Revenue generated from consultancy and corporate training during the year (INR in lakhs) (2022-23)

Name of the teacher-consultants	**Name of consultancy project/corporate training program**	**Consulting/Sponsoring agency with contact details**	**Revenue generated (INR in Lakhs)**
Dr Princy K.G	Water Quality Analysis	SCLPS, Kottackal	200
Dr. Maya Mathew	Masters Project work	SNM College, Maliyankara	4000
Gilu Mathew	Orientation for parents	SOCCORSO LP school Mala	2000
Gilu Mathew	Motivational session for Doctors	Smera Academy Kannur	5000
Gilu Mathew	Accounts Executive - Expert interaction with HSS students	SNVVHSS Aloor	1500
Gilu Mathew	Expert interaction with motivational speaker HSS students	SNVVHSS Aloor	1500
Gilu Mathew	Orientation for parents	Kottackal Church	3000
Gilu Mathew	Orientation for Students	Fr. Davis Chiramel Charitable Trust -MAMHSS, Koratty	1500
Gilu Mathew	Orientation for parents	Fr. Davis Chiramel Charitable Trust-UHSS, Annanad	1500
Gilu Mathew	Orientation for parents	SOCCORSO KG school Mala	3000
Gilu Mathew	Expert interaction with motivational speaker HSS students	GVHSS, Puthenchira	1500
Gilu Mathew	Orientation for 10^{th} graders	HCCHS(ICSE), Snehagiri	3000
Gilu Mathew	Orientation for Students	Fr. Davis Chiramel Charitable Trust- L F HSS Koratty	1500
Gilu Mathew	Orientation for parents	Fr. Davis Chiramel Charitable Trust - L F HSS Koratty	1500
Gilu Mathew	Orientation for Students	Lisuex HS, Irinjalakuda	3000
Gilu Mathew	Orientation for Students	SSMHSS, Azhicode	3000
Gilu Mathew	Orientation for Students	S N College N Paravur	3000
Keerthi Sophia Ponnachan	Orientation for Students	College of Forestry, Kerala Agricultural University, Vellanikkara	1500
Dr. Sr. Rini Rahael	Subject expert for government guest lecture interview	S.H College, Chalakudy	2000
Dr Princy K.G	Subject expert for promotion of faculty	L.F College, Guruvayoor	2000

CARMEL COLLEGE (AUTONOMOUS) Date:........................ MALA - 680732

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കോട്ടക്കൽ മാള

Date...7/7/2022...........

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സാർ,

ഈ വിദ്യാലയത്തിലെ വിദ്യാർത്ഥികൾക്ക് ഉച്ചഭക്ഷണം പാചകം ചെയ്യുന്നതിലേക്ക് എടുക്കുന്ന വെള്ളത്തിന്റെ ഗുണമേന്മ അറിയുന്നതിന് അത് Test ചെയ്യേണ്ടതായിട്ടുണ്ട്. അതിനാൽ വെള്ളത്തിന്റെ സാമ്പിൾ പരിശോധനയ്ക്കായി കൊടുത്തുവിടുന്നു. ആയത് പരിശോധിച്ച് അതിന്റെ Report തരണമെന്ന് താല്പര്യപ്പെടുന്നു.

വിശ്വസ്തതാപൂർവ്വം

SOCCORSO CONVENT LP SCHOOL KOTTAKKAL ★ MALA

Sr. [signature]

Headmistress
SCLPS KOTTAKKAL
MALA

SYNTHESIS AND CHARACTERIZATION OF THIN FILMS OF CTS USING SILAR TECHNIQUE

PROJECT REPORT

In partial fulfillment of the requirement for the award of
Master of Science in Physics

Submitted by

ARYA PU

M.Sc Physics

Register Number- 210011012999

MAHATMA GANDHI UNIVERSITY, KOTTAYAM

S.N.M COLLEGE, MALIYANKARA

Kerala- 683516

DECLARATION

I hereby declare that the project entitle "**Synthesis and characterization of thin films of CTS using SILAR technique**" was written and submitted by me under the guidance of **Dr. Maya Mathew** department of Physics, Carmel College (Autonomous),Mala. I also declare that this project has not been submitted at any time to any other university or institute for the award of any degree or diploma.

S.N.M College — Arya P U

Maliyankara — MSc Physics

ACKNOWLEDGMENT

Firstly I thank Lord almighty for guiding me in every step on my way to the completion of this project. Iwould like to thank our principle, **Dr. Jitha T H**, S.N.M college, Maliyankara**, Dr. Neena Prasad**, HOD, department of Physics, S.N.M college for their cordial support, informative interactions and motivations throughout the course of this project. Thanks to my guide **Dr. Maya Mathew**, Assistant Professor, Department of Physics Carmel College (Autonomous), Mala. For sparing her precious time in putting together all the bits and pieces I brought in as raw data to her. Her contribution towards the success of this project is unmatched. I also express my sincere gratitude to the principle of Carmel College (Autonomous). I would like to thank the department of Physics, Carmel College (Autonomous), Mala for the facilities provided for the synthesis of samples, STIC Cochin for the XRD,SEM and Cross-SEM analysis and C-MET Thrissur for the Hall measurement.

This project would not have been successfully materialized, had it not been for the several people who have helped us. I am extremely indebted to all of them and whole-heartedly thank everyone for their valuable support. I sincerely thank all my friends and classmates who in one way or the other helped in the completion of this work. I truly admire my parents for their constant encouragement and enduring support which was inevitable for the success of all the ventures

ABSTRACT

Thin film of copper tin sulfide (CTS) where prepared using successive ionic layer adsorption and reaction (SILAR) technique. Millimolar solutions of $CuCl_2.2H_2O$, $SnCl_2.2H_2O$ and Na_2S where used as precursors in the molar ratio of 2:1:3 with Cetrimonium Bromide as the surfactant. The amount of surfactant was varied in two samples and in the third sample no surfactant was used. The three samples were found to be of tetragonal phase and the crystallite size was determined using Sherrer equation. The crystallite size and film thickness of the samples were found to increase when the amount of surfactant decreased. The surface morphology of the samples was observed to be of particle nature when surfactant was used while uniform film was formed for the sample without surfactant. All the samples were found to be of p-type and have high mobility from Hall measurements which makes these samples potential materials for the absorber layer of solar cells.

