

“SYNTHESIS AND CHARACTERIZATION OF SILVER NANOPARTICLES USING GUARGUM (GG)”

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BY

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CERTIFICATE

This is to certify that Ms. Rewashri Vyas, a student of final semester M. Tech. (Nanoscience and Technology), Applied Physics Department, during the session 2012-2014 has successfully completed the project work on **“Synthesis and Characterization of silver nanoparticles using guargum (GG)”** at DTU, Delhi and has submitted a satisfactory report in partial fulfillment for the award of the degree of Master of Technology.

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Candidate Declaration

I hereby declare that the work which is being presented in this thesis entitled **“Synthesis and Characterization of silver nanoparticles using guargum (GG)”** is my own work carried out under the guidance of Dr. N. K. Puri, Assistant Professor, Delhi Technological University, and Delhi.

I further declare that the matter embodied in this thesis has not been submitted for the award of any other degree or diploma.

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ABSTRACT

Silver nanoparticles (AgNPs) are under constant investigation due to their novel physical and chemical properties. Their optical, antimicrobial, catalytic and electrical properties make them suitable for variety of applications, at the same time they can be synthesized using various green chemistry techniques.

In this project we have synthesized silver nanoparticles using guar gum (GG), which is a natural polymer as reducing agent. Guar gum also acts as capping agent. Ag/GG composite thin films were fabricated using simple dip coating and spin coating on glass substrates. Various characterization techniques were used to get structural, chemical and optical properties of synthesized materials.

SEM and AFM images revealed that particle size is approximately in 100-150nm range. FTIR spectrum confirms presence of guar gum as capping agent. XRD pattern shows crystalline nature of the films and face centered cubic (FCC) geometry. UV Vis spectrum gives absorption peak at 450 nm, and also gives the approximate optical band gap energy $E_g = 1.86$ eV.

These nanocomposites can find application in semiconductor physics and sensors.

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1. INTRODUCTION

1.1 Concept of nanotechnology

Nanotechnology is the study of manipulating matter on an atomic and molecular scale. Generally, it deals with developing materials, devices, or other structures possessing at least one dimension sized from 1 to 100 nanometers. And at this quantum – realm scale, quantum mechanical effects becomes important.

Nanotechnology is very diverse, ranging from extensions of conventional device physics to completely new approaches based upon molecular self – assembly. It is valuable in developing new materials with dimensions on the nanoscale to investigating whether we can directly control matter on the atomic scale. Nanotechnology evokes the application of fields of science as diverse as surface science, organic chemistry, molecular biology, semiconductor physics, micro fabrication, etc. [1].

1.2 Historical aspects of nanotechnology

Although nanotechnology is a relatively recent development in scientific research, the development of its central concepts happened over a longer period of time. In 1974, Norio Taniguchi introduced the term “Nanotechnology” to represent extra – high precision and ultra – fine dimensions, and also predicted improvements in integrated circuits, optoelectronic devices and computer memory devices. The emergence of nanotechnology in the 1980s was caused by the convergence of experimental advances such as the invention of the scanning tunneling microscope in 1981 and the discovery of fullerenes in 1985. The scanning tunneling microscope, an instrument for imaging surfaces at the atomic level, was developed in 1981 by Gerd Binnig and Heinrich Rohrer at IBM Zurich Research

Laboratory, for which they received the Nobel Prize in Physics in 1986. Fullerenes were discovered in 1985 by Harry Kroto, Richard Smalley, and Robert Curl, who together won the 1996 Nobel Prize in Chemistry.

Around the same time, K. Eric Drexler developed and popularized the concept of nanotechnology and founded the field of Molecular nanotechnology. In 1979, Drexler encountered Richard Feynman's 1959 talk "There's Plenty of Room at the Bottom" [2]. The term "nanotechnology", originally coined by Norio Taniguchi in 1974, was unknowingly appropriated by the Drexler in his 1986 book "Engines of Creation: The Coming Era of Nanotechnology"[3], which proposed the idea of a nanoscale "assembler" which would be able to build a copy of itself and of other items of arbitrary complexity. Drexler's vision of nanotechnology is often called "Molecular Nanotechnology" (MNT) or "molecular manufacturing,"

Applications of nanotechnology, although these were limited to bulk applications of nanomaterials, such as the Silver, Nano platform for using Silver nanoparticles as an antibacterial agent, nanoparticles-based transparent sunscreens, and carbon nanotubes for stain-resistant textiles. In the early 2000s, the field was subject to growing public awareness and controversy, with prominent debates about both its potential implications, exemplified by the Royal Society's report on nanotechnology, as well as the feasibility of the applications envisioned by advocates of molecular nanotechnology, which culminated in the public debate between Eric Drexler and Richard Smalley in 2001 and 2003. Many nations moved to promote and fund research into nanotechnology with programs such as National Nanotechnology Initiative, Centre for Nanotechnology.

1.3 Nanoparticles

Nanoparticles are generally considered to be the structures, consisting of atoms or molecules, with a radius less than 100 nm (or 1000 \AA as $1 \text{ nm} = 10 \text{ \AA}$). As the number of atoms increases in a cluster; the percentage of atoms

on the surface decreases. It is the presence of large percentage of atoms on the surface in the nanostructures, which make them behave differently compared to the bulk structures.

One nanometer (nm) is one billionth, or 10^{-9} , of a meter. By comparison, typical carbon-carbon bond lengths, or the spacing between these atoms in a molecule, are in the range 0.12-0.15 nm, and a DNA double-helix has a diameter round 2 nm. On the other hand, the smallest cellular life-forms, the bacterial of the genus *Mycoplasma*, are round 200 nm in length. By convention, nanotechnology is taken as the scale range 1 to 100 nm following the definition used by the National Nanotechnology Initiative in the US. The lower limit is set by the size of atoms since nanotechnology musty build its devices from atoms and molecules. The upper limit is more or less arbitrary but is around the size that phenomena not observed in large structures start to become apparent and can be made use of in the nano devices. These new phenomena make nanotechnology distinct from devices which are merely miniaturized version of an equivalent macroscopic device; such devices are on the great scale and come within the description of micro technology. As dimensions minimize to nano level the following changes come to the picture:

- Larger Surface to Volume Ratio
- Quantum Confinement
- Quantized Energy States

1.4 Various approaches for nanoparticles synthesis

Various synthesis techniques for nanomaterials have been reported so far. Broadly all techniques can be categorized as

- **Top down approach**
- **Bottom up approach**

The top-down method is the method of breaking up a solid substance; it can be sub-divided into dry and wet grinding. In the dry grinding method the solid substance is ground as a result of a shock, a compression, or by friction, using such popular methods as a jet mill, a hammer mill, a shearing mill, a roller mill, a shock shearing mill, a ball mill, and a tumbling mill. On the other hand, wet grinding of a solid substrate is carried out using a tumbling ball mill, or a vibratory ball mill, a planetary ball mill, a centrifugal fluid mill, an agitating beads mill, a flow conduit beads mill etc.

