of distilled water. The clear solution was removed and then remaining suspension filtered using suction pump and dried in the oven at 110ºC for 1h. This sample is designated as PCS.
4.3 Preparation of chitosan-ZnO complex

4.3.1 N30 complex

The extracted chitin was treated with a mixture of ZnCl₂ and NaOH with the proportions of 15% and 30%, respectively at 70°C for 2h and the formed chitosan-ZnO samples were purified by dissolving in 2% acetic acid. The supernatant solution was discarded, and the remaining formed precipitate was allowed to settle for 24 h by adding 200 parts of distilled water. The clear solution was removed and then remaining suspension filtered using suction pump and dried in the oven at 110°C for 1h. This sample is designated as N30.

4.3.2 N45 complex

The above process was repeated with increasing the sodium hydroxide concentration from 30% to 45% and this sample is designated as N45.

4.3.3. N60 complex

The above process was repeated with increasing the sodium hydroxide concentration from 45% to 60% and this sample is designated as N60.
Figure 4.1: Preparation setup of chitosan-ZnO complex
4.4 Preparation of chitosan-ZnO nanostructures

4.4.1 Z15N45 nanostructures

The extracted chitin (0.5 g) and 30 ml of ZnCl₂ 15% (W/V) were taken in 500 ml beaker and kept under constant stirring using magnetic stirrer. The temperature of the bath was slowly raised to 70ºC and kept for 2 h. Then 30 ml of prepared NaOH 45% (W/V) solution was added slowly, a pale yellow viscous solution turned to a white precipitate. The formed white precipitate was then allowed to settle for 24 h at room temperature. The supernatant solution was discarded, and the remaining suspension was washed 10 times (each time, the dilution of concentrated suspension was carried out with ratio of 1:30) and allowed to settle for 30 min. To collect the residue, it was filtered using suction pump and dried in an oven at 110ºC for 2 h. This sample is designated as Z15N45.

4.4.2 Z30N45 nanostructures

The above process was repeated with the concentration of 30% zinc chloride and 45% sodium hydroxide. This sample is designated as Z30N45.

4.4.3 Z30N30 nanostructures

The above process was repeated with the concentration of 30% zinc chloride and 30% sodium hydroxide. This sample is designated as Z30N30.
4.4.4 Z15N30 nanostructures

The above process was repeated with the concentration of 15% zinc chloride and 30% sodium hydroxide. This sample is designated as Z30N30.

4.5 Synthesis of graphene oxide–ZnO/chitosan hybrid composites

4.5.1. Graphene oxide

Graphene oxide (GO) was prepared from natural graphite powder by a modified Hummers and Offeman procedure [184]. Approximately 3 g of graphite powder was added to 12 ml of concentrated H₂SO₄ and kept at 80ºC for 5 h. After sonication at room temperature, the solution was filtered using a 200 nm porous filter to retrieve the pre-oxidized graphite powder. To exfoliate the pre-oxidized graphite powder into monolayer graphene sheets, 2 g of pre-oxidized graphite powder and 15 g of KMnO₄ were added into 120 ml of H₂SO₄ solution and stirred for 2 h, keeping the beaker in an ice-water bath to ensure the temperature remained below 10ºC. Then, 120 ml of H₂O₂ (30 wt% aqueous solution) was added to the above liquid, and the mixture was stirred for 2 h at room temperature. Finally, the resulting suspension was filtered and washed with 10% HCl then distilled water. The obtained graphene oxide was dispersed in distilled water to form a stable brown solution.
4.5.2 GO/ CH composite

Graphene oxide/chitin composite was prepared using the extracted chitin. 0.25 g was dissolved in 0.5% (v/v) acetic acid solution and then solution was added in 250 ml beaker and heated at 80ºC for 1 h with continuous stirring. Then already prepared 20 ml of GO solution mixed with 20 ml of deionized water was added to the solution at 80ºC for 2 h. Then micro-addition of 50 ml of 45% sodium hydroxide solution was added, and the precipitate was allowed to settle for 24 h. The washing process was repeated by discarding the supernatant solution and added the remaining suspension. To collect the residue, it was filtered the precipitate using suction pump and dried in a hot air oven at 110ºC for 2 h. This composite is designated as GO/CH.