TABLE OF CONTENT

5. CONCLUSION

6. REFERENCES

CHAPTER I

1. INTRODUCTION

1.1.BULK MATERIALS

Bulk materials are dry materials which are powdery, granular or lumpy in nature they are stored in large amount. They can be seen by our naked eye or by a simple microscope. Minerals, coal, sand, clay, cement, ash, salt, sugar, flour are examples of bulk materials. They are useful in traditional manufacturing, construction and everyday items. Compared to bulk materials, nano particles are smaller than bulk materials and they can be hardly seen by our naked eye or a simple microscope. Special microscope is used for it. carbon nanotubes, nanoparticles, graphene are some of the examples. nano materials are useful in nanoelectronics, nanomedicine and sensor.

Many materials which are available in bulk or large form which have useful in many fields like science and technologies are expected to convert to thin forms so that there is less wastage of bulk materials but this thin form will still retain the same properties and functions of the bulk form. For example, if we consider the first- generation computer which is very large and require many to move or operate it, but modern computer is small with their great function and speed .it also happened due to the help of thin film science and technologies.

There are many differences in properties between bulk and thin film, which is due to their structural and dimensional difference.

- **Thickness**: we know the difference in the thickness, bulk have a much greater thickness than thin film and volume too.
- **Structural organization**: Thin-film have different structural organization compared to bulk materials. Bulk material is a three-dimensional structure with long range order, while thin film is a two-dimensional structure with short range order.
- **Surface to volume ratio**: thin film have a high surface to volume ratio compared to the bulk material. In thin films thickness varies from nanometers to a few micrometers, while bulk is greater than that. Because of the Small thickness thin film have unique properties and functions.
- **Strain and defects**: thin film can experience strain due to lattice mismatch with substrate or growth induced stress. Defects is formed due to this strain. This strain and defects influence the properties of thin film.
- **Interface effects**: thin film is deposited on a substrate, there are effects on the structure, properties of thin film due to the relation between thin film and substrate
- **Optical properties**: thin film have unique properties compared to bulk materials. They both have different absorption, reflection and scattering properties which help in anti-reflection coating, optical filter.
- **Electrical properties**: electrical properties of thin film depend on materials of thin film, whether they are metallic films, semiconductor films, insulators films and the type of substrate they are deposited. Size effect is one of the main factors which influence the electrical properties. Charge carries inside a thin film have shorter mean pat compared to bulk material, which have more scattering points like

structural defects and grain boundaries result in reduction of electrical conduction.

- **Mechanical properties**: they both have different mechanical properties compared with each other. the existence of microstructures like grain boundaries, dopants and dislocations in the thin film results in larger hardness compared to their bulk counterpart.

1.2. THIN FILM

Thin film is a thin layer of material ranging from fraction of nanometer to micrometer in thickness. The controlled synthesis of materials as thin films is a fundamental step in many applications. Which can be referred as deposition, it is a technique of depositing a thin film of materials on a substrate or previous thin film. deposition technique falls in to two categories – chemical and physical. Thin films maintain a surface uniformity and they help to reduce the weight and bulkiness of the materials. Thin film technology involves deposition of individual atoms or molecules. One of the interesting part of synthesis of thin film is that we can combine two or more materials together on a single plate to achieve new type of films which have unique properties of the materials. During 20th century there is advances in thin film technology which have enabled a wide range of breakthrough in areas such as electronic semiconductor devices, LED, optical coating-antireflective coating, thin film solar cells, thin film batteries. It is also being applied to pharmaceuticals -thin film drug delivery.

The process of applying thin film on a surface is known as thin film deposition. This technique is divided into two – chemical or physical, depending whether the process is primarily using chemical or physical reactions. In Chemical deposition, a fluid undergoes a chemical change at a solid surface, leaving a solid layer.

- **Chemical bath deposition CBD:** is one of the thin film deposition techniques, here substrate are immerse into a liquid solution containing precursor chemicals. Under certain conditions, thin film is formed due to the chemical reaction of substrate. It is a cost-effective method. Their deposition covers large area uniformly.
- **Chemical vapor deposition (CVD)**: they generally use gaseous precursor chemicals onto a substrate. They are mostly used for high quality thin film including semiconductor manufacturing, optical coating and material research.
- **Electrodeposition:** also known as electroplating, is a process where metal or alloy is deposited onto substrate through an electric current. They are commonly used for decorative purpose, such as plating jewelry, silverware and automobile part.
- **Spin coating**: it is a simple and widely used technique for thin film deposition. In this process liquid precursor is applied on a substrate and spinning it at high speed for achieve the uniform thickness. Excess thickness is removed by spinning off the substrate.
- **Dip coating**: in this process substrate is fully submerged in the solution, and then withdraw under controlled condition. After withdrawal, solution adhere to the substrate form thin film.
- **Spray pyrolysis**: it is the technique where a precursor solution is atomizes into fine droplets and it is sprayed on a heated substrate.

Here the precursor goes through the chemical reaction which created the thin film as the solvent evaporates.

Choice of technique based on what we need material, desired film products, substrate techniques and the specific application.

In physical thin film deposition, it involves physical transfer of materials without chemical reactions.

- **Physical vapor deposition**: PVD is one of the widely used technique.it involves evaporation of the material which is solid in a vacuum chamber. After the material heated to its vaporization, the vapor is condensed on the substrate.
- **Thermal evaporation**: thermal evaporation is a technique that melt the material and the vapor created by heating in a vacuum have reached the substrate without reacting with or scattering against another atom in the chamber.
- **Electron beam evaporation**: in electron beam evaporation, high energy beam from an electron gun is used to melt a spot on the material. Here heating is not uniform.
- **Sputter deposition**: this process involves bombarding the material with high energy ions in vacuum. The atoms dislodging from material deposit onto the substrate to form a thin film.
- **Molecular beam epitaxy, (MBE)**: this process is chemical as well as physical, also known as atomic layer deposition. It involves evaporated elements that condensed on substrate to form thin film. we can add one layer of one element and one layer of another element. This technique is called molecular layer deposition, if the precursor is organic.

- **Ion beam deposition**: in IBD ions are accelerated towards a target material. Bombarding this ion in material dislodge atoms, which then deposit onto the substrate under vacuum condition.
- **Laser ablation**: here pulses of laser evaporate the target material and convert it to plasma. The vaporized material then condensed onto substrate thin film.

APPLICATIONS

- **Decorative coating**:
  It is one of the oldest applications of thin film coating. Thin golden leaves were used in India for a long time.
- **Optical coating:**
  optical coating involves depositing metallic or ceramic material as one or two layers on the surface of a glass, which is an optical materials.it helps in reducing the refection of the optical surface.
- **Protective coating**:
  Thin films are applied on surface of the materials and products to provide protection against corrosion, wear, and environmental degradation.
- **Thin film photovoltaic cells:**
  Use of thin film technology in photovoltaic sector help in reducing the cost of production of generation of electricity.

