## SYNTHESIS OF SILVER NANOPARTICLES BY EXTRACT OF NEEM (AZADIRACHTA), TULSI(OCIMUM TENUIFLORUM) AND MINT(MENTHA) LEAVES USING GREEN SYNTHESIS METHOD

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#### **CANDIDATE'S DECLARATION**

I, Rahul Kundara, Roll No. 2K18/NST/05 of M.Tech. Nanoscience and Technology, hereby declare that the project Dissertation titled "SYNTHESIS OF SILVER NANOPARTICLES BY EXTRACT OF NEEM (AZADIRACHTA) ,TULSI(OCIMUM TENUIFLORUM) AND MINT(MENTHA) LEAVES USING GREEN SYNTHESIS METHOD" which is submitted by me to the Department of Applied Physics, Delhi Technological University, Delhi in partial fulfillment of the requirement for the award of the degree of Master of technology, is original and not copied from any source without proper citation. This work has not previously formed the basis for the award of any Degree, Diploma Associateship, Fellowship or other similar title or recognition.

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#### CERTIFICATE

I hereby certify that the project dissertation titled "SYNTHESIS OF SILVER NANOPARTICLES BY EXTRACT OF NEEM (AZADIRACHTA), TULSI (OCIMUM TENUIFLORUM) AND MINT (MENTHA) LEAVES USING GREEN SYNTHESIS METHOD" by Rahul Kundara, Roll No. 2K18/NST/05, Department of Applied Physics, Delhi in partial fulfillment of the requirement for the award of the degree of Master of Technology, is a record of the project work carried out by the student under my supervision. As per the declaration of the candidate and to the best of my knowledge, this work has not been submitted or full for any Degree or Diploma to this University or elsewhere.

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## List of Symbols, abbreviations

NPs	Nanoparticles
AgNPs	Silver nanoparticles
mM	Millimolar
%	Percentage
°C	Degree centigrade
1	Per

~	Approximately
Mm	Micro mole
Ag+	Silver ions
AgNO3	Silver nitrate
DI	Deionized water
DW	Distilled water
mm	Millimeter
min	Minute
SC	Semiconductor
NaOH	Sodium Hydroxide
nm	Nano meter
Α	Absorbance
Т	Transmittance
рН	Power of hydrogen
SPR	Surface Plasmon Resonance
Ι	Intensity
XRD	X-Ray diffraction
λ	Wavelength
F.C.C	Face Centered Cubic
FWHM	Full Width Half Maxima

#### ABSTRACT

There are several possible uses of silver nanoparticles (AgNPs) in energy storage systems, antimicrobial sensors, and other biological sensors. Silver nanoparticles were synthesized using extract obtained from leaves of Azadirachta Indica (Neem), Ocimum Tenuiflorum (Tulsi), and Mentha (Mint). Thin films of various samples were then prepared using drop-cast method. In order to study their structural properties, they are then characterized with different analytic techniques like X-ray diffractometer (XRD) and ultraviolet visible Spectroscopy. To compare the particle size of NPs obtained from different plant leaves, XRD characterization was done. XRD patterns ratifies synthesis of silver nanoparticles in single crystalline phase and confirms the FCC structure with a particles size of 13.58 nm (obtained from neem extract). UV-Visible absorption spectrum for different samples neem, tulsi and mint were recorded in the wavelength ranges of (270-600) nm.

## CHAPTER 1 INTRODUCTION

#### **1.1 Introduction to Nanotechnology:**

Nanotechnology states that an atomic or molecular scale study of controlling matter, i.e. having dimensions of approximately 1-100 nm. Nanotechnology is an area of great multidisciplinary importance. In nanotechnology, two primary methods practised. By "bottom-up" approach, materials and devices are constructing or fabricating by the chemically assembled molecular atoms and In "top-down" approach the nano sized objects are created without atomic level control from large entities. On the nanoscale, materials' physical, chemical and biological properties are profoundly and valuably different with the attributes of particular molecules or atoms. Examples of nanotechnology is made of polymers based on molecular structure, and computer chip layout design is focused on Surface science.