4.5.3 GO/ CS composite

Graphene oxide / chitosan composites was prepared using 0.25 g of commercial chitosan was dissolved in 0.5% (v/v) acetic acid solution and then solution was added in 250 ml beaker and heated at 80ºC for 1 h with continuous stirring. Then already prepared 20 ml of GO solution mixed with 20 ml of deionized water was added to the solution at 80ºC for 2 h. Then micro-addition of 50 ml of 45% sodium hydroxide solution was added, and the precipitate was allowed to settle for 24 h. The washing process was repeated by discarding the supernatant solution and added the remaining suspension. To collect the
residue, it was filtered the precipitate using suction pump and dried in a hot air oven at 110ºC for 2h. This composite is designated as GO/CS.

4.5.4 GO-ZnO/CH hybrid composites

Chitin based graphene oxide-ZnO hybrid composite was prepared using the extracted chitin 0.25 g was dissolved in 0.5% (v/v) acetic acid solution, 50 ml of 15% zinc chloride solution was added in a 250 ml beaker and heated at 80ºC for 1 h with continuous stirring. Then, 20 ml of the prepared GO solution mixed with 20 ml of deionised water was added to the solution at 80ºC and heated for an additional 2 h. To initiate precipitation from the homogeneous brown color solution, 50 ml of 45% sodium hydroxide was added, and the precipitate was allowed to settle for 24 h. A washing process was performed 10 times by discarding the supernatant solution and adding fresh water to the remaining suspension. Each time, the dilution of the concentrated suspension (30:1 water: precipitate) was allowed to settle for 30 min. To collect the residue, it was filtered using a suction pump and dried in an oven at 110ºC for 2 h. This hybrid composite is designated GO-ZnO/CH.

4.5.5 GO-ZnO/CS hybrid composites

Chitosan based graphene oxide-ZnO hybrid composite was prepared from the above process was repeated with commercial chitosan instead of chitin, and this sample is designated as GO-ZnO/CS.
4.6 Characterizations

4.6.1 FTIR spectroscopy

FTIR is most useful for identifying chemicals that are either organic or inorganic. It can be utilized to quantitate some components of an unknown mixture. It can be applied to the analysis of solids, liquids, and gasses. In the present work, Fourier transform infrared (FT-IR) spectra were recorded on Nicolet 5700 instrument (Thermo-Nicolet-380 Madison, USA model) using KBr pellet in the range between 4,000 - 400 cm\(^{-1}\) at room temperature. The samples were thoroughly ground in a smooth agate mortar to form powered sample. 2 mg of this powdered sample was intimately mixed with 350 mg of pure, dry, powdered spectroscopic grade potassium bromide. The mixture of sample and potassium bromide was pressed with special dies under a pressure of 10,000 to 15,000 psi for 10 minutes in the hydraulic pressure and then released to get a transparent disk. The disk was loaded on to the FTIR spectral instrument to perform spectral analysis over a range of 400 - 4000 cm\(^{-1}\) and analyzed.

4.6.2 X-ray diffractrometry

X-ray diffraction (XRD) is a very powerful tool in the study of solid-state materials. The uniqueness of this technique is that it is a non-destructive technique and furnishes multiple results including crystal lattice details down to the atomic level. Coating electrodeposits and electrochemical reaction studies in chemistry, study of the structure of nucleic acids in biological sciences, crystal structural analysis in solid state physics materials science etc., are some of the
fields that have used the new potentials of this technique. In X-ray diffraction methods of analysis, single crystal diffractometer are essentially used to resolve the fundamental crystallographic details, while X-ray powder (polycrystalline) diffractometer rule the applied X-ray crystallography.

