2.1 Introduction

This chapter deliberates the complete description of materials and experimental methodologies for the synthesis of supramolecular metallogel which have been used for active electronic device fabrication. The current chapter also discusses about the various modern characterization techniques such as Fourier Transform Infrared (FTIR) Spectroscopy, UV-visible spectroscopy, X-Ray Diffraction (XRD), Transmission Electron Microscopy (TEM), Energy-Dispersive X-Ray Spectroscopy (EDAX). In addition to this, it further covers semiconducting properties of synthesized metallogel and also stated about the fabrication of M-S junction based Schottky barrier diode.

2.2. Materials

The present study is established the development of economically viable and energy efficient route for the metallogel synthesis. The required chemicals of this study were AR grade and used without any further alteration and listed in the **Table 2.1**. Throughout the whole experiments, ultrapure water was utilized

Table 2.1 list of chemicals

Name	Chemical formula	Physical state	Manufacturer
Succinic acid	$C_4H_6O_4$	Solid	Sigma Aldrich
Hydrazine hydrate	$N_2H_4.H_2O$	liquid	Sigma Aldrich
Trimesic acid	$C_9H_6O_6$	solid	Sigma Aldrich
Salicylaldehyde	$C_7H_6O_2$	liquid	S D Fine Chem Ltd.
2,6-Diaminopyridine	$C_5H_7N_3$	solid	Sigma Aldrich
Methanol	CH_3OH	liquid	S D Fine Chem Ltd.
Ethanol	C_2H_5OH	liquid	S D Fine Chem Ltd.
Isopropyl alcohol	$(CH_3)_2CHOH$	liquid	S D Fine Chem Ltd.

Acetone	$(CH_3)_2O$	liquid	S D Fine Chem Ltd.
Dimethyl sulphoxide	$(CH_3)_2SO$	liquid	S D Fine Chem Ltd.
Dimethylformamide	$HCO(CH_3)_2$	liquid	S D Fine Chem Ltd.
Tetrahydrofuran	C_4H_8O	liquid	S D Fine Chem Ltd.
Dimethyl sulphoxide-d_6	$(CD_3)_2SO$	liquid	S D Fine Chem Ltd.
Hydrochloric acid	HCl	liquid	S D Fine Chem Ltd.
Sulphuric acid	H_2SO_4	liquid	S D Fine Chem Ltd.
Nitric acid	HNO_3	liquid	S D Fine Chem Ltd.
Sodium chloride	$NaCl$	solid	Alfa Aesar
Sodium nitrate	$NaNO_3$	solid	S D Fine Chem Ltd.
Potassium nitrate	KNO_3	solid	S D Fine Chem Ltd.
Aluminium nitrate	$Al(NO_3)_3.9H_2O$	solid	S D Fine Chem Ltd.
Ferric Nitrate	$Fe(NO_3)_3.9H_2O$	solid	Sigma Aldrich
Ferrous chloride	$FeCl_3$	solid	Sigma Aldrich
Manganese nitrate	$Mn(NO_3)_2.6H_2O$	solid	Sigma Aldrich
Chromium nitrate	$Cr(NO_3)_3.9H_2O$	solid	Sigma Aldrich
Potassium dichromate	$K_2Cr_2O_7$	solid	Sigma Aldrich
Nickel nitrate	$Ni(NO_3)_2.6H_2O$	solid	Sigma Aldrich
Cobalt nitrate	$Co(NO_3)_2.6H_2O$	solid	Sigma Aldrich
Copper nitrate	$Cu(NO_3)_2.5\ H_2O$	solid	Sigma Aldrich
Zinc nitrate	$Zn(NO_3)_2\ .6\ H_2O$	solid	Sigma Aldrich
Cadmium nitrate	$Cd(NO_3)_2.4\ H_2O$	solid	Sigma Aldrich
Cadmium nitrate	$Cd(NO_3)_2.4\ H_2O$	solid	Sigma Aldrich
Mercuric nitrate	$Hg(NO_3)_2.\ H_2O$	solid	Sigma Aldrich
Lead nitrate	$Pb(NO_3)_2$	solid	Sigma Aldrich

2.3 Methods

2.3.1 Synthesis of ligands

The procedure for the synthesis of all ligands was mentioned in the respective chapter.

2.3.2 Synthesis of metallogels

The metallogels were synthesized either by introducing them to sonication or injection addition methods, which were mentioned in respective chapters.