Nano-sized substance classified like a collection of substances where at least one of dimension at least is smaller than about 100nanometers. one nm is about 100,000 times the one millionth of a millimeter lesser than diameter of human hair. Nano-particles are the topic of interest because unique thermal, optical, magnetic, electronic, electrical, physical and other attributes come out at this scale. All these emerging properties at nanoscale have the potential for large impacts in the fields of cancer treatment, electronics, optics and other fields also.

Engineered Nano-materials are modality design at level of the molecule (nanometer) take significance of the miniature size and recent attributes generally seen in their traditional, bulk(larger or accumulated part) specimen. Two key explanations what is cause of materials may consist of various attributes at the nanoscale are, first, Increment in the relatival surface area & secondly, the recent quantum effect. Nano-materials have a very larger surface area to volume ratio as compare to their bulk form, which may proceed to larger chemical reactivity & affect theirs potency also. Quantum effect can as well as become even more vital in assessing material attributes and characteristize at the nanoscale, resulting in optical, physical, electronics, electrical and magnetic properties[1-2]. These emerging productions having the potential for larger impacts in area of telecommunications, target drug delivery in medicine and other fields as well[3-7]. The products obtained from the synthesis have been characterized by the use of both chemical and instrumental methods. Using that instrumental method the magnetic and electrical properties of silver nanoparticles were investigated.

#### **1.2 Nanomaterials:**

Nanomaterials are foundation stone of the Nanoscience & technology. Nanoscience & technology is a broad and inter-disciplinary branch (or area) of R&D activities which has grown very fast universally in recent yrs. Nanoscale substances are classified in manner in which collection of atleast-one dimensional material that is less than about 100 nanometres. A nm is approximately 100,000 times lesser as compared to the thickness of a human hair by a billionth of a meter[8,9].

#### **1.3 Classification of Nanomaterials:**

As the Term tells, nanomaterials are the substances where dimension of the particular building blocks are smaller than 100nm, atleast in 1D. Hence on the underlay of dimension nanomaterials can be classified into 3 classes [10,11]:

• Materials with 1 dimension in nano-range: thin films.

• Materials with 2 dimensions in nano-range: nanowire, carbon nanotubes, quantum wells

• Materials with 3 dimensions in nano-range: nanoparticles, nanopowders, and quantum dots.

#### 1.3.1 Quantum Dot:

It is a SC in which excitons are bounded within all 3 axis of direction in space. Accordingly, like substances have intermediate electronic attributes among those of large size SC & those of separate molecules. Researchers studied quantity points inside transistor, solar cell, lasers for Light emitting diodes & Diode[12]. The size of the crystal will generally be smaller and there will be large band gap of energy. The energy odds among the higher VB and the valance CB is similarly greater therefore more energy is required to incite the dot and at same time, large amount of energy is liberated when the crystal comeback to its sit back position .with florescent dye usage, for example, it equates to larger frequencies of illumination after dot excitations as crystal dimension increases smaller result, in a shift of color from red to blue in emitted illumination[13].

#### **1.3.2 Quantum Wire:**

A nanowire is a nanostructure that has a nanometer-order diameter (9 to 10 metres). Alternately, nanowire may be known as structures which keep limited thickness (diameter) to tens of nm or smaller than of it and an unconstrained length. Quantum mechanical effects which govern term "quantum wires" are most vital at these dimensions. There are numerous various kinds of nanowires inclusive of metallic behaviour (e.g. ,SiO2, TiO2). In the next to about future the nanowires might be employed to connect small components to the enormously tiny ckts. These kind of component may be produced from chemical compound using nanotechnology.

#### **1.3.3 Carbon Nanotubes:**

CNT's are carbon allotropes which have cylindrical shaped nanostructure of carbons. Nanostructures have ratio of length-to - diameter up to 132,000,000: 1 were constructed, enough larger as compared to any other material[14]. All the cylindrical shaped carbon molecule has new characteristics which made them potentially useful in numerous usage in nanoscience and nanotechnology, electronics, optics, electrical, mechanical and other fields of material science. Also, they can have applications in body armor design. They show exceptional strength and special electrical properties, too.