1.3. COPPER TIN SULFIDE THIN FILMS

In material science and semiconductor manufacturing, producing thin films from bulk materials is a common process. For depositing thin films from bulk materials, there are two techniques are widely used: chemical vapor deposition (CVD) and physical vapor deposition (PVD). Usually, thin film deposition techniques are used to produce copper tin sulfide (Cu_2SnS_3 or CTS) thin films. Generally, we used the chemical solution deposition method to make CTS thin films. Both CVD and PVD have advantages and disadvantages; the choice between them depends on factors such as film thickness, specific material, and desired properties of thin films.

1.4. DEPOSITION METHODS

Chemical solution deposition method

In this method, precursor compounds are dissolved in a solvent and then deposited on a substrate, followed by a thermal annealing process to form a thin film.

Materials and precursors

1. **Copper source:** here we use copper acetate, copper chloride,copper nitrate as precursors.
2. **Tin source**: Tin chloride or tin acetate can be used as a tin precursor.
3. **Sulfur source**: thiourea or thioacetamide are commonly used.
4. **Solvent:** We need a suitable solvent, a mixture of water and anorganic compound like ethanol is usually used to dissolve theprecursor.

5. **Dopant precursors**: precursors of elements we wanted to dope.

- Pulsed laser deposition (PLD)

PLD can be used to deposit CTS thin films, and it is a physical vapor deposition technique. When a high-energy laser removes a target material, and to form thin films, the removed material condenses on a substrate.

- **Sputtering**

  It is another vapor deposition technique used to deposit thin CTS films. Here, bombarding a target with ions to eject materials and then depositing them on a substrate.

- **Chemical vapor deposition (CVD)**

It forms a solid thin film on a substrate, and it involves the chemical reaction of gaseous precursors. The following steps are used to produce CTS thin films by using chemical vapor deposition:

1. **Precursor selection**: choose suitable chemical precursors thatcontain elements required for the CTS compound. for example, complex oxides that contain metal halide precursors or metal organic.

2. **Substrate preparation**: by using suitable materials, prepare a clean and flat substrate (e.g., silicon, sapphire, or a specific oxide substrate) and make sure that it is free from impurities.

3. **Reactor setup**: to maintain a precise temperature, pressure, and gas flow control, set up a CVD reactor.

4. **Precursor delivery**: here we will introduce selected precursor gases into the reactor chamber in controlled proportions.

5. **Substrate deposition**: heating the substrate to the desired temperature and maintaining the precursor gases at a suitable pressure. Chemical reactions will occur on the substrate, resulting in the deposition of CTS thin films.

6. **Control parameters**: deposition parameters are controlled, including temperature, pressure, gas flow rates, and deposition time, to achieve the desired film thickness and quality.

7. **Cooling and venting**: after deposition, to avoid thermal stress, cool the substrate gradually and then vent the reactor to release any remaining gases.

8. **Characterization**: to confirm the composition and properties of the deposited thin film using techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), and electrical measurements.

Physical vapor deposition (PVD)

It involves the physical transfer of material from bulk sources to substrates.

1. **Target material:** obtain a target that is made of CTS material. This can be sintered pellets of the compound; here, sinter and pellets are agglomerated forms of iron ore.

2. **Substrate preparation:** a clean substrate is prepared as mentioned in the CVD process.

3. **Deposition chamber:** toset up a PVD chamber, it typically includes a target holder, a vacuum system, and a substate holder.

4. **Vacuum pump:** in the chamber, we created a high vacuum environment to minimize contamination and also allow for controlled deposition.

5. **Target evaporation:** by usingelectron beam evaporation, sputtering, or pulsed laser deposition (PLD), heat the target material to its vaporization temperature.

6. **Substrate deposition:** the substrate is placed in the deposition chamber.and allow the evaporated material to deposit on its surface.

7. **Film thickness control:** To achieve the desired film thickness, control the deposition rate and time.

APPLICATIONS

1. **Solar cells**: CTS thin films have been offered in thin-film solar cell 111technology. In solar cells, they can be used as an absorber layer due to their suitable bandgap for solar absorption.

2. **Thermoelectric devices**: making thermoelectric devices is potentially useful because it converts waste heat into electricity. CTS thin films also show thermoelectric properties.

3. **Optoelectronic devices**: CTS thin films can be combined into optoelectronic devices. LEDs and photodetectors are optoelectronic devices due to their semiconductor characteristics.

CHARACTERISATION

- **X-ray diffraction (XRD):** it is used to determine the phase of CTS thin films and the crystal structure.

- **Scanning electron microscope (SEM):** It is used to study the surface morphology of thin films.

- **Electrical and optical measurements**: these measurements are used for assessing the optical absorption, electrical conductivity, and other properties of CTS thin films.

- **Hall measurement**: it is used to determine accurate carrier density, electrical resistivity, and the mobility of carriers in semiconductors.

Both CTS nanoparticles and thin films offer various applications and opportunities in the fields of renewable energy and optoelectronics. There are two methods used to create copper tin sulfide (Cu_2SnS_3 or CTS): the chemical method and doping-down (or codoping) method, which typically involves a multi-step process. Here we start with precursor materials and adjust their composition by incorporating dopant elements.

1.5 Chemical method for CTS synthesis:

1. Materials and precursors

- Copper precursor
- Tin precursor
- Sulfur precursor
- Dopant precursor

2. **CTS synthesis**

- To create a precursor solution with the desired stoichiometry for CTS, we dissolve the copper, tin, and sulfur precursors in a suitable solvent. Here, the molar ratios should match the composition we want to achieve.

3. **Doping-down**

- To achieve the desired level of doping, the concentration of the dopant should be controlled. Here we introduce the dopant precursor(s) in the desired amount into the precursor solution.

4. **Deposition**

- By using a suitable deposition technique like spin-coating, or blade coating, we apply the doped precursor solution to a clean substrate.

5. **Annealing**

- heat-coated substrate in a controlled atmosphere to anneal the precursor layer. This step converts the precursors into the doped CTS thin film through chemical reactions.

6. **Cooling and characterization**

- After annealing, cool the sample slowly to room temperature and characterize the doped thin film by using different characterization techniques.