2.4 Characterization of meatllogel

The as-prepared ligands and organometallogels were characterized by the various instrumentation technique which are deliberated briefly.

2.4.1 NMR spectroscopy

In the deuterated DMSO solvent, room-temperature NMR spectra were obtained on a Bruker Avance 500 MHz spectrometer.

2.4.1.1 Working Principle:

The operating principle of Nuclear Magnetic Resonance (NMR) relies on the behavior of atomic nuclei with spins. When a nucleus possesses a non-zero spin, it generates a magnetic field due to its charged particle motion. By subjecting the sample to a powerful magnetic field and supplying the appropriate energy, the non-zero spin nuclei undergo a transition from a lower energy state to a higher energy state. The strength of the applied magnetic field governs the energy difference between these two states. The specific type of nucleus and its chemical environment in the molecule influence the amount of energy absorbed during this transition. This excitation, or "flipping," of the nuclei from one orientation to another, leads to the detection of an induced voltage caused by the absorption of energy from a radio frequency field, which further enhances the magnetic field. The resulting signal in the time domain, known as the free induction decay, is then converted into the frequency domain using a Fourier algorithm. By analyzing the field strength at which protons absorb energy and examining the area under a peak, one can determine the molecular structure. Notably, the area under the peak is directly proportional to the number of nuclei undergoing this "flipping" process.

Figure 2.1 Photograph of Nuclear Magnetic Resonance (NMR)

2.4.2 Transmission Electron Microscopy (TEM)

The Transmission Electron Microscope (TEM) is a sophisticated imaging instrument that utilizes a focused beam of electrons to examine specimens at a highly detailed level. By passing electrons through the specimen, it generates a magnified image with extraordinary resolution and allows for the observation of ultrafine structures, such as nanoparticles, viruses, and cellular organelles. The TEM's magnification capability surpasses that of a traditional light microscope by over two million times, facilitating the analysis and characterization of specimens in terms of their morphology, composition, and crystalline arrangements. This powerful microscopy technique has been instrumental in advancing scientific research across various fields, including materials science, biology, and nanotechnology.

This method involves using a heated tungsten filament within the electron gun to generate electrons, which are then concentrated onto the specimen through the condenser lenses (as shown in Figure 2.1). When the electrons encounter the specimen, they are scattered and focused by magnetic lenses, resulting in a magnified and distinct image. Passing through a fluorescent screen creates a polychromatic image. A darker image is produced when the specimen is denser because more electrons are scattered, leading to fewer electrons reaching the screen for visualization. Conversely, thinner and more transparent specimens appear brighter due to fewer scattering events.

The transmission electron microscope (TEM) operates based on similar principles to the light microscope, but instead of using light, it employs electrons. Electrons possess both wave and particle characteristics, and due to their smaller de Broglie wavelength compared to light, they offer higher resolution capabilities. This allows users to investigate intricate details, even down to the scale of a single column of atoms, which is tens of thousands of times smaller than what can be resolved with a light microscope.

In the present study, the TEM analysis of the sample was performed on TECNAI 20 G2-

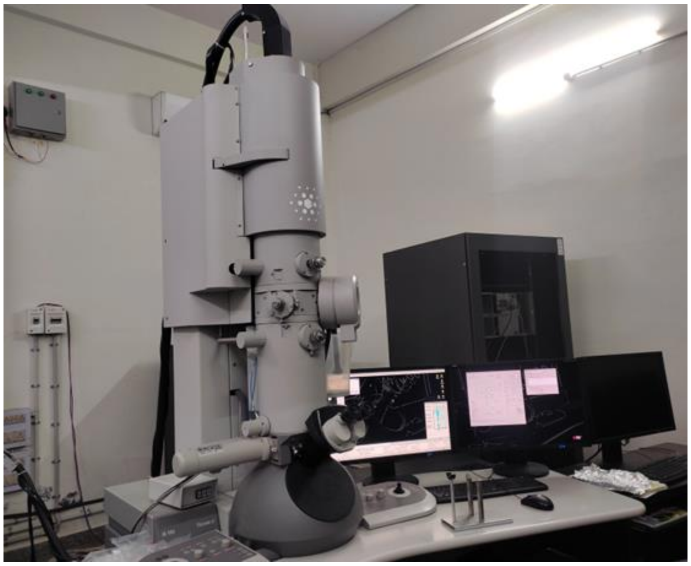

Figure 2.2 Photograph of Transmission electron microscopy (TEM).