The bottom-up approach is roughly divided into gaseous phase methods and liquid phase methods. For the former, the chemical vapor deposition method (CVD) involves a chemical reaction, whereas the physical vapor deposition method (PVD) uses cooling of the evaporated material. Although the gaseous phase methods minimize the occurrence of organic impurities in the particles compared to the liquid phase methods, they necessitate the use of complicated vacuum equipment whose disadvantages are the high costs involved and low productivity. The CVD procedure can produce ultrafine particles of less than 1 μ m by the chemical reaction occurring in the gaseous phase. The manufacture of nanoparticles of 10 to 100 nm is possible by careful control of the reaction. Performing the high temperature chemical reaction in the CVD method requires heat sources such as a chemical flame, a plasma process, a laser, or an electric furnace. In the PVD method, the solid material or liquid material is evaporated and the resulting vapor is then cooled rapidly, yielding the desired nanoparticles. To achieve evaporation of the materials one can use an arc discharge method. The simple thermal decomposition method has been particularly fruitful in the production of metal oxide or other types of particles and has been used extensively as a preferred synthetic method in the industrial world. For many years, liquid phase methods have been the major preparation methods of nanoparticles; they can be sub-divided into liquid/liquid methods, and sedimentation methods. Chemical reduction of metal ions is a typical example of a liquid/liquid method, whose principal

advantage is the facile fabrication of particles of various shapes, such as nanorods, nanowires, nanoprisms, nanoplates, and hollow nanoparticles. With the chemical reduction method it is possible to fine-tune the form (shape) and size of the nanoparticles by changing the reducing agent, the dispersing agent, the reaction time and the temperature.

The chemical reduction method which we have employed for synthesis in this report carries out chemical reduction of the metal ions to their 0 oxidation states (i.e $M^{n+} \rightarrow M^0$), the process uses non-complicated equipment or instruments, and can yield large quantities of nanoparticles at a low cost in a short time. Besides the chemical reduction method which adds a reducing agent (direct reduction method), other reduction methods are known, such as photoreduction using gamma rays, ultrasonic waves, and liquid plasma which can be used to prepare nanoparticles. These methods that do not use a chemical reducing substance have the attractive feature that no extraneous impurities are added to the nanoparticles. Other than these methods, spray drying, spray pyrolysis, solvothermal synthesis methods are also known.

The general technique in the sedimentation method is a sol-gel process, which has been used extensively for the fabrication of metal oxide nanoparticles. This procedure transforms a solution of a metal alkoxide into a sol by hydrolysis, followed by polycondensation to a gel.

2. REVIEW OF LITERATURE

2.1 History of nanosilver

From the historical point of view, synthesis of citrate stabilized colloidal silver was reported as early as 1889 by M.C. Lea. In the early 1890 nanosilver was one of the earliest materials that was motivation for colloidal science which deals with synthesis and characterization of extremely small particles [4]. In the following years stabilization of silver nanoparticles with proteins in 1902, Under the name “Collargol” such a kind of nanosilver has been manufactured commercially since 1907 and gelatin stabilized silver nanoparticles were synthesized in 1953 [5].

2.2 Motivation for synthesis of silver nanoparticles

2.2.1 Properties of silver and silver nanoparticles

Silver is basic element making up our planet, but is rare, and naturally occurring element. silver is slightly harder than gold and ductile and malleable. Pure silver has the highest electrical and thermal conductivity of all metals and has the lowest contact resistance. Silver can be present in four different oxidation states: Ag^0 , Ag^{2+} , Ag^{3+} . The former two are the most abundant ones, the latter are unstable in the aquatic environment. Metallic silver itself is insoluble in water, but metallic salts such as AgNO_3 and Silver chloride are soluble in water [6].

Silver nanoparticles are of interest because of the unique properties (i.e. tunable optical, electrical, and magnetic properties) which can be incorporated into antimicrobial applications, biosensor materials, composite fibers, cryogenic superconducting materials, cosmetic products, and electronic components and optoelectronic devices.

2.2.1.1 Optical properties

There is growing interest in utilizing the optical properties of silver nanoparticles as the functional component in various products and sensors. Silver nanoparticles are extraordinarily efficient at absorbing and scattering light and, unlike many dyes and pigments, have a color that depends upon the size and the shape of the particle. The strong interaction of the silver nanoparticles with light occurs because the conduction electrons on the metal surface undergo a collective oscillation when excited by light at specific wavelengths. Known as a surface plasmon resonance (SPR), this oscillation results in unusually strong scattering and absorption properties. In fact, silver nanoparticles can have effective extinction (scattering + absorption) cross sections up to ten times larger than their physical cross section. The strong scattering cross section allows for sub 100 nm nanoparticles to be easily visualized with a conventional microscope. A unique property of spherical silver nanoparticles is that this SPR peak wavelength can be tuned from 400 nm (violet light) to 530 nm (green light) by changing the particle size and the local refractive index near the particle surface. Even larger shifts of the SPR peak wavelength out into the infrared region of the electromagnetic spectrum can be achieved by producing silver nanoparticles with rod or plate shapes [7-8].

2.2.1.2 Antimicrobial properties

Silver is a safe and effective anti-bactericidal metal because it is non-toxic to animal cells and highly toxic to bacteria such as *Escherchia coli* (E.coli) and *Staphylococcus aureus* [9]. Silver based compounds have been used in recent years to prevent bacterial growth in applications such as burn care. Nano silver in the form of powders as well as suspensions, due to the high surface to volume ratios, has been used as anti-bacterial because it enables the loading of small quantities of silver and thus makes the product cost effective. Silver nanoparticles are harmful to bacteria, they bind closely to the surface of microorganisms causing visible damage to the cells, and demonstrating good self-assembling ability [10].

Silver ions (Ag^+) and its compounds are highly toxic to microorganism exhibiting strong biocidal effect on many species of bacteria but have a low toxicity towards animal cells. Bactericidal behavior of nanoparticles is attributed to the presence of electronic effects that are brought about as a result of change in local electronic structure of the surface due to smaller sizes. The effects are considered to be contributing towards enhancement of reactivity of silver nanoparticles surface. Silver in ionic form strongly interacts with thiol groups of vital enzymes and inactivates them. That lead DNA loses its replication ability once the bacteria are treated with silver ions [11].

2.2.1.3 Catalytic properties

Due to high surface area and high surface energy predetermine metal nanoparticles for being effective catalytic medium. Growing small particles of silver have been observed to be more effective catalysts than stable colloidal particles. These growing particles catalyzed the borohydride reduction of several organic dyes. The reduction rate catalyzed by growing particles is distinctly faster compared to that of stable and larger silver particles, which are the final products of growing particles. Catalytic activity of silver nanoparticles can be controlled by its size, as redox potential depends on the nanoparticle size [12].