#### **1.3.4 Quantum Well:**

It is a potential well which has only distinct values of energy. One quantization technology is used to contain particles that were originally free to travel in 3 or 2 dimensions, forced them to acquire a planer region[15]. The Impact of quantity containment arise when quantity well width is equal to carriers' De-Broglie wavelength (usually holes and electrons), retaining the energy levels known as "energy sub-bands," that is. such carrier may have only distinct(discrete) values of energy.

#### 1.3.5 Thin Films:

On the nanoscale, nanostructured surfaces have 1-D, i.e., only the width of surface of an object in range 0.1-100 nm. Many forms of deposition techniques were evolved and used in different industry, Provides a great driving impulse for potential growth &

advancement of deposit techniques. The methods of film development may generally be split into the two scopes: deposition of vapor phase & growth base on liquid. For example, evaporation, molecular beam epitaxy, sputtering, chemical vapour deposition & atomic layer deposition. Self-assembly monolayer (SAM's)[16] are examples of the latter.

#### **1.4 Importance and Application of Nanomaterials:**

Due to theirs exceptional electrical, mechanical, Optical, and magnetic attributes[17], Below are some examples of these:

- Since these are more ductile than coarse-grained ceramics at elevated temperatures.
- Metallic nano-sized powders were employed for the output of gas-tight substances, compact part & porous coating.
- For achievement of gas sensors (NOx, carbonmonoxide, carbondioxide, methane and aromatic hydrocarbons) by increased sensitive property and selective property, nano-structured metal oxide thin film receives growing attention. Metaloxide nanostructured (MnO2) use for rechargeable batteries, automotive or in the consumers usage batteries.
- Polymer dependent combination with a more (larger) inorganic particle contents resulting in a high value of dielectric constant.

#### **1.5 Silver Nanoparticles:**

AgNPs are the NPs which are in range 1 to 100 nm in size. Because their greater surface-to-volume ratio as compared to bulk silver atoms, Silver contains a huge percentage of SilverOxide. Different forms of nano-particles may be created or manufactured be dependent on attributes. Bulk Ag, when reduced to nano-level, shows remarkable property changes which make it more environmentally friendly and useful[18]. Nano Silver can be defined as a group of micro-sized(nano) Ag bits that are either covered or suspended in a medium. Compared to the macro-sized Silver, nano-silver has specific physical, optical, electrical & chemical properties. AgNPs which are most widely consumed are spherical in shape. Diamond, octogonal, and thin sheets are

common as well. Their incredibly broad surface area value condemns a huge count of the ligands to coordination. The attributes of Silver nanoparticles in lab and to understand the animal studies, potential impact, venomousness and expenses are applicable to human treatment and under investigation. Due to their unique physical and chemical attributes, silver nano-particles consumed various industries example: food, health and electronics etc. Silver nanoparticles include high electrical conductivity, optical, electrical, biological, thermal and mechanical and electronics attributes[19]. Because of these properties, they have been used in a number of usages for different work, like as an antibacterial(antimicrobial) agents, in manufacturing, health-concerned goods, for consumer goods items, for coating of medical instruments (device), for making optical sensors, uses in cosmetic industries, in pharmaceutical companies, in food industries, in automobile industries, in diagnose centres, orthopedics, target drug delivery, plays important role in treatment of cancer as an anticancer agent and eventually has increased cancer killing affects of anticancer medicines [20]. Silver nanoparticles keeps recently applications in numerous fields like textiles, in wound dressings role in preventing infection, and for the making medical instruments.