The crystallite size and phase structure of chitosan-ZnO complexes were measured using X-ray diffractometry (X’Pert PRO PANalytical diffractometry) using CuK$_{\alpha}$ radiation ($\theta = 0.15406$ nm) with the scanning rate of 0.01° /step with 2$\theta$ ranging from 10° to 80°.

4.6.3 SEM and EDAX analysis

The scanning electron microscope uses a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. The signals that derive from electron-sample interactions reveal information about the sample including external morphology, chemical composition, and crystalline structure and orientation of materials making up the sample. In most applications, data are collected over a selected area of the surface of the sample, and a 2-dimensional image is generated that displays spatial variations in these properties. Areas ranging from approximately 1 cm to 5 microns in width can be imaged in a scanning mode using conventional SEM techniques. The SEM is also capable of performing analyses of selected point locations on the sample; this approach is especially useful in qualitatively or semi-quantitatively determining chemical compositions, crystalline structure, and crystal orientations. The design and function of the SEM is very similar to the
EPMA and considerable overlap in capabilities exists between the two instruments.

Energy-dispersive X-ray spectroscopy (EDAX) is an analytical technique used for the elemental analysis or chemical characterization of a sample. It relies on the investigation of an interaction of some source of X-ray excitation and a sample. Its characterization capabilities are due in large part to the fundamental principle that each element has a unique atomic structure allowing unique set of peaks on its X-ray spectrum. To stimulate the emission of characteristic X-rays from a specimen, a high-energy beam of charged particles such as electrons or protons or a beam of X-rays, is focused into the sample being studied. At rest, an atom within the sample contains ground state (or unexcited) electrons in discrete energy levels or electron shells bound to the nucleus.

The incident beam may excite an electron in an inner shell, ejecting it from the shell while creating an electron hole where the electron was. An electron from an outer, higher-energy shell then fills the hole, and the difference in energy between the higher-energy shell and the lower energy shell may be released in the form of an X-ray. The number and energy of the X-rays emitted from a specimen can be measured by an energy-dispersive spectrometer. As the energy of the X-rays is characteristic of the difference in energy between the two shells, and of the atomic structure of the element from which they were emitted, this allows the elemental composition of the specimen to be measured
The surface morphology and chemical composition of the prepared chitosan and its complexes were depicted using scanning electron microscope (Hitachi S3000 HSEM).

### 4.6.4 UV-vis spectroscopy

Ultraviolet measures the intensity of light passing through a sample ($I$), and compares it to the intensity of light before it passes through the sample ($I_0$). The ratio $I / I_0$ are called the transmittance, and are usually expressed as a percentage ($\%T$). The absorbance, $A$, is based on the transmittance:

$$ A = - \log (\%T / 100\%) $$

The UV-vis absorption spectra of prepared chitosan and its complexes were recorded using UV-visible spectrophotometer (2401 PC model; Shimadzu, Kyoto, Japan), the wavelength of incident ray was selected in the range of 200-800 nm.

### 4.6.5 Photoluminescence spectroscopy

Photoluminescence is a process in which a substance absorbs photons (electromagnetic radiation) and then re-radiates photons. Quantum mechanically, this can be described as an excitation to a higher energy state and then a return to a lower energy state accompanied by the emission of a photon. This is one of many forms of luminescence (light emission) and is distinguished by photoexcitation (excitation by photons).
The period between absorption and emission is typically extremely short, in the order of 10 nanoseconds. Under special circumstances, however, this period can be extended into minutes or hours.

The simplest photoluminescent processes are resonant radiations, in which a photon of a particular wavelength is absorbed and an equivalent photon is immediately emitted. This process involves no significant internal energy transitions of the chemical substrate between absorption and emission and is extremely fast, of the order of 10 nanoseconds.

More interesting processes occur when the chemical substrate undergoes internal energy transitions before re-emitting the energy from the absorption event. The most familiar such effect is fluorescence, which is also typically a fast process, but in which some of the original energy is dissipated so that the emitted light photons are of lower energy than those absorbed. The generated photon in this case is said to be red shifted, referring to the loss of energy as Jablonski diagram shows.