2.2.1.4 Electrical properties

Conductivity of various metal nanoparticles embedded in a polymer matrix has been an important field for research. Bulk silver is an excellent conductor of electricity. In case of silver nanoparticles it has been reported that conductivity of thin films depend upon film thickness and particle loading, films with thickness lower than 150 nm shows dielectric behavior and then with increasing thickness causes transition from dielectric region to semiconducting and conducting zone. [13]

Most of the research on silver nanoparticles investigate their antimicrobial activities and optical properties, but their electronic properties are also gaining attention these days and hence silver nanoparticles embedded in a natural polymer are under investigation in this report.

2.2.2 Application of silver nanoparticles

Silver nanoparticles find applications in variety of fields. Most investigated applications of silver nanoparticles are in pharmaceuticals, medicine and dentistry. They can be used to cure various infections due to their antimicrobial properties. They find applications in treatment of dermatitis, treatment of ulcerative colitis, remote laser light-induced opening of microcapsules silver/dendrimer nanocomposite for cell labeling molecular imaging of cancer cells [14].

In electronic industries, direct writing technologies have been introduced where the material having desirable electronic properties is deposited in particulate form on the substrate and then converted into conductive elements. Due to their high electrical conductivity, inks based on silver nanoparticles are excellent candidates for inkjet printable electronics. One of the major challenges in the inkjet printing technology is the formulation of the ink. The inks have to meet strict physicochemical properties (viscosity, surface tension, adhesion to a substrate, etc.) to achieve optimal performance and reliability of the printing system and to obtain good printed patterns. Several specific characteristics of metallic nanoparticles, such as enhanced reactivity of surface atoms, decrease in melting point with size, and high electric conductivity, make them very attractive for the fabrication of electronic devices [15].

2.3 Various synthesis routes for silver nanoparticles

Like all other nanomaterials silver nanoparticles can be synthesized by top down and bottom up approaches. physical, chemical, biological and photochemical methods have been reported. CVD and PVD and sputter deposition techniques are very popular but they are not very cost effective. In case of noble metals chemical reduction method is most suitable from economical point of view and simplicity.

2.4 Green chemistry approaches

Green chemistry encourages the design and synthesis of processes and products that minimize the use and generation of toxic and hazardous substances. Green chemistry applies to chemical engineering, biotechnology, nanotechnology and physical chemistry. It promotes uses of natural, nontoxic and environment friendly products and raw material.

Various synthesis routes have been reported employing green chemistry. silver NPs have been synthesized using various plant extracts (phytochemicals) as mulberry leaves extract, *Azadirachta indica* leaves extract, *Ocimum tenuiflorum* leaves extract, grape fruit extract, *cucuma longa* extract, aloe vera extract[16-19].

Various chemical reducing agents as sodium alginate, chitasan, glucose, are also used and are nontoxic in nature[20].

2.5 Reduction of silver salts using guar gum

Guar gum is primarily the ground endosperm of guar beans. The guar seeds are dehusked, milled and screened to obtain the guar gum. It is a free-flowing, off-white powder.

The most important property of guar gum is its ability to hydrate rapidly in cold water to attain uniform and very high viscosity at relatively low concentrations. Another advantage associated with guar gum is that it is soluble in hot & cold water and provides full viscosity in even cold water. Apart from being the most cost-effective stabilizer and emulsifier it provides texture improvement, and water-binding; and controls crystal formation. It is inert in nature.

while synthesizing silver nanoparticles using guar gum it acts as reducing agent as well as capping and stabilizing agent. guar gum can be used with some other reducing agent and can act as stabilizing agent ,with this idea we have used guar gum (cyamopsis tetragonolobus), and turmeric(*curcuma longa*) together to reduce silver salt.

silver and guar gum composites have been reported to fabricate gas sensor in ammonia sensing. further applications of these nanocomposites are under investigation.[21]

3. MATERIALS AND METHODS

3.1 materials for synthesis

3.1.1 Silver nitrate (AgNO_3)

Silver salt used as precursor and silver source was silver nitrate, and was purchased from central drug house (CDH), New Delhi. AgNO_3 was laboratory reagent and of high purity (99.98%).

Molecular weight of AgNO_3 is 169.87 and is a colorless or white crystalline solid which darkens on exposure to radiation or organic materials, therefore kept in a black sheet and opaque container to prevent from decomposition.



Fig. 3.1 silver nitrate (AgNO_3)

3.1.2 Reducing agents

For reduction of silver nitrate solution in elemental silver we have used guar gum which is a natural polymer. Guar gum acts as reducing agent as well as capping agent in the solution. Laboratory grade guar gum was purchased from Rajat Dyes and Chemicals, New Delhi. Other green and non-toxic reducing agent turmeric (*Curcuma longa*) is also investigated.



Fig 3.2 (a) guar gum (b) turmeric powder

3.2 Experimental procedure and flow chart

3.2.1 Experimental procedure

For synthesis of silver nanoparticles 2% (w/v) solution of guar gum powder was prepared in 10 ml of distilled water with the help of magnetic stirrer, as guar gum is completely soluble even in cold water. After complete dissolution, temperature of reaction medium was raised to 50⁰C. Then 10 ml of 20 mM silver nitrate solution was added drop wise with help of dropper, this reaction mixture was stirred continuously for 60 minutes with help of magnetic stirrer. Similar procedure was followed for sodium alginate powder and turmeric powder. In case of reduction with turmeric powder guar gum was also used as stabilizing and capping agent. For guar gum different concentrations of AgNO₃ solution i.e. 20mM, 40mM, 60mM and 80mM were also investigated.

All the glassware used was cleaned with the help of ultrasonic cleaner YJ5120-1 for 10 minutes at 40⁰ C.



Fig. 3.3 ultrasonic cleaner

Guar gum and sodium alginate solutions were made by simple dissolution of powder in water while turmeric solution was further filtered to prepare clear solution which is free from insoluble impurities.