Silver nanoparticles have used for different purposes. Different techniques or approaches have been used for their synthesize to satisfy all the requirements of Ag nanoparticles. In general traditional method to synthesize nanoparticles is physical & chemical approach[21] are looking very costly & includes risk dangerous for us. Nanoparticles prepared biologically for Ag exhibit large yield, dissolubility as well as more stability[22]. Green synthesis approaches are convenient, have low risk, intense, non-toxic to environment, reliable method than other and biogenic synthesis method among various synthetic methods for Ag nanoparticles, which may output of exactlydefined size & structure (morphology) under improved circumtances for translational and different kind of research. Before determining toxicity or bio-compatibility[23], it is important to evaluate the characteristic properties of Ag nanomaterials like as dimension, shape, size division ,volume, surface area, form, stability, dissolubility and agglutination etc. To characterise the formed nanomaterials, numerous analytical techniques have employed such as inclusive of UV spectroscopy, X-ray diffractometry (XRD), Fourier transform infrared spectroscopy, X-ray photoelectronspectroscopy, Dynamiclightscattering, Scanning electronmicroscopy(SEM), TransmissionElectron microscopy (TEM), Atomic force microscopy (AFM)[24]. Ag nanoparticles' biological activity rely on the elements like dimension, surfacechemistry of particle, size division, shape of particle, morphology of particle, particle conformation, coating & capping, summation and disintegration rate of particles, reactivity of solution particle, ion released capacity, and cell type; A crucial factor in determining cytotoxicity is the kind of reduction agents employed to the formation of the Ag NPs[25].

Due to AgNPs usages in turmor treatment as an antiturmor agent, in diagnose & in sampling, Ag nanoparticles have recently shown great interest. Taking into account the literature, we concentrate on the formation, characterization, attributes and biological usages of Ag nanoparticles in a single platform, primarily on antimicrobial activity, anti-turmor agent, antifungal, antiviral, anti-inflammatory & anti-angiogenic attributes.

#### **1.6 Synthesis of Silver Nanoparticles:**

For example, nanoparticles are created naturally by plants, algae and volcanic activity, and can be natural or manmade. Too much progress has already been made in the synthesis, assembly, and manufacture of nanomaterials, and equally significant in the future applications of these materials in a wide range of technology. Many scientists reported various methods[41] To production of interest NPs and of varying dimension & structure (morphology). In general, Ag nanoparticles are synthesized dealing with three various methods including physical method, chemical method, and biological approach. For the physical method Ag nanoparticles are formed using means of the process of evaporation condensation employing a furnace tube onto atmospheric temperature [42]. The advantages of physical approaches are radiation used as reduction agent, speed & used not harmful chemicals. Yet there are also certain disadvantages such as small product and large energy utility, short of constant delivery and contamination by solvents. Chemical methods employ organic solvent or water to make Ag nanoparticles [41]. Control of particle size and shape and reproducibility in the methods for the synthesis of nanophase materials are prime and important factor. Nanomaterials are produced in two general ways. The first method is to start using a large size substance and after that use mechanical, chemical or different types of the energies to split it into smaller parts which is called the top down approach. An opposite approach is to synthesize the material through chemical reactions from atomic or molecular species, and is called a bottom-up approach. This approach typically has three important components, like as precursor to metal, reducing agents (plant extract), capping agents. Generally, reduction of Ag salt (silver ion) requires the nucleation in two stages first, and the subsequent development in another.

#### 1.6.1 Top-down Approach:

It is an approach refers to bifurcating (slicing) a large substance successively into extremely small particles to receive nano-sized particles.

It involves dismantling the large (bulk) material into nano-sized materials or substances. "Top-down" approach achieved by slicing, dividing approach that are accomplished with bulk or film machining, surface matching and lithographic molding. Large material machining uses photolithography which is used for etching procedure, whereas soft lithography is used in mold machining [44]. Top-down approach relates to:

- ➢ High energy milling
- Chemical mechanical milling
- Vapor phase condensation
- Electro-explosion
- ➢ Laser ablation
- Sputtering

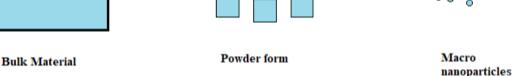


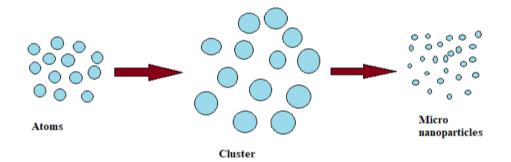
Figure 1.1: Top-down approach.