Photoluminescence is an important technique for measuring the purity and crystalline quality of semiconductors such as GaAs and InP. Several variations of photoluminescence exist, including photoluminescence excitation (PLE).

Time-resolved photoluminescence (TRPL) is a method where the sample is excited with a light pulse and then the decay in photoluminescence with
respect to time is measured. This technique is useful in measuring the minority carrier lifetime of III-V semiconductors like Gallium arsenide (GaAs.)

An even more specialized form of photoluminescence is phosphorescence, in which the energy from absorbed photons undergoes intersystem crossing into a state of higher spin multiplicity, usually a triplet state. Once the energy is trapped in the triplet state, transition back to the lower singlet energy states is quantum mechanically forbidden, meaning that it happens much more slowly than other transitions. The result is a slow process of radiative transition back to the singlet state, sometimes lasting minutes or hours. This is the basis for "glow in the dark" substances.

The emission spectra of prepared chitosan and its complexes were measured using the room temperature photoluminescence spectra (Cary Eclipse fluorescence spectrophotometer) and in the range of 200-800 nm with an excitation at 325 nm.

4.6.6 TEM analysis

Electron Microscopes are scientific instruments that use a beam of highly energetic electrons to examine objects on a very fine scale. This examination can yield the information like topography, morphology, composition as well as crystallographic information’s. Working principle is exactly as their optical counterparts except that they use a focused beam of electrons instead of light to "image" the specimen and gain information as to its structure and composition.
The main use of this technique is to examine the specimen structure, composition or properties in submicroscopic details so that this microscopy technique is significantly involved in numerous fields. In TEM there is no change in the refractive index of the medium when the illumination beam is deflected, the vacuum in the lens is the same as the vacuum in the column.

Deflection is in this case only due to the electromagnetic properties of the lens which are defined by electromagnetic plates that are only able to influence the path direction of the electrons, since all of the electrons carry a negative charge [203]. Those electrons that pass through the sample go on to form the image while those that are stopped or deflected by dense atoms in the specimen are subtracted from the image. In this way a black and white image is formed and then remaining electrons which passes close to heavy atom and get only slightly deflected make their way down the column and contribute to the image.

There are three main reasons why the microscope column must be operated under very high vacuum. The first of these is to avoid collisions between electrons of the beam and stray molecules. Such collisions can result in a spreading or diffusing of the beam or more seriously can result in volatization event if the molecule is organic in nature. Such volatizations can severely contaminate the microscope column especially in finely machined regions such as apertures and pole pieces that will serve to degrade the image. The size and shape of the particles were analyzed using Transmission electron microscopy (PHILIPS CM200).
4.6.7 Cyclic Voltammetry (CV)

Cyclic voltammetry commonly known as “CV” is a very much popular and most extensively used electrochemical technique among the potential sweep techniques. It is one of the simplest and widely employed analytical tools used in the investigation of several processes in electrode/electrolyte interface [180, 181]. Cyclic voltammetry concerns with the scanning of working electrode potential between the potential limits of $V_1$ and $V_2$ at a known scan rate $v$, in both the forward and reverse direction and measuring the current of the electrochemical cell. The resultant current of the system involves the faradaic current due to the various electrochemical phenomenons occurring on the electrode surface such as electron transfer redox reactions and adsorption processes in addition to the capacitive current due to the double layer charging at these potentials.

The interpretation of cyclic voltammograms for semiconductor electrode position studies is often not straight forward owing to the combined influence of a number of processes involving absorption, electro deposition and coupled chemical reactions. A plot of measured current as a function of applied potential is known as “cyclic voltammogram”. It is an electrochemical spectrum indicating the potentials at which several processes occurring can be obtained rapidly and from the dependence of current on the sweep rate, the involvement of coupled homogenous reactions and the process like adsorption can also be recognized. Apart from these, the kinetic parameters and the mechanism of different
heterogeneous reactions occurring on the electrode surface can also be determined.