Electron Microscope operated at accelerating voltage 200 kV (**Figure 2.2**). The samples were prepared by simply mounted the dilute solution of the sample on carbon coated TEM grid and dried under table lamp for 5 h after that vacuum dried overnight.

2.4.3. X-ray Diffraction (XRD)

XRD is indeed a powerful technique used to study the atomic and molecular structure of crystalline materials. When X-rays are directed onto a crystal, they interact with the crystal's electrons and scatter in different directions. The scattering pattern is determined by the arrangement of atoms within the crystal lattice. The key principle behind XRD is constructive interference. When the X-rays interact with the crystal lattice, they can be thought of as waves that diffract, meaning they change direction due to the interaction with the atoms in the crystal. The X-rays scattered by different planes of atoms in the crystal can either reinforce each other (constructive interference) or cancel each other out (destructive interference), depending on the angle at which they are scattered and the distance between the atoms.

The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law (**equation 2.6**).

$$n\lambda = 2d \sin \theta \qquad \ldots\ldots\ldots\ldots\ldots \quad 2.6$$

Where n is an integer value, λ represents the wavelength of X-rays, d is interplanar spacing generating the diffraction, and θ is the diffraction angle.

The resulting diffraction pattern is collected on a detector, typically a photographic film or a digital detector, which records the intensities and angles of the scattered X-rays. From this diffraction pattern, researchers can extract valuable information about the crystal's lattice parameters (spacing between lattice planes), crystal symmetry, and atomic arrangement. By analyzing the peaks and their positions in the diffraction pattern, researchers can determine the crystal's lattice structure and its orientation relative to the incident X-rays.

X-ray diffraction has applications in various fields, such as materials science, chemistry, physics, geology, and even biology. It is widely used to identify unknown crystalline substances, study crystal defects, analyze phase transitions, and investigate the atomic structure of complex materials. Overall, XRD is an indispensable tool for understanding the fundamental properties of crystalline materials and their behavior.

In typical X-ray diffraction, an X-ray is generated by a cathode ray tube and the filtered monochromatic radiation is collimated and directed toward the sample. The interaction of the incident rays with the sample produces constructive interference i.e. diffracted ray when it satisfies Bragg’s law. These diffracted X−rays are then detected, processed, and counted and all possible diffraction directions of the lattice should be attained due to the random orientation of the powdered material by scanning the sample through a range of 2θ angles. X-ray diffractometers are composed of three primary components: an X-ray tube, a sample holder, and an X-ray detector. Within a cathode ray tube, X-rays are produced by heating a filament to generate electrons. These electrons are accelerated towards a target by applying voltage, and they collide with the target material, dislodging inner shell electrons. This interaction leads to the production of characteristic X-ray spectra.

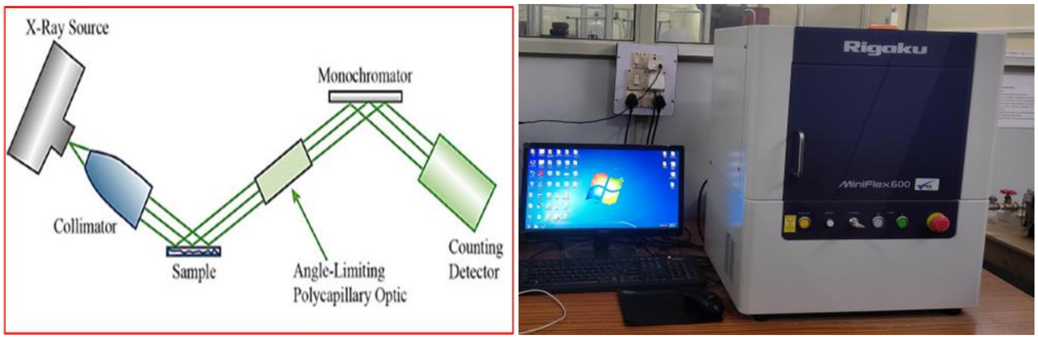

Figure 2.3 Schematic representation of principle of X-Ray diffraction and Photograph of X-Ray diffraction spectroscopy (XRD).