Doping-down method

Here, we introduce dopant elements during the synthesis process to incorporate them into the CTS lattice structure. Added the dopant precursor in controlled amounts to achieve the desired doping level. Doping concentration will influence the properties of the resulting CTS material, including electronic and optical properties.

To produce copper tin sulfide (Cu_2SnS_3 or CTS) thin films, we need chemicals that are given out by precursors of copper (Cu), tin (Sn), and sulfur (S) in a suitable solvent. Depending on the synthesis method and desired stoichiometry of the CTS compound, the specific chemicals used can vary.

- **Copper precursor**
- Copper acetate ($Cu(CH_3COO)_2$)
- Copper chloride ($CuCl_2$)
- Copper nitrate ($Cu(NO_3)_2$)

- **Tin precursors**
- Tin chloride ($SnCl_2$)
- Tin acetate (Sn (CH3COO)2)

- **Sulfur precursors**
- Thiourea ($SC(NH_2)_2$)
- Thioacetamide ($CH_3C(S)NH_2$)

These chemicals are chosen because they contain essential elements required for the formation of CTS thin films. Copper-tin sulfide thin films exist in various phases.

- **Stannite phase:** it is the most commonly observed phase of CTS thin film. Here the copper, tin, and sulfur atoms are arranged in a crystalline structure. It has a tetragonal crystal structure.
- **Kesterite:** Kesterite is a quaternary compound that includes zinc (Zn) in addition to copper, tin, and sulfur. Sometimes CT thin films contain a mixture of phases.
- **inhomogeneous phases:** CTS thin films may also exhibit inhomogeneous or mixed phases. It affects their properties. It includes variations in composition or crystal structure.

- **Metastable phases:**

It may form temporarily. These phases are not thermodynamically stable. When compared with stable phases, it may have different Properties.

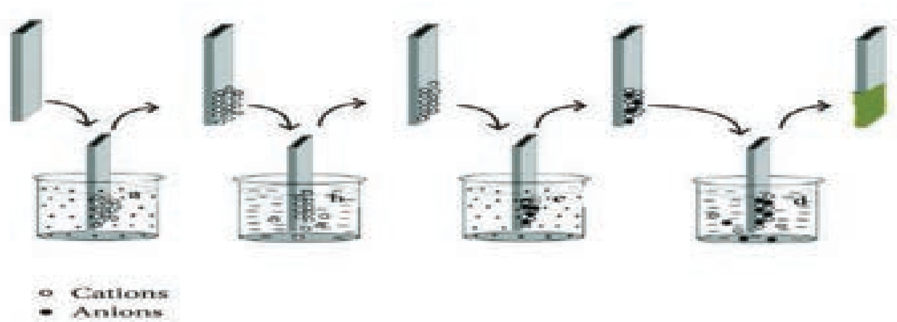
CHAPTER II

2. SYNTHESIS

2.1 SILAR TECHNIQUE

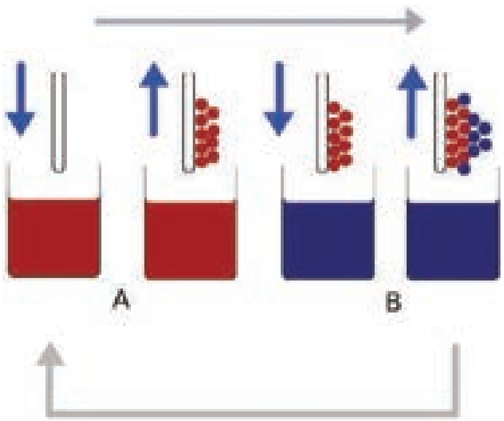
Successive Ionic Layer Adsorption and Reaction is one of the chemical methods for creating large area thin films. As the name suggest this method is based on the adsorption of a layer of ionic species onto the surface, followed by a reaction triggered by a successive Adsorption of a different ionic species. This reaction creates a thin film. If we repeat the process more times, we can increase the thickness of this film. In this process we have cations and anion solutions. Due to the simplicity and cost effectivity of the process, it had gained a lot of popularity. Advanced applications of SILAR now a days are focused on transparent electrodes, solar cells and photoelectrochemical devices. In SILAR in comparison with other coating techniques, thin film is usually deposited on a plane surface and about the reactant solution, which is a solution of desired salt dissolved in solvents. we can change the concentration of the salt in solvent according to the desired thickness and the number of cycles. They are generally conducted at room temperature.

A basic SILAR have four steps, immersion of the substrate into cationic and anionic solution followed by rinsing in between every immersion cycle for the elimination of excessive materials. The steps are adsorption, rinsing, reaction, rinsing.



First is adsorption, where cation precursor is adsorbed on the surface. By the second step rinsing, excessive particles are eliminated. In next step anions are introduced to the system and they react with the surface species. At last the reaction by product and excess particles are eliminated by rinsing.

Water is one of the common rinsing solution, but it is not mandatory. So, without this rinsing step uncontrolled deposition of material is expected.



Metal chalcogens are one of the common materials deposited using SILAR. Their deposition has increased interest in SILAR method.

Here we created 3 CTS thin films here using SILAR technique in which Copper Chloride, Tin Chlorideand Sodium Sulphide as precursors, which are taken in millimolar ratio 2:1:3. Here we use a surfactant-CTAB, which are chemical components that help to reduce surface tension or interfacial tension between different components.we have created a thin film in three different ways, at the first two we change the concentration of surfactant and in the third we didn't use it. So, we get three thin films, which are CTS1, CTS2 and CTS3 correspondingly. Thin film is coated on a glass substrate with 25 cycles at 40°C of temperature.

CHAPTER III

3. CHARACTERIZATION

3.1. COPPER TIN SULFIDE THIN FILM CHARACTERIZATION

Characterizing a copper-tin sulfide (Cu_2SnS_3) thin film is an important step. to evaluate its properties and suitability for various applications, such as photovoltaics, optoelectronics, and semiconductors. To understand its composition, structure, optical, electrical, and physical properties thin film Characterization involves a range of techniques.

Common methods used for characterizing Cu_2SnS_3 thin films:

1. X-ray diffraction (XRD) is used to determine the crystal structure. of the thin film. It exhibits a kesterite crystal structure. It also gives information about the crystallinity, lattice parameters, and crystal orientation.

2. Scanning electron microscopy (SEM) is used to examine the topography and surface morphology of the thin film It also gives the information about grain size, distribution, and film thickness.

3. Transmission electron microscopy (TEM): it provides higher resolution than SEM, and it gives information about the internal structure of the thin film, grain boundaries, including defects and film thickness.