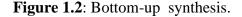
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#### **1.6.2. Bottom-up approach:**

This approach involves building up a small particle from the bottom side, atom by atom, molecule by molecule or cluster by cluster of a material to form nanoparticles. This approach of the formation of nanomaterials 1<sup>st</sup> builds nano-structured building blocks (NPs) & after that collects these nanoparticles into the end material, manipulating the building blocks via controlled chemical Rx to self-assemblies & making nano-structures like carbon nanotubes and quantum dots [44]. This approach involves:

- Solution combustion synthesis
- Sol-gel formation process
- Micro-emulsion process
- Reverse micelle approach
- Chemical precipitation method





#### **1.6.3 Chemical Solution Deposition (sol-gel) Method:**

The chemical solution deposition method, in materials science, it is a way for formation solid materials by using very tiny molecules. This process is employed to make metal oxides, such as silicon, titanium and silver oxides. Today this method for thinfilm coatings are commonly used for various usages like as optical and protective coatings, electro-optic and non-linear optical films.

This method involves chemical changes of a liquid solution into a gel form by using subsequently post-treatment & the changes into solidoxides.

This method involves creation of inorganic mechanism by forming a colloidal (sol) suspension and solution gelation to create a mechanism in a sustained liquid (gel) process. Precursors for the synthesis of these all colloids may commonly consist of a metal/ metalloid substance, surrounded with the different reactionary ligands[50].

Initially the material is expected to build a solvable oxide & in touch by water/ dilute acidcum also become a sol. Expulsion of liquid by the sol originates wax, and the transformation from sol / wax influences, material's particle dimension and structure. Gel calcinations yield the oxide... Sol-gel cycle refers to alcoxide-base precursor hydrolysis and condensation, likes tetraethyl orthosilicate, (Si(OEt)4) or TEOS.

This method chemistry reaction is depended on the hydrolysis and condensation of metallic alcoxides M(OR)z, and may be defined as given below:

 $MOR + H2O \rightarrow MOH + ROH$  (hydrolysis)

 $MOH + ROM \rightarrow M-O-M + ROH$  (condensation)

This forming process of nanomaterials is very famous among chemists, & is extensively used in the preparation of oxide materials[50].

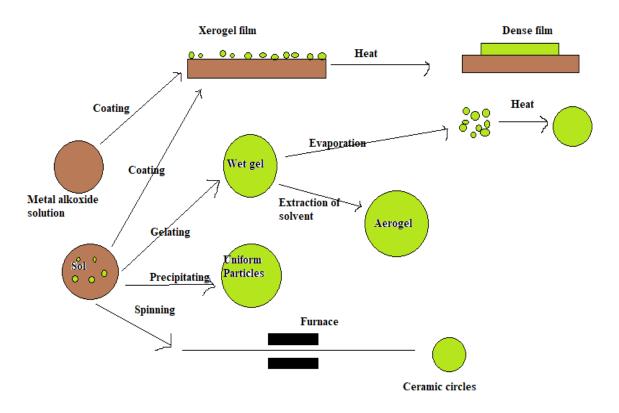


Figure 1.3: Sol-gel process.

This method cycle can be separated by separate steps.

- Conformation of various Irreplaceable alcoxide sol. or solved precursor metal.
- Gelation results conformation of the oxides or alcoholbridged mechanism N/W (the gel) through polycondensation Rx which consequences in a dramatic increment of the sol. viscosity.
- Aging of gel, continuing until the during the poly-condensation reactionsTransforms fully in the solid mass, along with the squeezing of gel mechanism & the removal of solvent by the pores of the gel. Ostwald maturation ( it is the mechanism whereby tiny particles are consumed by bulk particles at the time of the development process) & phase transitions happen coincidentaly. Gels aging procedure may outstrip 6 days & is very crucial for the prevent cracks in gels casted.
- Gel drying, if water & another variable fluids are separated by gel network. This method is difficult because of the fundamental transformation in gel framework. The process of drying ourselves was divided into four separate stages:
- Rate period=constant,
- Crucial point,
- Falling rate time (duration),
- Second falling rate Duration.