2.4.4. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy (FTIR) is a technique utilized to identify distinct functional groups within molecules or compounds, whether they are organic or inorganic. It serves as a potent tool for recognizing the types of chemical bonds present in a molecule, generating an infrared absorption spectrum that acts like a unique "fingerprint" for the molecule. The term FTIR refers to an advancement in how data is gathered and converted from an interference pattern into a spectrum. This technique relies on the principles of a Michelson interferometer, which incorporates a beam splitter, a fixed mirror, and a movable mirror that moves with precision.

The process initiates with radiation from the source striking the beam splitter, dividing into two beams. One beam proceeds through the beam splitter to the fixed mirror, while the other beam reflects off the beam splitter to reach the moving mirror. The fixed and moving mirrors then direct the radiation back to the beam splitter. Once again, half of this reflected radiation is transmitted, while the other half is reflected at the beam splitter. This results in one beam passing through the sample, subsequently detected by a detector. The resulting spectra are displayed on a computer, while the second beam retraces its path back to the radiation source.

In our investigation, we employed the "PerkinElmer Spectrum 100" instrument to conduct FTIR spectroscopy on the sample. The radiation sources were transmitted through a KBr window, and the resulting data was captured by a LiTaO3 detector, with spectra displayed in transmitted mode. The sample pellets were created by blending the sample with KBr in a 1:100 ratio. These prepared pellets were then scanned within the 400-4000 cm-1 range, utilizing a spectral resolution of 4.0 cm-1 and a scan speed of 0.2 cm/sec.

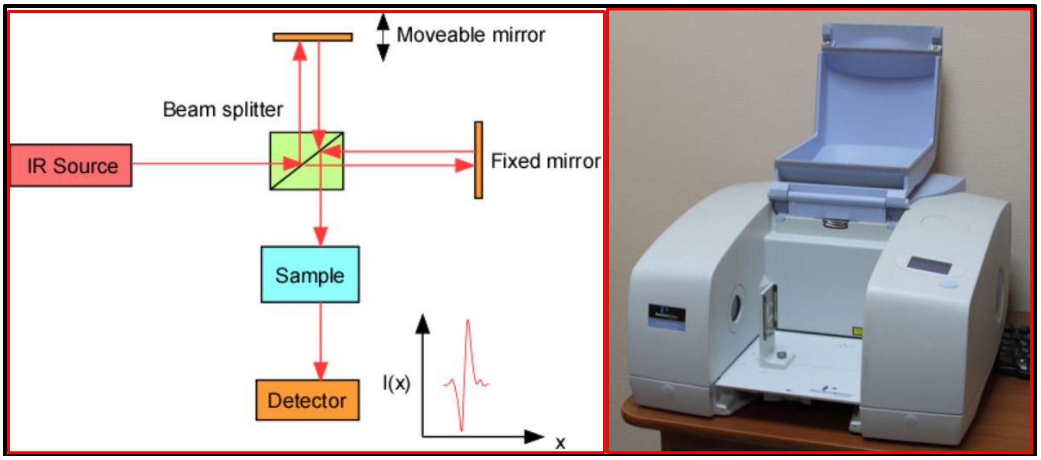

Figure 2.4 Schematic representation of working of Fourier transform infrared (FTIR) spectrophotometer and its photograph.

2.4.5. X-ray Photoelectron Spectroscopy (XPS)

X-ray Photoelectron Spectroscopy (XPS) stands out as the primary choice for surface analysis due to its applicability to a wide array of materials and its ability to furnish crucial quantitative and chemical state data regarding the surface of the material under investigation. XPS is particularly valuable because it not only identifies the elements present in a sample but also reveals the elements to which they are bonded. Typically, XPS probes to an average depth of about 5 nanometers.

The fundamental principle behind XPS analysis involves irradiating the target material with X-rays possessing adequate energy in an ultrahigh vacuum environment, typically at a pressure of around 10^{-9} millibar (mbar). This irradiation excites electrons in specific bound states. Some of these photo-ejected electrons undergo inelastic scattering as they traverse through the sample en route to the surface, while others are promptly emitted without losing energy as they escape from the surface into the surrounding vacuum. Subsequently, these photo-ejected electrons are gathered by an electron analyzer, which measures their kinetic energy. The electron energy analyzer generates an energy spectrum that illustrates intensity (the number of photo-ejected electrons versus time) in relation to binding energy (the energy the electrons possessed before departing the atom). Each distinct energy peak in the spectrum corresponds to a specific element. The intensity of these peaks also provides insights into the quantity of each element present in the sample. The area under each peak is directly proportional to the number of atoms of the respective element, and the determination of chemical composition involves calculating the relative contributions from each peak area.