4. X-ray photoelectron spectroscopy (XPS): it can give information about the chemical bonding and oxidation states of copper, tin, Sulfur. XPS is used to examine the surface composition and chemical state of the element present in the thin film

5. Raman spectroscopy is used to examine the vibrational modes and crystal quality of the thin film. It can help to investigate different phases and estimate the film's structural integrity.

6. UV-Vis-NIR spectroscopy: this method is used to measure the transmission properties and vibrational modes of thin films in the ultraviolet (UV), visible (Vis), and near-infrared (NIR) regions. It can give information about the optical constants, film bandgap, and absorption coefficients.

7. Hall measurement: We use this technique to measure electrical conductivity, carrier concentration, and mobility of thin films. It can give information about the film's electrical properties and potential for electronic applications.

8. Fourier transform infrared spectroscopy (FTIR): it can be used to evaluate the vibrational modes of chemical bonds in thin films,

helping to determine its chemical composition and bonding characteristics.

These characterization methods give a complete understanding of the properties of Cu_2SnS_3 thin films. the choice of these characterizations Techniques depend on the specific properties of interest and available equipment. Here are a few characterization methods for getting information:

About CTS thin films, they are given below.

- X-ray diffraction (XRD)
- Scanning electron microscopy (SEM)
- Cross-SEM analysis
- Hall measurement

3.2.X-RAY DIFFRACTION (XRD)

XRD stands for x-ray diffraction. It is a strong analytical method used to study the structure of crystalline material. XRD helps to determine the arrangement of ions or atoms within a crystal lattice. It can give information about crystal size, shape, orientation, and atomic spacing.

Principles of x-ray diffraction

XRD is based on a principle that says that when a beam of x-rays of specific wavelength moving to a crystalline material They interact with atoms. planes within the crystal. This interaction results in constructive interference and gives the diffraction pattern of x-ray beams that are scattered at different angles. These diffracted patterns give information. about the crystal's atomic arrangement.

Components of an XRD experiment

- X-ray source: this source creates the x-rays used in the experiment. X-ray tubes or synchrotron radiation are some common sources.

- Monochromator: the x-ray beam is frequently passed through a monochromator to select a specific wavelength of x-ray.

- Sample holder: the crystalline material under examination is mounted in a sample holder and positioned in the x-ray beam.

- Detector: A detector is used to detect and record the intensity and angle of the diffracted x-rays. Scintillation detectors, or position-sensitive detectors, are some common detectors.

It is widely used to determine the crystal structure of thin films and is also used for different scientific and industrial fields of characterization.

Application of XRD

- Crystal structure determination is used to determine the three-dimensional arrangement of atoms in a crystal lattice. This information is important for understanding material properties.

- Phase identification is used to recognize the crystalline phases present in a sample. It also helps to confirm the composition and purity of materials.

- Stress and strain analysis is especially used in engineering and manufacturing applications. It can be used to measure stress and strain in materials.

- Thin film characterization is used to examine the crystal structure of thin films and multilayer coatings.

- Quality control is used to ensure the consistency of materials.

3.3.SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

It is a powerful technique used for imaging and analysing the surface morphology and composition of materials. SEM stands for scanning electron microscopy.

Principles of SEM analysis

SEM operates on the principle of electron-matter interactions. An electron beam is scanned over the surface of the sample, and different signals are generated as the result of these interactions. To create images and gather information about the surface of the sample, these signals are used.

Components of SEM

1. Electron gun: the electron creates a high-energy electron beam, which is focused into a fine, narrow beam.

2. Electron lenses: to focus and control the position and size of electron beams, we use electromagnetic lenses.

3. Scanning system: a set of scanning coils or plates that direct the electron beam across the surface of the sample in a controlled manner.

4. Sample chamber: the sample to be analysed is placed inside the sample chamber. It is used to prevent the scattering of electrons.

5. Detectors: detectors collect different types of signals created during electron-sample interactions. Secondary electrons (SE), backscattered electrons (BSE), and x-ray detectors for energy-dispersive x-ray spectroscopy (EDS)

Applications of SEM Analysis

- **Surface morphology**
- **Elemental analysis**
- **Particle size and shape analysis**
- **Material characterization**
- **Failure analysis**
- **Nanotechnology**
- **Quality control**

3.4.HALL MEASUREMENT

Hall measurement is a technique used in physics to determine the electrical properties of materials, mainly their charge carrier concentration, mobility and conductivity. This phenomenon was observed by Edwin Hall in 1979. It depends on hall effect, which occurs when a magnetic field is applied perpendicular to the direction of current flow in a conductor. This set up a voltage across the conductor, whose polarity depends on effective charge of the carrier. Hall measurement of thin film help to find out the electrical parameters- carrier concentration, mobility, type of charge carriers. Where carrier concentration means the number of electrons or holes per unit volume within the film. Mobility is the measurement of how quickly carriers move under the influence of an electric field and thus understand the thin film conductivity. The sign of the hall voltage indicates the charge carriers are electrons or holes and identify the materials conductivity type explains the type of charge carriers.

Here We use The Ecopia HMS 3000 VER 3.15.5 equipment for hall measurement. They help in measuring the resistivity, carrier concentration, p/n type, and mobility of various materials at temperature 300K. They also have features in software including I-V curve for checking ohmic integrity.

This system can be used to characterize various materials including semiconductors and compound semiconductors. For doing this process we should know about correct thickness of the sample. So, by using this we identify that our thin films are p-type or n-type. In the case of semiconductors, we can identify between p-type and n-type using hall effect.

3.5.CROSS-SECTIONAL ANALYSIS

Cross- section SEM analysis is a technique, where cross-section of the sample is prepared and scanned by scanning electron microscope (SEM). SEM is a microscope which uses electron for generating images of samples by scanning the surface by focused electron beams. In the process the electrons in the electron beam interact with the atoms in the sample, this generates signals which gives information about structural topography and composition of the sample. In a cross-sectional SEM they reveal the internal micro structure of the sample and give accurate measurements of thickness of the thin film, while SEM analysis only show the surface part.