Fig. 3.4 magnetic stirrer with hot plate

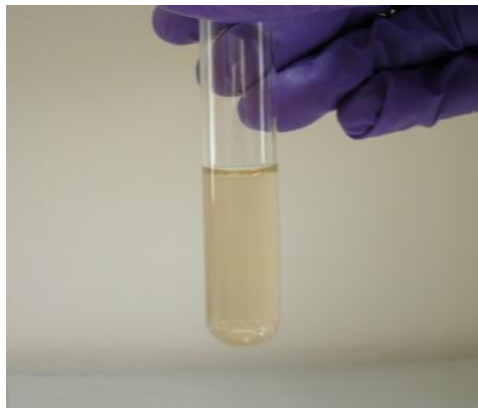
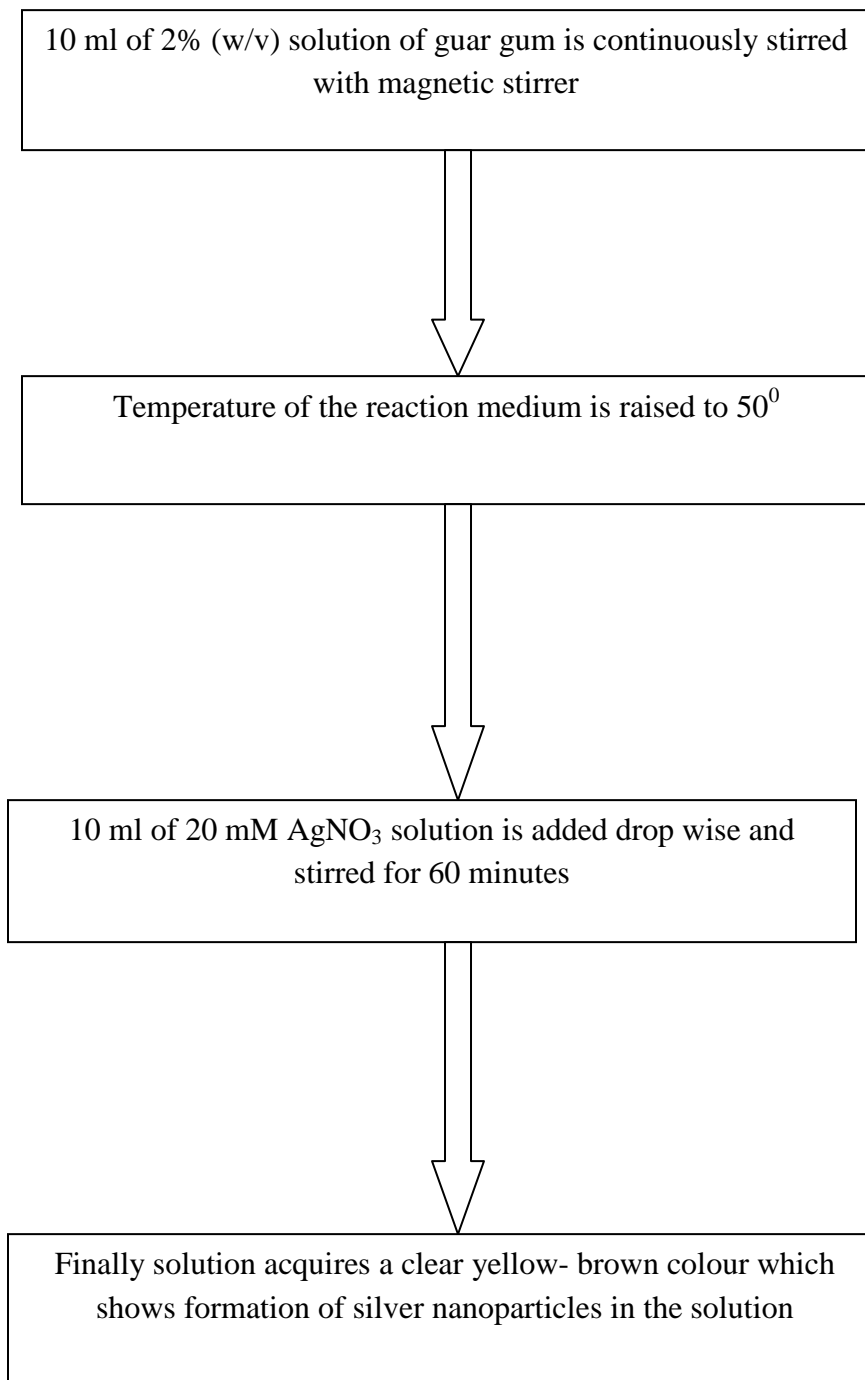


Fig 3.5 silver nanoparticles in solution

3.2.2 Flowchart for the experimental procedure



3.3 Characterization techniques and sample preparation

Characterization of nanomaterials is as important as their synthesis. Imaging in nanoscience does not merely mean creation of the image but also its deep understanding and analysis. Many methods are available to image nanostructured materials and to characterize their chemical and physical properties. In general, characterization techniques can be broadly categorized as

- Microscopic techniques for imaging.
- Spectroscopic techniques for analysis of physical and chemical properties.

Following are the characterization techniques employed in this project, to characterize synthesized silver nanocomposites. Also the sample preparation methods are described as different techniques require differently prepared samples.

3.3.1. UV-Vis-NIR spectroscopy

Ultraviolet and visible spectrometers are very important characterization and have been in research for last 35 years.

When a beam of radiation strikes any object it can be absorbed, transmitted, scattered, reflected or it can excite fluorescence. With scattering it can be considered that the radiation is first absorbed then almost instantaneously completely re-emitted uniformly in all directions, but otherwise unchanged. With fluorescence a photon is first absorbed and excites the molecule to a higher energy state, but the molecule then drops back to an intermediate energy level by re-emitting a photon. The processes concerned in absorption spectrometry are absorption and transmission. Usually the conditions under which the sample is examined are chosen to keep reflection, scatter and fluorescence to a minimum.

With solid sample it is usually found that the material is in a condition unsuitable for direct spectrometry. The refractive index of the material is high and a large proportion of the radiation may be lost by random reflection or refraction at the surface or in the mass. Unless the sample can be easily made as an homogenous polished block or film, it is usual to eliminate these interfaces by dissolving it in a transparent solvent.

Liquids may be contained in a vessel made of transparent material such as silica, glass or plastic, known as a cuvette. The faces of these cells through which the radiation passes are highly polished to keep reflection and scatter losses to a minimum.

We have recorded absorption spectra for silver samples using perkin elmer lambda 750 spectrometer.



Fig 3.6 UV Vis spectroscopy

Sample preparation

Our samples obtained were in liquid form and showed high viscosity, so 1:10 solution of sample was prepared in distilled water as solvent. Before recording spectra samples were ultra sonicated. Absorption spectrum was recorded at 1nm interval and from 250 nm to 700 nm.

3.3.2 Scanning electron microscope (SEM)

Scanning electron microscopy (SEM) is a powerful and popular technique for imaging the surfaces of almost any material with a resolution down to about 1 nm. The scanning electron microscope (SEM) used a focused beam of high-energy electrons to generate a variety of signals at the surface of solid specimens. It utilizes the secondary electrons (SEs) and backscattered electrons (BSEs) being emitted from different areas of the specimen as well as the trajectories they take in relation to the location of the detector. The signals that derive from electron sample interactions reveal information about the sample including its size, shape, external morphology (texture). 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 (using EDS), crystalline structure, and crystal orientations (using EBSD).

Surface morphology of sample is examined using scanning electron microscope (Hitachi s-3700N).



Fig 3.7 scanning electron microscope

Sample preparation

For characterization with SEM thin films of the samples were fabricated on glass slides with dip coating and were allowed to dried at room temperature only. Further they were scratched from glass slides and carefully placed on carbon tape and coated with gold to make these films conductive.