Figure 2.5 Typical photograph of XPS instrument.

2.4.6. UV-visible Spectroscopy

UV-visible (UV-Vis) spectroscopy involves measuring the absorption of electromagnetic radiation in the UV and visible regions of the spectrum by molecules. This absorption occurs due to electronic transitions, where electrons move from lower energy orbitals to higher energy orbitals when they absorb energy from incident light. In UV-Vis spectroscopy, the absorption of radiation at various wavelengths is recorded and typically plotted as a graph. This graph is known as an absorption spectrum. It shows how the absorbance (or absorptivity) of a compound varies with different wavelengths of light in the UV and visible range. These absorption maxima are often referred to as "regions of absorption." Different compounds or functional groups exhibit characteristic absorption maxima in UV-Vis spectra, allowing for their identification and differentiation. UV-Vis spectroscopy is a widely applicable technique because many organic and inorganic compounds contain chromophores or absorbing species.

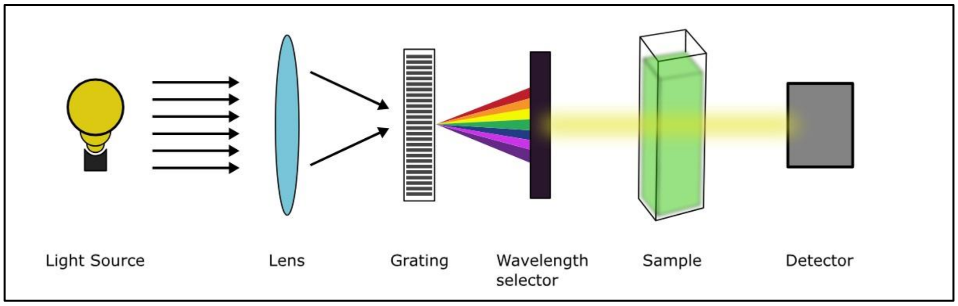

Figure 2.6 Schematic diagram of UV-visible spectrophotometer

Figure 2.7 Typical photograph of UV-visible spectrophotometer

This versatility makes it a valuable tool in various fields, including chemistry, biochemistry, pharmacology, and environmental science. Researchers can use UV-Vis spectroscopy to identify compounds, assess their concentration, and study their electronic structure.

In the present study, UV-visible absorption spectra of the synthesized materials were acquired on “Evaluation 201 Thermo Scientific” using 1 cm path length quartz cuvettes in the wavelength range of 200–700 nm. The photograph of the UV-visible spectrophotometer is depicted in (**Figure 2.7**).

2.4.7. Scanning electron microscope (SEM)

The SEM images were JEOL-6700F microscope instrument and the sample was prepared by dropping solution on a glass cover slip, dried under vacuum. SEM is a type of electron microscope that produces images of a sample by scanning the surface with a focused beam of electrons. The electrons interact with atoms in the sample, producing various signals that contain information about the surface topography and composition of the sample. The electron beam is scanned in a raster scan pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image. In the most common SEM mode, secondary electrons emitted by atoms excited by the electron beam are detected using a secondary electron detector (Everhart-Thornley detector). The number of secondary electrons that can be detected, and thus the signal intensity, depends, among other things, on specimen topography. SEM can achieve resolution better than 1 nanometer. Specimens are observed in high vacuum in conventional SEM, in low vacuum or wet conditions in variable pressure or environmental SEM, and at a wide range of cryogenic or elevated temperatures with specialized instruments

2.4.7.1. Principles of SEM

The scanning electron Microscope uses emitted electrons. The Scanning electron microscope works on the principle of applying kinetic energy to produce signals on the interaction of the electrons. These electrons are secondary electrons, backscattered electrons, and diffracted backscattered electrons which are used to view crystallized elements and photons. Secondary and backscattered electrons are used to produce an image. The secondary electrons emitted from the specimen play the primary role of detecting the morphology and topography of the specimen while the backscattered electrons show contrast in the composition of the elements of the specimen.

Due to the very narrow electron beam, SEM micrographs have a large depth of field, yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. A wide range of magnifications is possible, from about 10 times (about equivalent to that of a powerful hand lens) to more than 500,000 times, about 250 times the magnification limit of the best light microscopes.