When the thermal evaporation isolates it, the outcome monolith is called xero-gel. If solvent is extracting beneath supercrucial circumstances (such as water), the produce yield is an aero-gel.

- 5. Loss of water, at the time which surface constrain groups of MOH are extracted and stabilized against rehydration by gel. It is usually accomplished by monolith calcination at temperature till 800<sup>0</sup>C.
- ♦ 6. Gel thickening & dissociation at very large temperatures (T>8000 C). The holes of gel network break & the rest organic species are transpire.

#### **1.6.4 Wet Chemistry Method:**

Wet chemical method was employed for the formation of AgNPs. The NPs for spherical Ag were prepared in pursuance of procedure explained by P.K. Sahoo [46]. Ag nanoparticles were prepared in this process by using NaBH4 as a reduction agent. In a beaker inclusive of (50mL of DI water) was added an aquatic sol. of trisodium citrate (0.5 mL, 6 mM). Following this, an aqueous AgNO3 solution (1 mL, 1 mM) was applied to the flask. Freshly formulated sodium borohydride solvent water solution (0.5mL, 10 mM) was easily applied, & suspension converted a light into yellow color light immediately. This suspension converted after 10s from light yellow into a dark yellow color or dark brown color subsequently another 20 seconds of reaction.

#### **1.6.5 Monosaccharide Reduction:**

It is a fast method for Ag nanoparticles prepared by a microwave house-hold. The process includes Ag nanoparticles were made for some time by an aqueous solution containing AgNO3 and D-glucose which are both microwaved. When microwaving reaches a temperature of up to 80 degrees Calcius, the temperature at the end. For this, it was not necessary to have capping agents and an oxygen free environment. Full Ag nanoparticles are produced when a tenfold molar superfluous of glucose exceeds silver nitrate, that is stable in non-appearance of stabilizing agents for several months. Ultraviolet-visible spectroscopy of colloidal mixture produced, demonstrated a large Plasmon Resonance band in wavelength among of 380-470nm by a maximum wavelength which is almost 450nm suggesting the formation of the metal Ag nanoparticle. Scanning tunnelling microscope & Transmission electron microscopy show that Ag nanoparticles were frequently egg-shaped in size, & scale ranges between the 5-50nm by the 25 nm population[52]. Further HRTEM analysis shows the significant (main) fringes due to interference with steady well-ordered spacing. This is the first Ag nanoparticles synthesis utilizing a household microwave. This process requires a green solution & also decreases the formation time into few minute which is ideally suited for an experiment in the laboratories of undergraduate teaching.

#### **1.6.6 Citrate Reduction Process:**

A simple method of chemical reduction had prepared uniform and well-dispersed Ag nanoparticles. The particles were synthesized as a surfactant by reducing AgNO3 by the absorbent and trisodium citrate[53]. The resulting Ag nanoparticles have been studied

through different characteristic techniques X-ray diffraction investigation, transmission electron microscopy, and scanning electron microscopy. By using these all characterization technique we can say so that size of Ag nanoparticles range between 35–80nm, by a mean size of 50nm. The effects on the morphology of the Ag nanoparticles of Trisodium Citrate & Ascorbic Acid, which behave like a reduction agent & surfactant respectively; Did they study ... This was observed that in the increment conc. of trisodiumcitrate led to a decrease in the size of Ag nanoparticles, although an increase in ascorbic acid concentration showed the reverse effect. Also, the quasi-spherical shape of as-synthesized Ag nanoparticles is more uniform with trisodium citrate increased. A slight alter in shape and size of the particles by quasi-sphere to polygonal was observed as concentrations of ascorbic acid increased or decreased.