CHAPTER IV

4. RESULT AND DISCUSSION

4.1. X-RAY DIFFRACTION (XRD)

The XRD patterns of SILAR deposited CTS thin films are shown in figure 4.1. All the samples show preferred orientation of (112) plane. The XRD patterns match well with the tetragonal phase of CTS corresponding to the standard diffraction pattern of JCPDS 089-4714. There is a slight shift in the position of the peaks due to quantum confinement. Peak shifting in XRD pattern of nanostructures can be due to several reasons. The change in synthesis conditions can cause the lattice constants to change. The presence of surfactant can increase the strain in the thin film samples causing slight shifts in the position of peaks . The data on the full width at half maximum for the preferred orientation of all the samples along with the peak positions and crystallite size have been tabulated in table 4.1. From the table it can be observed that the full width at half maximum decreases as the amount of surfactant decreases during the synthesis.
Consequently, the crystallite size of the samples increases as the amount of surfactant decreases. The crystallite size of the samples was determined using Scherrer's formula given by equation1.

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

where, D is the crystallite size, K is a constant which is taken as 0.9, λ is the wavelength of Cu K_α radiation which is taken as 1.54 Å, β is the full width at half maximum (FWHM) in radians and θ is the diffraction angle.

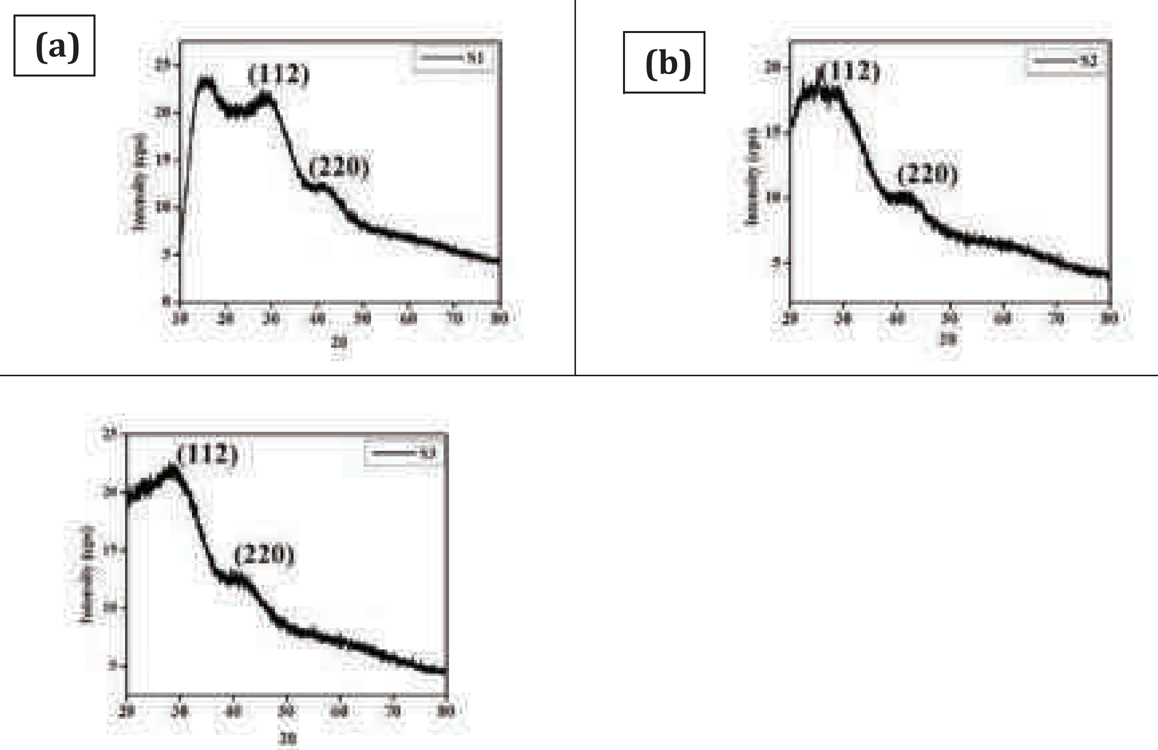


Figure 4.1. XRD patterns of (a) S1, (b) S2 and (c) S3

Table 3.1. Parameters of crystallites of S1,S2 and S3

Sample	**Preferred Orientation**	**2θ (degrees)**	**FWHM (radians)**	**Crystallite size**
S1	(112)	28.82	0.436	32.8 nm
S2	(112)	26.908	0.364	39.2 nm
S3	(112)	28.445	0.361	39.6 nm

4.2. SCANNING ELECTRON MICROSCOPY(SEM)

The Scanning Electron Microscopy (SEM) images of the three samples are shown in figure 4.2. The particle nature of the samples can be seen for S1 and S2 where surfactant has been used whereas in the case of S3, a uniform film nature has been observed. The surfactant caps the particles in the case of S1 and S2 while such a phenomenon is not found in the absence of surfactant. The film thickness was determined using cross- sectional SEM technique and the values have been tabulated in table 4.2. The cross-sectional SEM images are shown in figure 4.3.

From the values it has been observed that the film thickness is less for the sample with higher amount of surfactant while the film thickness increases as the surfactant decreases. The surfactant that is used here is cetrimonium bromide (CTAB) which is a long chain organic compound with a positive hydrophilic head and a hydrophobic tail. The hydrophilic end binds the nanoparticles and controls its growth whereas the hydrophobic end is free. Due to the stearic hindrance there is lower agglomeration in particles. In the absence of surfactant there is higher amount of growth and the particles are highly agglomerated.