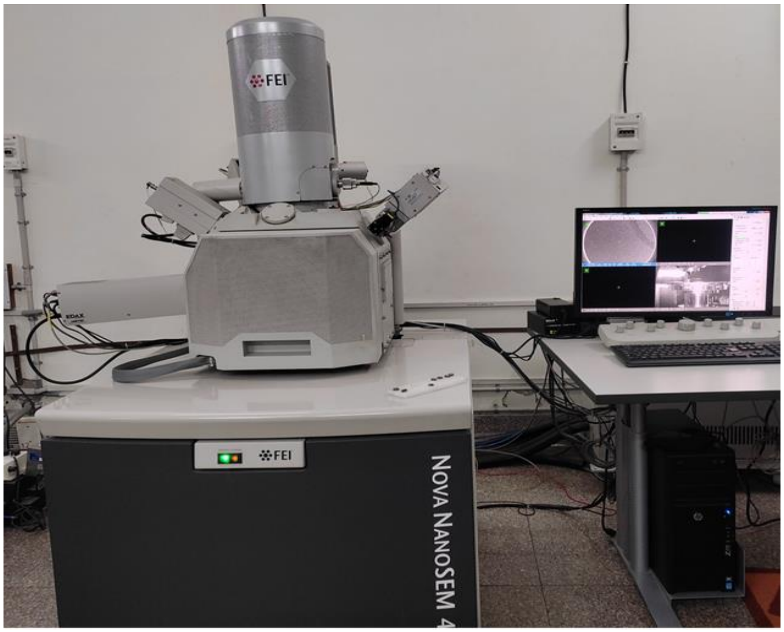

Figure 2.8 Typical photograph of Scanning electron microscope (SEM)

2.4.7.2. Sample preparation

SEM samples have to be small enough to fit on the specimen stage and may need special preparation to increase their electrical conductivity and stabilize them so that they can withstand the high vacuum conditions and the high energy beam of electrons. Samples are generally mounted rigidly on a specimen holder or stub using a conductive adhesive. SEM is used extensively for defect analysis of semiconductor wafers, and manufacturers make instruments that can examine any part of a 300 mm semiconductor wafer. Many instruments have chambers that can tilt an object of that size to 45° and provide continuous 360° rotation.

Nonconductive specimens collect charges when scanned by the electron beam, and especially in secondary electron imaging mode, this causes scanning faults and other image artifacts. For conventional imaging in the SEM, specimens must be electrically conductive, at least at the surface, and electrically grounded to prevent the accumulation of electrostatic charge. Metal objects require little special preparation for SEM except for cleaning and conductively mounting to a specimen stub. Non-conducting materials are usually coated with an ultrathin coating of electrically conducting material, deposited on the sample either by low-vacuum sputter coating or by high-vacuum evaporation. Conductive materials in current use for specimen coating include gold, gold/palladium alloy, platinum, iridium, tungsten, chromium, osmium, and graphite. Coating with heavy metals may increase the signal/noise ratio for samples of low atomic number (Z). The improvement arises because secondary electron emission for high-Z materials is enhanced.

2.4.8. Atomic force microscopy (AFM)

AFM belongs to a family of instruments called scanning probe microscopes that make NT-MDT NTEGRA Prima. It measures surface structure in length scale 10 nm- 100 μm with high resolution and accuracy (lateral resolution~30 nm, vertical resolution~0.1 nm).

2.4.8.1 Basic principle

The principle of AFM functioning is completely different from the scanning electron microscope and provides a better analysis of nanoscale materials with minimal sample preparation. It is based on a physical scanning of samples at the sub-micron level using a probe tip of the atomic scale and offers an ultra-high resolution in particle size measurement. Depending upon properties, samples are usually scanned in contact or noncontact mode.

In contact mode, the cantilever/tip is allowed to be in contact with the surface of the sample during the entire imaging process. The contact mode measures the surface topography of the sample by using a cantilever, which is made of a material having a lower spring constant.