Fig 3.8 sample plate and coating unit

3.3.3 Energy dispersive analysis of X-rays (EDAX)

EDAX is a widely used technique to analyze the chemical components in a material under SEM. This method detects the X-rays produced as the result of the electron beam interactions with the sample. Mapping of the distribution of the different chemical elements constituting the specimen can be obtained. X-ray data is processed to obtain the percentage of each measured element present in the individual particles. The compositional and morphological data are then combined for data analysis.

EDAX equipment was attached to SEM, and same samples were characterized for energy dispersive analysis.

3.3.4 Atomic force microscopy (AFM)

The basic principle of atomic force microscopy is that it measures interactive forces (attractive or repulsive) forces between the probe tip and surface of the sample. Probe is located at the end of a flexible cantilever. Cantilever scans the surface and fluctuations are recorded as deflection of a laser beam that bounces off reflective back of the cantilever and an optical detector is employed to detect this. Probe is continuously moved on the surface and cantilever deflection is monitored constantly. A feedback loop changes the height of the probe on surface in order to keep atomic force constant thus vertical movement of the tip is recorded to create a topographic image of the sample surface.

In order to get the 3D image of the sample park systems XE-100 was used and spin coated films of the sample on glass substrates were used.

3.3.4 Fourier transform infrared spectroscopy (FTIR)

FTIR (Fourier Transform Infrared) or simply FTIR Analysis, is most useful technique for identifying chemicals that are either organic or inorganic. It can be applied to the analysis of solids, liquids, and gasses. FTIR spectroscopy is a multiplexing technique, where all optical frequencies from the source are observed simultaneously over a period of time known as scan time. In this technique whole data is collected and converted from an interference pattern to a spectrum.

By interpreting the infrared absorption spectrum, the chemical bonds (functional groups) in a molecule or molecular structure of materials, whether organic or inorganic can be determined. FTIR spectra of pure compounds are generally so unique that they are like a molecular “fingerprint”. While organic compounds have very rich, detailed spectra, inorganic compounds are usually much simpler. The technique works on the fact that

bonds and groups of bonds vibrate at characteristic frequencies. A molecule that is exposed to infrared rays absorbs infrared energy at frequencies which are characteristic to that molecule i.e. those frequencies where the infrared light affects the dipolar moment of the molecule. Thus monatomic (He, Ne, Ar, etc) and homopolar diatomic (H₂, N₂, O₂, etc) molecules do not absorb infrared light. During FTIR analysis, a spot on the specimen is subjected to a modulated IR beam. The specimen's transmittance and reflectance of the infrared rays at different frequencies is translated into an IR absorption plot consisting of reverse peaks. The resulting FTIR spectral pattern is then analyzed and matched with known signatures of identified materials in the FTIR library

In general FTIR spectra for analysis purpose is divided into two regions

1. Functional group region which extends from 4000 cm⁻¹ to 1450 cm⁻¹
2. Fingerprint group region which extends from 1450 cm⁻¹ to 400 cm⁻¹

3.3.5 X-ray diffractometer (XRD)

X-ray diffraction (XRD) is a versatile, non-destructive analytical technique for identification and quantitative determination of the various crystalline forms, known as “phase” of compounds present in powdered and solid samples. The result of an XRD measurement is called a XRD pattern or diffractogram. Following information can be obtained from the pattern

- Phases present from peak positions
- Phase concentrations from peak heights
- Amorphous content from background bump
- Crystallite size from peak widths

A perfect crystal would extend in all directions to infinity, so no crystal is perfect due to its finite size. This deviation from perfect crystallinity leads to a broadening of the diffraction peaks. However, above a certain size (100-500nm) this type of broadening is negligible. Scherrer (1918) first observed that small crystallite size could give rise to line broadening. He derived a well-known equation for relating the crystallite size to the broadening, which is called Debye-Scherrer formula is given by

$$D = \frac{k\lambda}{\beta \cos \theta} \dots\dots\dots(3.1)$$

Where

D = average crystallite size

k = Scherrer's constant whose value varies from 0.89 to 1

λ = wavelength of the radiation used

β = full width at half maxima in radians for a certain peak position

4. RESULTS AND DISCUSSIONS

4.1 UV Vis spectrum

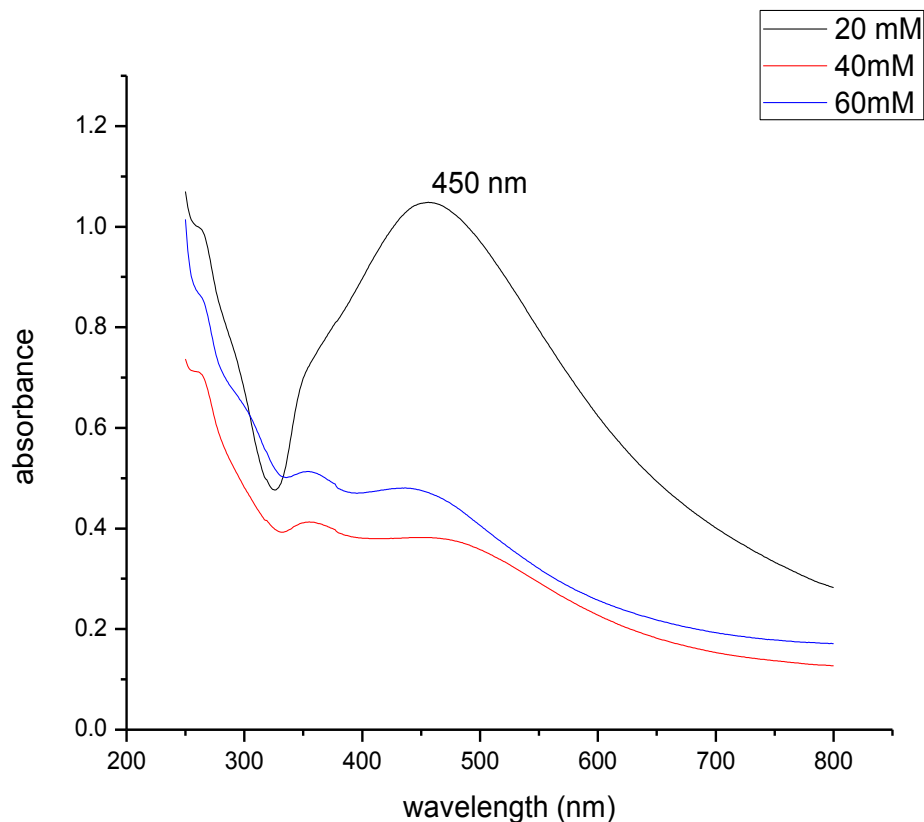


Fig. 4.1 absorption spectra of guar gum samples

Absorption spectrum shows sharp peak at 450 nm for 20 mM sample. Further increasing concentration of AgNO₃ gives rise to double peaks. Second peak at around 320 nm shows presence of AgNO₃ in sample which has not been reduced due to insufficient concentration of guar gum. The absorption peak of pure AgNO₃ solution comes at 310 nm.