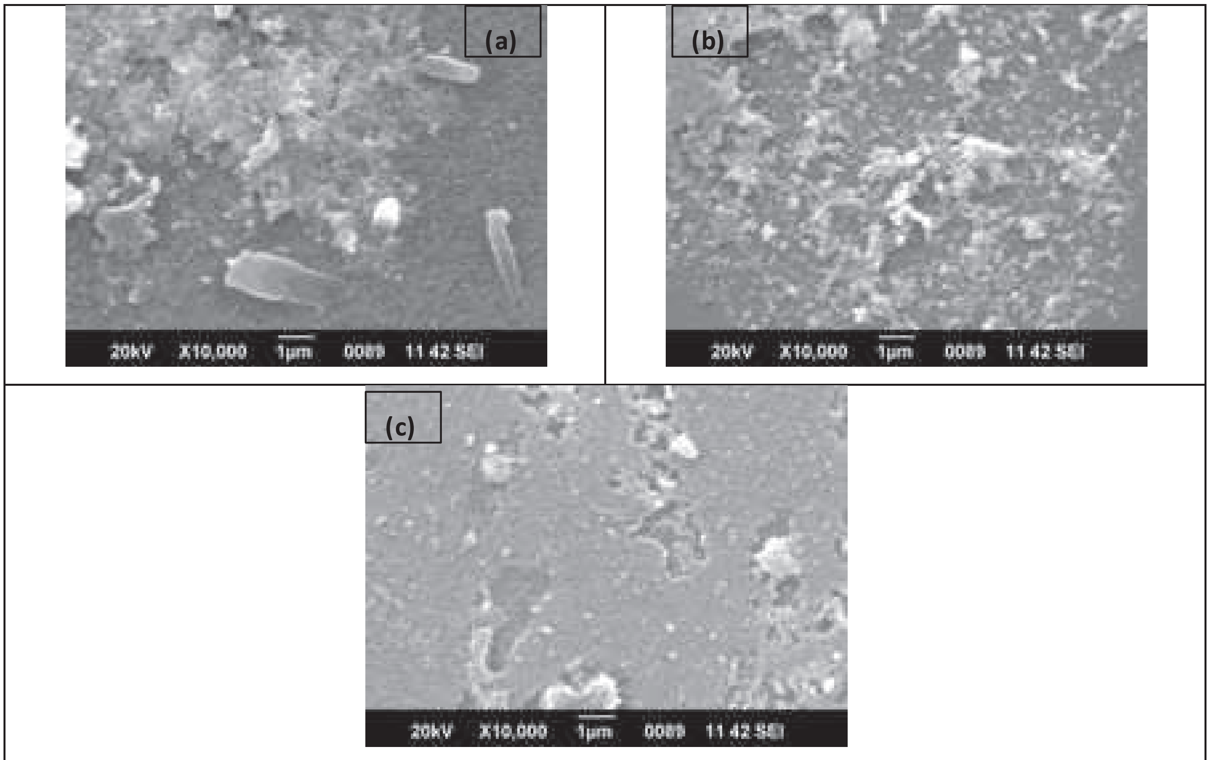


Figure 4.2. SEM images of (a) S1, (b) S2 and (c) S3

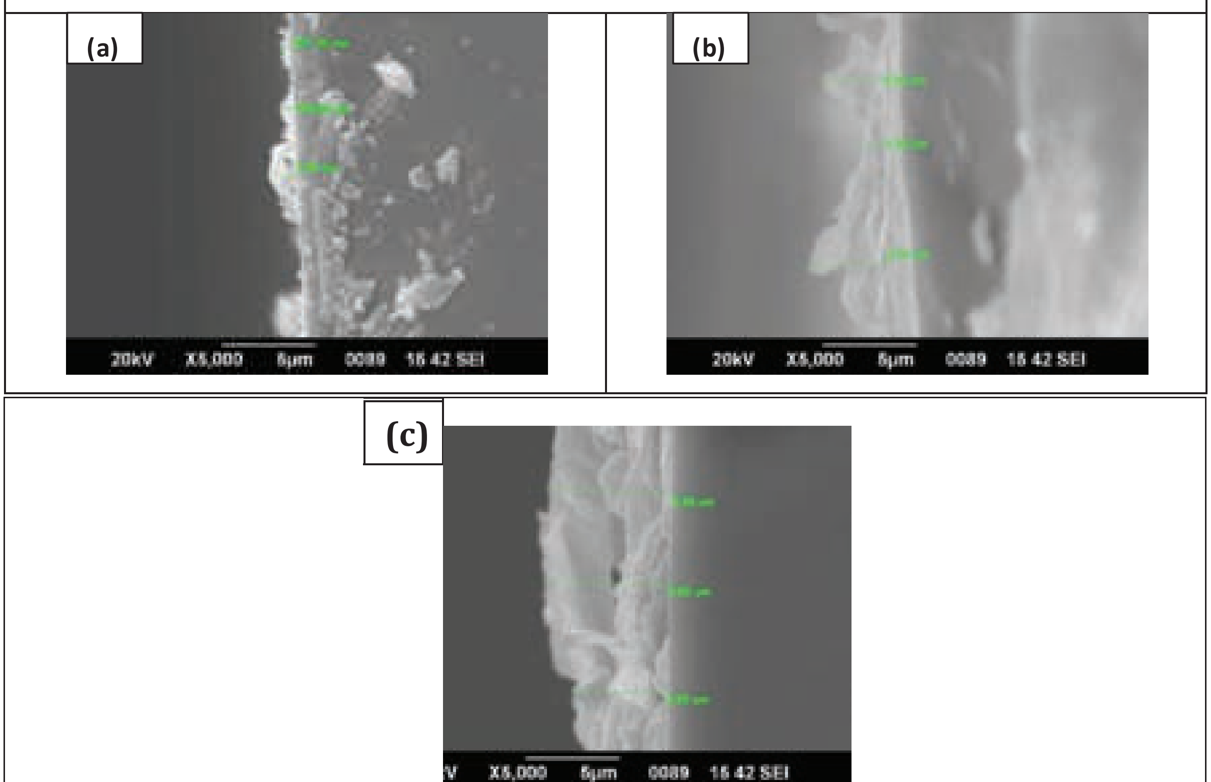


Figure 4.3. Cross Sectional SEM images of (a) S1, (b) S2 and (c) S3

Table 4.2. Thickness of the three samples

Sample	Thin film thickness
S1	601.33 nm
S2	1.24 μm
S3	4.96 μm

4.3. HALL MEASUREMENT

Hall Measurement of the three samples was taken and several parameters such as its semiconducting type, mobility and average Hall coefficient were measured as tabulated in table 4.3. All the three samples were found to be p-type in nature. The mobility of S1 and S2 is fairly high of the order of 10^2. S3 showed higher mobility in the order of 10^3. Although CTAB is used for the confinement of particles, its use hinders the free movement of charge carriers. So mobility was found to be higher when surfactant was not used.

Table 4.3. Hall measurement data of three samples

Sample	Semiconducting type	Mobility (cm^2/Vs)	Average Hall coefficient
S1	p-type	2.077×10^2	3.97×10^5
S2	p-type	2.062×10^2	8.961×10^7
S3	p-type	1.215×10^3	1.16×10^9

CHAPTER V

5. Conclusion

Through this work thin films of copper tin sulphide (CTS) were prepared using SILAR technique and its structural, morphological and electrical measurements were made. Without the use of surfactant, the mobility of the sample showed mobility 10 times that of the samples without surfactant. Owing to high mobility and p-type nature, the samples can be used in the absorber layer of solar cells.