As the cantilever remains at the surface, the tip gets deflected by changing the height of the surface features. The height of the tip can be adjusted using the feedback system of AFM to follow the contour and maintain a constant tip pressure at the surface. The use of low spring constant materials and the feedback serves to preserve the integrity of the tip while imaging the sample. During the imaging, an abrupt change in surface feature height may occur due to the incapability of the system to handle and damage and destroy the tip. The AFM requires the samples should be mounted on a very flat surface because the height limitation of many AFMs is only in the 10-15 µm range. To avoid the damaging to the sample, tapping or intermittent contact mode imaging is preferred. In this mode, the cantilever is driven by a piezoelectric resonator in a frequency range of 150 – 300 kHz. In this mode, the cantilever used is made up of higher spring constant material, which provides higher resonant frequency to the cantilever/tip and the overall system.

One of the prime advantages of AFM is its ability to image non-conducting samples without any specific treatment. This feature allows the imaging of delicate biological and polymeric Nano and microstructures. Moreover, AFM (without any mathematical calculation) provides the most accurate description of size, size distribution, and real picture, which helps in understanding the effect of various biological conditions.

2.4.9. Thermogravimetric Analysis

Thermal analyses were carried out with a TA Instruments SDT Q600 under a nitrogenous atmosphere with a flow rate of 100 mL/min in a platinum crucible at a rate of 10 °C/min. It is a simple analytical technique that measures the amount and rate of change in the weight of a material as a function of temperature or time in a controlled atmosphere. Measurements are used primarily to determine the composition of materials and to predict their thermal stability at temperatures up to 1000 °C. It is the most widely used thermal method, which can characterize materials that exhibit weight loss or gain due to decomposition, oxidation, or dehydration.

2.4.9.1 Basic principle

As materials are heated, they can lose weight from a simple process such as drying, or from chemical reactions that liberate gases. Some materials can gain weight by reacting with the atmosphere in the testing environment. Since weight loss and gain are disruptive processes to the sample material, knowledge of the magnitude and temperature range of those reactions is necessary to design adequate thermal ramps and holds during those critical reaction periods. Such analysis relies on a high degree of precision in three measurements: weight, temperature, and temperature change. A plot of weight change versus temperature is referred to as the thermogravimetric curve (TGA curve), which helps in revealing the extent of purity of analytical samples and determining the mode of their transformations within the specified range of temperature. As many weight loss curves look similar, the weight loss curve may require transformation before results may be interpreted. A derivative weight loss curve can be used to tell the point at which weight loss is most apparent. Therefore, TGA curves can provide information about the composition of multi-component systems, thermal stability, and oxidative stability of materials, decomposition kinetics of materials, and the effect of reactive or corrosive atmospheres on materials and moisture content of materials.

2.4.10. Rheology

Rheology is defined as the study of the flow and deformation of matter which describes the interrelation between force, deformation, and time. The term rheology originates from the Greek words 'rheo' translating as 'flow' and 'logia' meaning 'the study of', although as from the definition above, rheology is as much about the deformation of solid-like materials as it is about the flow of liquid-like materials and in particular deals with the behavior of complex viscoelastic materials that show properties of both solids and liquids in response to force, deformation and time.

2.4.10.1 Definition of terms:

Shear stress

$$\text{Shear stress} = F / A$$

shear force F (in N, Newton) and shear area A (in m), The unit for shear stress is 1 N/m = 1 Pa (Pascal). A rheometer records the shear force via the torque at each measuring point.

Shear strain

$$\text{Shear strain} = s / h$$

deflection path s (in m), and shear gap h (in m), The unit for shear strain (deformation) is (m/m) = 1, which means that deformation is dimensionless. Usually, the value is stated as a percentage

Storage modulus (G')

The storage modulus G' (G prime, in Pa) represents the elastic portion of the viscoelastic behavior, which quasidescribes the solid-state behavior of the sample.

Loss modulus (G'')

The loss modulus G" (G double prime, in Pa) characterizes the viscous portion of the viscoelastic behavior, which can be seen as the liquid-state behavior of the sample G′ and G″ are calculated from the measured phase angle (δ):

$$G' = G^* \cos \delta$$

$$G'' = G^* \sin \delta$$

$G' = G^*$ ($\delta = 0°$) for an ideal solid and $G'' = G^*$ ($\delta = 90°$) for an ideal liquid. The rheometer directly shows the moduli in Pascal. $G' > G''$ is the rheological definition of a gel and shows that the elastic behavior is dominant in these materials. Three different kinds of measurements are carried out to confirm the gel nature.