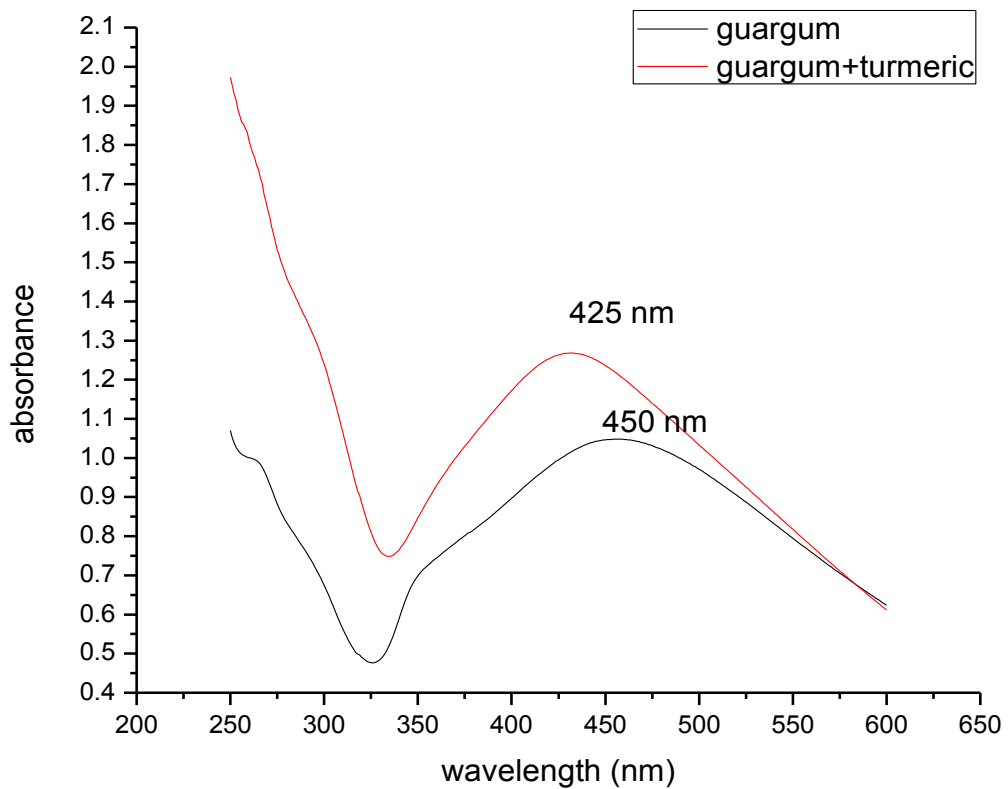


Fig. 4.2 absorption spectra of guar gum and guar gum/turmeric

When turmeric is added as reducing agent spectrum shows a blue shift. from the cut off value of λ_{max} approximate value of optical band gap is determined

For guar gum sample $\lambda_{\text{max}} = 664 \text{ nm}$

Value of optical band gap energy = 1.86 eV

For guar gum+turmeric sample $\lambda_{\text{max}} = 649 \text{ nm}$

Value of optical band gap energy = 1.91 eV

4.2 SEM and EDAX results

4.2.1 SEM

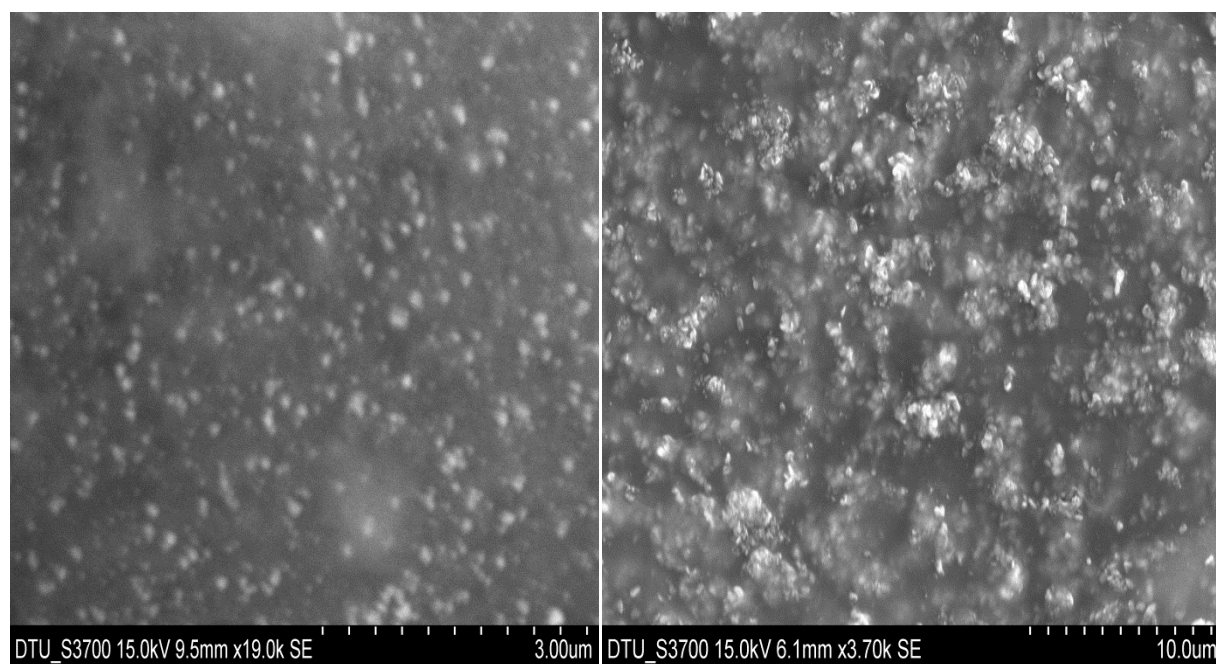


Fig 4.3 (a) guar gum sample (b) guar gum/ turmeric sample

SEM images reveal that particles have smaller size and less agglomeration in case of guar gum sample than the latter. From SEM images particle size in first sample is approximately 100-150 nm.

4.2.2 EDAX

EDAX results showed 95.57 % silver by weight in the guargum sample. and 52.97% in the guargum/turmeric sample.