Normally the shape of the cyclic voltammogram depends on the type of redox reactions. The ratio of concentrations of oxidant and reductant species of a reversible reaction is given by Nernst equation and a concentration gradient exists within the region near the electrode surface known as Nernst diffusion layer, where the concentration gradient of the electroactive species is linear. Also the Nernstian equilibrium is always maintained at the electrode surface and at all the potentials. The cyclic voltammetric measurements were carried out in 1M HCl using the CHI 1022 electrochemical analyzer/workstation (CH Instruments, USA).

4.6.8 Impedance spectroscopy

Electrical impedance, or simply impedance, describes a measure of opposition to a sinusoidal alternating current (AC). Electrical impedance extends the concept of resistance to AC circuits, describing not only the relative amplitudes of the voltage and current, but also the relative phases. Impedance spectroscopy is a technique used in research and development because it involves relatively simple electrical measurements, which can be readily automated, and also the results may often be correlated with many complex material properties.

Impedance is a more general concept than the resistance because it takes phase difference into account, and it has become a fundamental branch of the
Impedance spectroscopy or AC spectroscopy is the study of the dielectric properties as a function of frequency. The general approach of impedance spectroscopy is to apply an electrical signal namely a known voltage or current to the electrodes and observe the resulting current or voltage. It is assumed that the properties of the electrode material are time invariant, and it is one of the basic purposes of the impedance spectroscopy to determine these properties and their inter relationship. The impedance/gain phase analyzer with a frequency ranges 10 MHz to 100 KHz and fully automated with a potential computer to study the electrical transport properties of the samples. The semicircle which is attributed to the parallel combination of double-layer capacitance ($C_{dl}$) and charge transfer resistance ($R_{ct}$) in series with a Warburg impedance / constant phase element. Electrochemical impedance spectroscopy (EIS) was carried out (Autolab) under atmospheric conditions, with AC signals of amplitude of 50 mV and a frequency range of 0.05 to $10^5$ HZ.

4.6.9 Raman spectroscopy

Raman spectroscopy is a spectroscopic technique used to observe vibrational, rotational, and other low-frequency modes in a system. It relies on inelastic scattering, or Raman scattering, of monochromatic light, usually from a laser in the visible, near infrared, or near ultraviolet range. The laser light interacts with molecular vibrations, phonons or other excitations in the system, resulting in the energy of the laser photons being shifted up or down. The shift in
energy gives information about the vibrational modes in the system. Infrared spectroscopy yields similar, but complementary, information.

Spontaneous Raman scattering is typically very weak, and as a result the main difficulty of Raman spectroscopy is separating the weak inelastically scattered light from the intense Rayleigh scattered laser light. Historically, Raman spectrometers used holographic gratings and multiple dispersion stages to achieve a high degree of laser rejection. In the past, photomultipliers were the detectors of choice for dispersive Raman setups, which resulted in long acquisition times. However, modern instrumentation almost universally employs notch or edge filters for laser rejection and spectrographs (axial Transmissive (AT), Czerny-Turner (CT) monochromatic, or FT (Fourier transform spectroscopy based), and CCD detectors.

If the final vibrational state of the molecule is more energetic than the initial state, then the emitted photon will be shifted to a lower frequency in order for the total energy of the system to remain balanced. This shift in frequency is designated as a Stokes shift. If the final vibrational state is less energetic than the initial state, then the emitted photon will be shifted to a higher frequency, and this is designated as an Anti-Stokes shift. Raman scattering is an example of inelastic scattering because of the energy transfer between the photons and the molecules during their interaction. The Raman spectra were measured in the range of 4000 - 400 cm\(^{-1}\) using (HORIBA Jobin Yvon) model Lab RAM HR800 Micro Raman spectrometer.
4.6.10 Atomic Force Microscopy (AFM)

Binning, Rohrer, Gerber and Weibel have developed Scanning Tunneling Microscope (STM) in 1982. Atomic Force Microscope (AFM) is an improvement of STM that was also developed by Binning and Quade. During the past 2 decades, the atomic force microscopy has been established very sensitive surface-imaging and probing instrument. Scanning Probe Microscopy (SPM) consists of a family of microscopy like STM, AFM, Magnetic Force Microscope (MFM), Electric Force Microscope (EFM) etc depending upon the kind of probe being used for scanning.