Dynamic Frequency Sweep

Dynamic Frequency Sweep: The dynamic frequency sweep plot shows G′ and G″ at the constant strain in dependency on increasing frequency

Dynamic Time Sweep

Dynamic time sweep gives a plot of G′ and G″ with constant frequency and constant strain over time, which confirms the gel nature of a material over time.

2.4.11. Schottky diode Device

The device was designed with a sandwich-like configuration, consisting of different layers of Indium tin oxide (ITO), metallogel, and Aluminium. The choice of materials and layer arrangement in this configuration is significant for the device's operation. Here is a breakdown of the layers and their roles:

- Indium Tin Oxide (ITO) layer: The ITO layer is commonly used as a transparent conducting oxide (TCO) in electronic devices. This Schottky barrier diode likely serves as the substrate or bottom electrode. The ITO layer provides electrical conductivity while allowing light to pass through, which can be important for certain applications.
- Metallogel layer: The metallogel thin film is the central component of the Schottky diode device. It acts as the Schottky contact or Schottky barrier. The metallogel, with its unique properties, forms a stable contact with the semiconductor material, creating the rectifying junction required for the diode's operation. The specific composition and structure of the metallogel thin film would have been tailored to achieve the desired electrical and material properties.
- Aluminium layer: The Aluminium layer serves as the top electrode in this configuration. It provides electrical contact to the metallogel layer and allows for current flow in the diode. Aluminium is commonly used as a metal electrode in electronic devices due to its good conductivity and compatibility with many materials.

2.4.11.1. Working principle of a Schottky diode

The working principle of a Schottky diode with a metallogel can be explained as follows:

Formation of the Schottky barrier: When the metallogel is brought into contact with the semiconductor material, a Schottky barrier is formed at the interface between them. The Schottky barrier is a potential barrier that controls the flow of current in the diode.

- **Rectification:** The Schottky barrier causes rectification behavior in the diode. Rectification refers to the ability of the diode to allow current flow in one direction (forward bias) while blocking it in the opposite direction (reverse bias).
- **Forward bias:** When a positive voltage is applied to the metal electrode of the Schottky diode with the metallogel, it reduces the height of the Schottky barrier, allowing electrons to flow from the semiconductor material into the metal. This results in a current flow through the diode in the forward direction.
- **Reverse bias:** When a negative voltage is applied to the metal electrode, it increases the height of the Schottky barrier, effectively blocking the flow of electrons from the metal to the semiconductor. This prevents current flow in the reverse direction.

2.4.11.2. Fabrication of Schottky diode

Fabricating a metal-semiconductor (MS) junction Schottky barrier diode on indium-doped tin oxide (ITO) coated glass substrates involves specific steps to create the Schottky contact on the ITO surface. Below is a general outline of the fabrication process:

- **Cleaning and Preparation:** Begin by cleaning the ITO-coated glass substrate to remove any contaminants and native oxides from the surface. Use a standard cleaning procedure, which may include solvents, ultrasonic cleaning, and rinsing with deionized water.
- **Deposition of semiconducting material:** Deposition of semiconducting material such as metallogel solution on the as cleaned ITO coated glass substrate. The process was optimized and repeated two times (at the same speed and duration) to obtain a thickness of ∼100 nm.

- **Deposition of Metal:** Deposit the metal layer on the ITO-coated glass surface. The choice of metal will depend on the desired characteristics of the Schottky diode. Commonly used metals for Schottky diodes on ITO substrates include aluminum (Al), silver (Ag), or platinum (Pt)
- **Annealing:** Perform annealing to improve the adhesion and electrical properties of the metal-semiconductor interface. Annealing involves heating the substrate with the metal layer to a specific temperature for a defined time in a controlled atmosphere.
- **Photolithography:** Use photolithography to pattern the metal layer and define the size and shape of the Schottky contact. Apply a photoresist on the metal layer, expose it to UV light through a mask, and develop it to create the desired pattern**.**
- **Metal Etching:** Use an appropriate etchant to selectively remove excess metal and retain the patterned Schottky contact on the ITO surface.
- **Characterization and Testing:** After completing the fabrication steps, the Schottky diodes on ITO-coated glass substrates should be characterized and tested for their electrical performance, including current-voltage characteristics, barrier height, and ideality factor**.**

It's important to mention that the fabrication process may involve variations and additional steps based on specific device requirements and intended applications. Also, the choice of materials and fabrication techniques may evolve with advancements in technology and materials research.