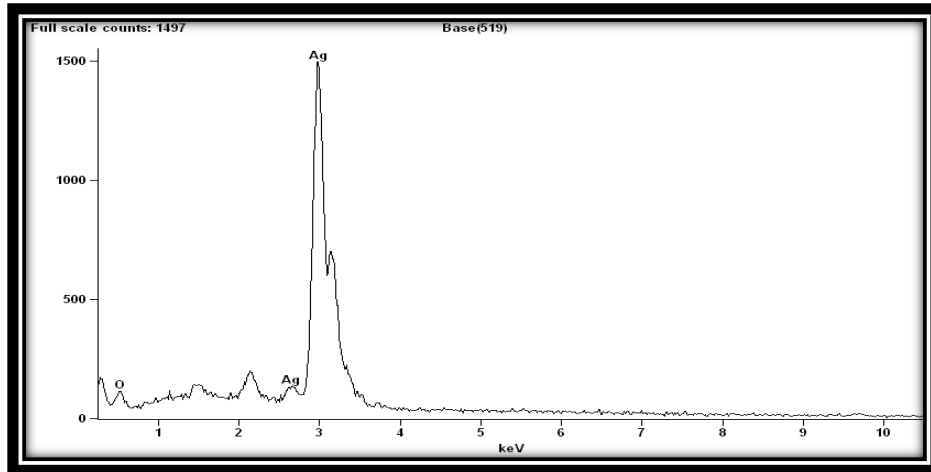


Fig 4.4 EDAX spectrum of guargum sample

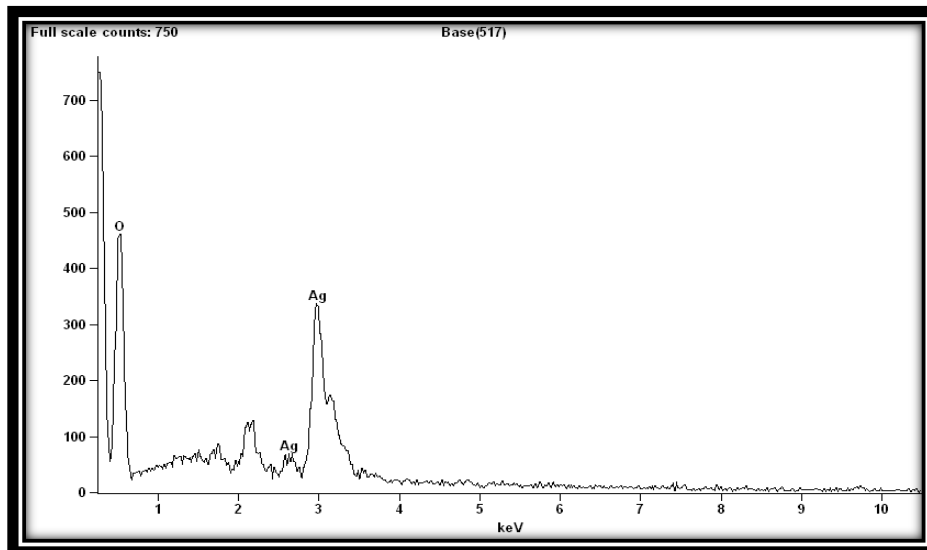


Fig 4.5 EDAX spectrum of guargum/turmeric sample

4.3 Atomic force microscopy (AFM)

Surface topology of the sample was studied with the park systems XE-100 atomic force microscopy.

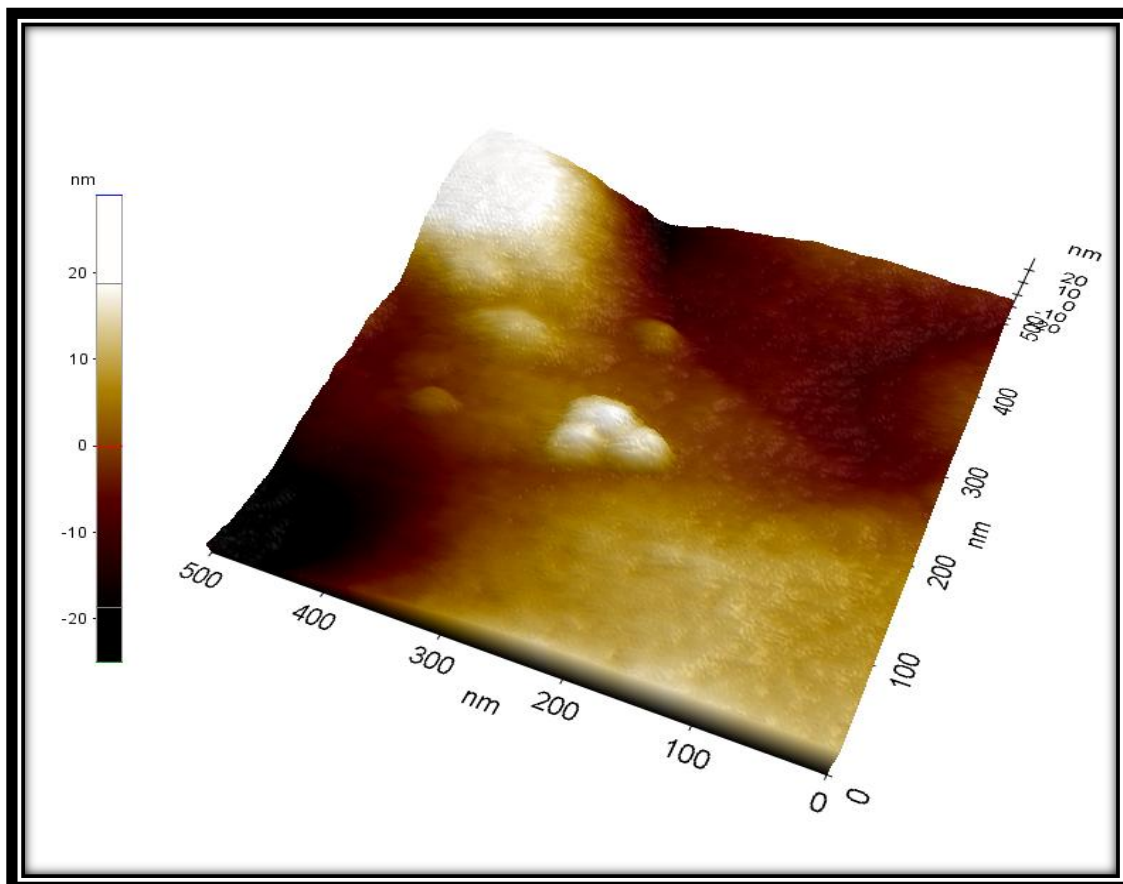


Fig. 4.6 AFM image of guargum sample

AFM image gives the three dimensional surface morphology of the sample. AFM image also shows particle in the range 100-150 nm, Also agglomeration of particles can be seen.

4.4 Fourier transform infrared spectrum (FTIR)

FTIR spectra were recorded with thermo scientific nicholet 380 FTIR spectroscope. These spectra were recorded from 4000 cm^{-1} to 500 cm^{-1} .