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KERALA AGRICULTURAL UNIVERSITY
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KAU P.O, Vellanikkara, Thrissur, Kerala- 680 656

10th August 2022

DUTY CERTIFICATE

This is to certify that Ms. Keerthy Sophiya Ponnachan, Assistant Professor, Carmel College, Mala has attended the duty as a Resource person for handling a topic on "Techniques for English Language Proficiency" in connection with the orientation programme of Forestry Work Experience (FOWE) of 2019 BSc Forestry batch at the College of Forestry on 10th August 2022.

Dean of Faculty

DEAN OF FACULTY
College Of Forestry
KAU, Vellanikkara,
Thrissur - 680 656

COLLEGE OF FORESTRY
KERALA AGRICULTURAL UNIVERSITY
Date

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No.A/CAS/CHY/LFC/2022

11.07.2022

To

The Principal
Carmel College,
Mala

Respected Sister

Sub: Request for relieving Dr. Princy k G, Associate Professor, Department of Chemistry for the selection committee of Dr. Moly P P, Promotion reg.

Ref: 67418/GA II- SPECIAL CELL-I-ASST-2/2022/Admn. Dated (11/04/2022)

As per the reference cited above Dr. Princy k G, Associate Professor, Department of Chemistry, has been appointed as University nominee for the promotion of Dr. Moly P P, Department of Chemistry, Assistant Professor Academic level 12 to Associate Professor Academic Level 13 A. The meeting of the selection committee has been scheduled at Principal's chamber Little Flower College, Guruvayoor, on 22nd July 2022 at 2 pm. I request you to make necessary arrangements for relieving Dr. Princy k G, to attend the same.

Thanking You

Faithfully,

Valsa. M.A.

PRINCIPAL
Little Flower College
GURUVAYOOR

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Website: www.littleflowercollege.edu.in
E-mail: lfcollegegvr@gmail.com
College : 0487 2556957

DUTY CERTIFICATE

This is to certify that Dr. Princy k G, Associate Professor, Department of Chemistry, Carmel College Mala, has been the University Nominee and Subject Expert as per Letter No: 67418/GA II- SPECIAL CELL-I-ASST-2/2022/Admn. Dated (11/04/2022) for assessing the CAS Promotion documents from Assistant Professor Academic level 12 to Associate Professor Academic Level 13 A of Dr. Moly P P, Department of Chemistry, Little Flower College Guruvayur, 680103 on 22nd of July 2022.

LITTLE FLOWER COLLEGE Accredited by NAAC With A Grade (3.41 CGPA) GURUVAYOOR

Valsa.M.A.
PRINCIPAL
Little Flower College
GURUVAYOOR

Ph. (Off.) : 0480 2701159
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E-mail : shcollegecky@gmail.com Website : www.sacredheartcollege.ac.in

Date :

03.04.2023

WHOMSOEVER IT MAY CONCERN

This is to certify that Dr. Sr. Rini Raphael, Assistant Professor of Zoology, Carmel College, Mala, has discharged duty as Subject Expert in connection with Selection Committee for appointment of Guest Lecturer in the Post Graduate Department of Zoology, Sacred Heart College, Chalakudy on 3rd April 2023.

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PRINCIPAL
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Mrs. Gilu Mathew
MBA, MSC.(psychology)
Life coach, counsellor, Trainer & speaker
Present employment as
Asst. Professor & student counsellor
Carmel College (Autonomous) Mala
Mrs. Gilu Mathew
Inaugural Address : Rev.Sr. Jeena C. M. C
Headmistress
SCGHS kottakkal, Mala
Orientation class : Mrs. Gilu Mathew
Asst. Prof. Carmel College Mala

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General Education Department

SNVVHSS ALOOR

908029

II YEAR OFFICE OPERATIONS EXECUTIVE

OFFLINE EXPERT INTERACTION

Session -I

Topic : Interaction with Motivational Speaker

Counsellor, Speaker & Motivational Trainer

Expert:

GILU MATHEW

Accademic Co-ordinator

Carmel College, Mala

ON 31/08/2022

Time: 3.30 pm To 4.30 pm

GENERAL EDUCATION DEPARTMENT
GOVERNMENT OF KERALA VHSE WING

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INSTITUTE INDUSTRY INTERACTION CELL

SNVVHSS ALOOR (908029)

Accounts Executive

Expert Interaction Class - 1

17 September 2022
11.00AM

Topic:- Scope of effective communication and customer satisfaction

Gilu Mathew

Asst. Prof. Carmel College Mala
Director - GM Formaction
MBA, MSc. (Psychology)

Counsellor, Speaker & Motivational Trainer

IIIC INSTITUTE INDUSTRY INTERACTION CELL

GVHSS PUTHENCHIRA
Dept. Of General Education
VHSE
Govt .Of Kerala

OFFICE OPERATIONS EXECUTIVE

EXPERT INTERACTION SESSION 5

INTERACTION WITH MOTIVATIONAL SPEAKER

19/10/2022
11 AM

GILU MATHEW

ASSISTANT PROFESSOR
CARMEL COLLEGE, MALA

SOCCORSO SCHOOL MALA

conducts a talk on

Positive Parenting

Date
2022
October 14

Time
10:30
am

Venue
Carmel College
Auditorium
Mala

Resource person

Mrs GILU MATHEW
MBA,MSc (Psychology)
Trainer,Counsellor

Asst.Prof.
Carmel College, Mala

ORIENTATION SESSION FOR 10TH GRADERS

AT HOLY CHILD CENTRAL SCHOOL
SNEHAGIRI (ICSE)

ON SATURDAY, 22ND OCTOBER 22

FROM 9:30 AM - 3:30 PM

Resource person

Mrs GILU MATHEW

MBA, M SC PSYCHOLOGY
TRAINER& COUNSELLOR

ASST. PROFESSOR
CARMEL COLLEGE (AUTONOMOUS) MALA

HOW TO CONVERT DREAM INTO ACTION

The Best Way to Mend our children

THURSDAY
17 NOV 2022
10.00 AM

GILU MATHEW
LIFE COACH,
COUNSELLOR,
TRAINER &SPEAKER
ASST. PROFESSOR
&STUDENT
COUNSELLOR

LCEMHS KATTUNGACHITA
@SCHOOL AUDITORIUM

RG

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S N Arts and Science College Women Cell Orientation Programme

Problems faced by youth

Resource Person

GILU MATHEW
MBA, M.Sc. (Psychology)

Life Coach, Counsellor, Trainer and Speaker
Asst. Professor and Student Counsellor
Carmel College (Autonomous) , Mala
Trissur District

Time - 11 am

Date - 15/12/2022

Venue - S N College Auditorium


പരിവർത്തൻ-2022


MENTAL HEALTH CLASS