Main components in SPM instrument are Microscope, Controller, display monitor and control monitor. The main parts of the Microscope are the following: Microscope head that consists of tip holder, tip deflection detection system like laser diode and quad photo diode detector and associated optics. Tip holder has a cantilever with a nano tip mounted. Piezoelectric device scan the given sample along $x$, $y$ and $z$ direction of the sample. The AFM was analyzed using an XE70 model from Park Systems, Korea, and the microscopy was performed using the non-contact mode function.

4.6.11 Four- probe resistivity measurements

Four- probes is a simple apparatus for measuring the resistivity of semiconductor samples. By passing a current through two outer probes and measuring the voltage through the inner probes allows the measurement of the substrate resistivity.
The sheet resistivity of the top emitter layer is very easy to measure experimentally using a "four point probe". A current is passed through the outer probes and induces a voltage in the inner voltage probes. The junction between the n and p -type materials behaves as an insulating layer and the cell must be kept in the dark. In typical usage the current is set to 4.53 mA so that the resistivity is simply the voltage reading in mV. The measurement of bulk resistivity is similar to that of sheet resistivity except that a resistivity in cm$^{-3}$ is reported using the wafer thickness (t).

While simple in principle, there are experimental issues to take into account when using a four-point probe. In particular, the application of a metal to a semiconductor forms a schottky diode rather than an ohmic contact. Very high or very low resistivity samples require adjustment of the drive current to obtain a reliable reading. Samples with as cut or as lapped surfaces are easier to measure than samples with polished surfaces.

For high resistivity samples the current is reduced so as not to have an excessively larger voltage at the contacts. It is recommended that voltage on the inner probes be less than 100 mV / mm.

Low resistivity samples are usually much easier to measure as the contacts to the silicon are ohmic. For very low resistivity you will have to increase the current to 45.3 mA and set the voltmeter to a lower scale. For very low resistivity samples the current passing through the sample causes the resistance.
Electrical conductivity measurements were carried out using four probe resistivity methods by a Keithley DMM-2101 at room temperature.

4.6.12 Current-Voltage (I-V) technique

A current–voltage characteristic or I–V curve (current–voltage curve) is a relationship, typically represented as a chart or graph, between the electric current through a circuit, device, or material, and the corresponding voltage, or potential difference across it.

In electronics, the relationship between the direct current (DC) through an electronic device and the DC voltage across its terminals is called a current–voltage characteristic of the device. Electronic engineers use these charts to determine basic parameters of a device and to model its behavior in an electrical circuit. These characteristics are also known as I–V curves, referring to the standard symbols for current and voltage.

A more general form of current–voltage characteristic is one that describes the dependence of a terminal current on more than one terminal voltage difference; electronic devices such as vacuum tubes and transistors are described by such characteristics. The figure to the right shows a family of I–V curves for a MOSFET as a function of drain voltage with overvoltage \(V_{GS} - V_{th}\) as a parameter.

The simplest I–V characteristic involves a resistor, which according to Ohm's Law exhibits a linear relationship between the applied voltage and the resulting electric current. However, even in this case environmental factors such
as temperature or material characteristics of the resistor can produce a non-linear curve.

The trans-conductance and early voltage of a transistor are examples of parameters traditionally measured with the assistance of an I–V chart, or laboratory equipment that traces the charts in real time on an oscilloscope. I-V characterization studies were carried out using CHI 405A electrochemical workstation.

4.7 Conclusion

In this chapter, the chemical method of sample preparation for individual sample was discussed. The details about the characterization techniques were elaborately explained. The prepared samples, its designation and techniques used for characterization are summarized in the Table 4.1. The results of the test and discussions were given in the subsequent chapters.
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