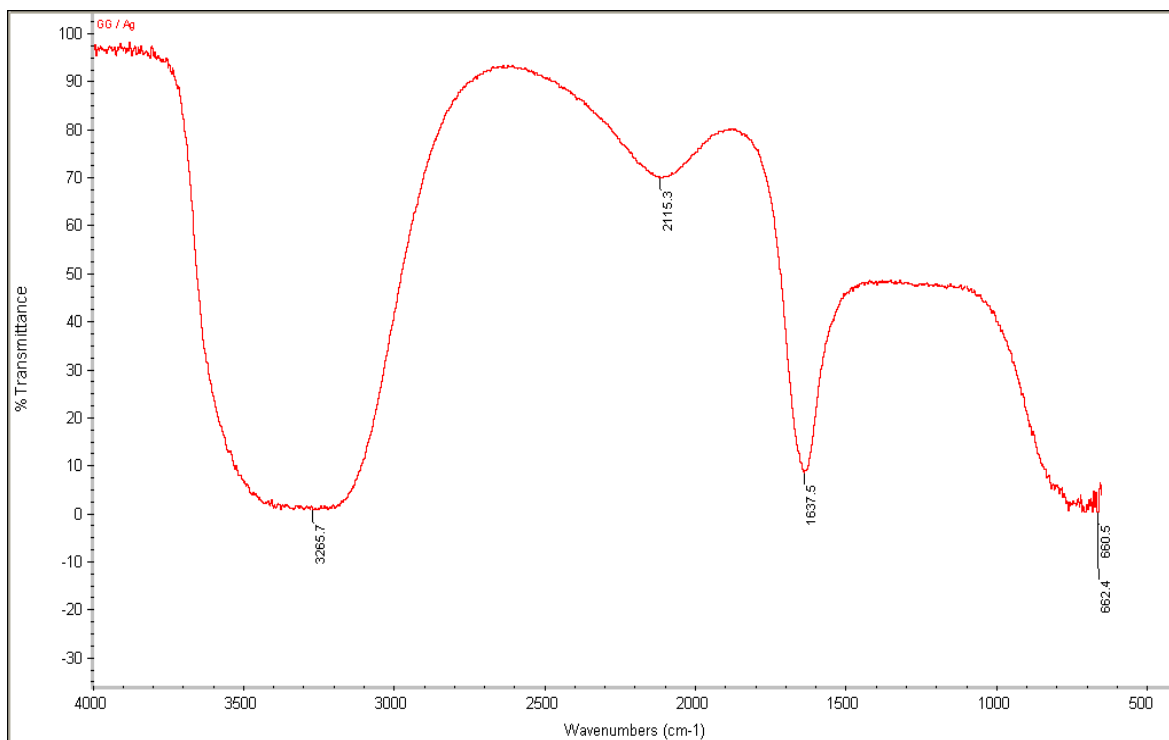


fig 4.7 FTIR spectrum of guar gum sample

FTIR spectrum can be divided in two regions

1. Functional group region (4000 cm^{-1} to 1450 cm^{-1})

In this region three downward peaks at 3265.7 cm^{-1} , 2115.3 cm^{-1} , 1637.5 cm^{-1} are observed. From FTIR data base it is determined that first peak which is broad and wide correspond to OH bond present in structure of guar gum. Second and third peaks may

correspond to $C=C$ and $C-C$ respectively. Thus this region conforms the presence of guar gum as capping agent.

2. Fingerprint group region (1450 cm^{-1} to 500 cm^{-1})

Each molecule has its own fingerprint absorption in this region which means two different molecules will not have same downward peak in this region. For our sample peak in this region comes at 600.5 cm^{-1} .

4.5 X- ray differactogram (XRD)

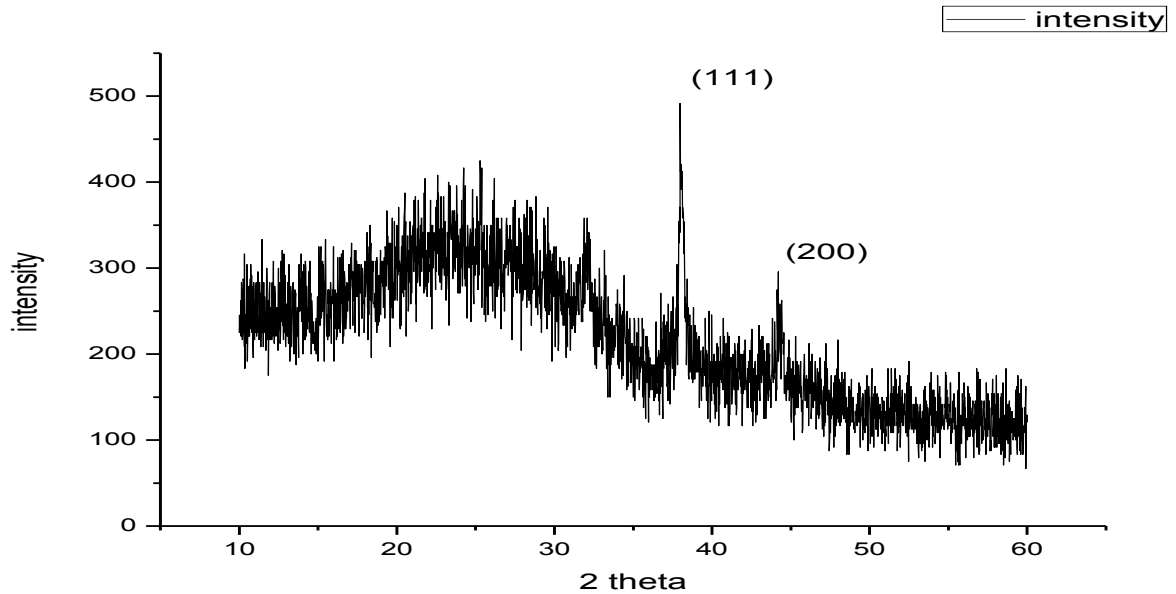


Fig 4.8 X ray diffraction pattern of guar gum sample

The X ray diffraction pattern of the guar gum sample is plotted here. the presence of peaks at 38.07° and 44.21° confirms the presence of face centered cubic phase of elemental silver according to JCPDS files corresponding to planes as (111) and (200).

Using Sherrer’s formula and taking $k = 0.9$ and $\lambda = 1.54 \text{ \AA}$, crystallite size corresponding to the first peak comes out to be 2.43 \AA .

Further calculating lattice parameter using following equation

$$a = d * (h^2 + k^2 + l^2)^{1/2} \dots\dots\dots(4.1)$$

comes out to be 4.20 \AA which is in good agreement with 4.08 \AA .

5. CONCLUSION AND SCOPE OF FUTURE WORK

In summary, we have synthesized silver and guar gum nanoparticles nanocomposites (Ag/GG nanocomposites) and fabricated their films using dip coating and spin coating.

Sample characterization were carried out in order to determine their physical and chemical properties. UV vis spectrum reveals about the absorption peak and give the approximate idea about band gap energy, which indicates the semiconducting properties.

SEM and AFM images reveal that particles are nearly in the 100-150 nm range and evenly distributed. agglomeration of some particles was also observed.

FTIR spectrum confirms the presence of capping agent guar gum on nanoparticles. XRD pattern confirms the crystalline nature of sample and also face centered cubic phase.

Further investigations can be carried out to determine conductivity of these films and their applications in semiconductor electronics and sensing.

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