#### **1.6.7 Polyol Process:**

## **1.6.7.1** Production of Silver nano-cubes and Nano-rods by using Polyol process:

Silver atoms mainly pack together in the cubic fcc structure [54]. The relative levels increasing at the surface in a specific plane can be managed to generate unique morphologies. Polyol method is used for reducing the metal salt in hot ethylene glycol when forming cubic particles and rods. This acts as a solvent for the rection as well as the reduction agent which reacts at 160 degrees Calcius with silver according to the reaction:

 $HOCH_2-CH_2OH \rightarrow CH_3CHO + H_2O$ 

 $2CH_3CHO + 2AgNO_3 \rightarrow 2CH_3CO-COCH_3 + 2Ag + HNO_3$ 

Poly (Vinyl Pyrolidone) is the structure guiding agent that interacts on the surface of metal crystal through the nitrogen and oxygen motifs within the heterocyclic ring to cap [100] planes, thus facilitating growth on the [111] planes as the polymer interacts weakly with that particular surface. This results in the rod structures around the center having a pentagonal symmetry (like a nanoparticle of zero dimensions). The cube morphologies can be developed by balancing the growth rate of [111] and [100] lattice planes using different concentrations of silver, the concentration of the capping agent and the growth time. If [100] grows 0.58 times faster than [111] face, a cube will be

formed. If it is up to 1.73 times faster than it will form tetrahedral and octahedral particles. Compared with the silver concentration, these concentrations are determined by the amount of poly present in the solution. When the Ag ions concentration is less than a certain threshold with the polymer concentration remaining constant can form nanorods. In order to preserve the proper reaction time and ratio, the salt Ag and the polymer must be added into the hot solvent at controlled rate.

Ag nanocubes process using polyol method:

- A heating mantle and an oil bath, able to reach magnetic stirring temperature of at least 160 degrees.
- Take a glass vessel with 5ml of anhydrous ethylene glycol. A needle is poked through to allow gas to get out while the reactants are delivered.
- Take 0.25 mol / L 3 ml Silver nitrate in a solution of ethylene glycol. The dissolution of 0.127 gram of AgNO3 into 3 ml of ethylene glycol will form this. Keep this solution in dark to avoid decrease.
- Now take 3ml of a 0.375 mol solution per litre. The dissolution of 0.123 gram of polymer in 3ml of ethylene glycol will form this.
- A centrifuge able to comfortably handle 50ml of falcon pipe. We need water here for the tubes to grow. We do need scale for centrifuging to align the samples.

Procedure to make nano cubes of Silver using method Polyol:

- Add 5ml of ethylene glycol to the magnetic stirrer bar and clamp it in the oil bath.
- Fill with the Ag solution one syringe with the polymer solution, and another syringe. Fit them both into the syringe pump and cover with the foil the Silver solution that covers the bottle.
- Engage both the bpumps and let the tubing solutions pass through until they're just about to get out of the needle. Now switch the pump off.
- Turn on the heating mantle and fix to a temperature of 160 degrees. Allow the ethylene glycol solution to balance for one hour.
- Set the syringe pump to produce a 0.375ml / min. Ag and polymer solution. Turn the pump on and let the solutions drip under stirring into the ethyl glycol. All the reactants are supplied within 8 minutes and the solution turns from

colorless to yellow and then red and eventually to a thick red green as the silver nanocubes form. Put on for an additional 45 minutes.

- Turn off the heating mantle now and remove the oil bath then allow the solution to cool down.
- Move all reactant contents to a falcon tube, and dilute the sample using water. Now centrifuge the sample at full velocity for 10 minutes. Remove the centrifuge tube 's top solution, and disperse water. To get rid of ethylene glycol, repeat the same centrifugal step twice or much more times. Now we are getting the Ag nanocubes dispersion solution.

#### 1.6.8 Green Chemistry Approach:

In organic strategy, concentrates, for example, plants and microorganisms can be utilized either as lessening or defensive operator for the manufacture of metal nanoparticles [43]. These concentrates are seen as a blend of different biomolecules having lessening operators can be found in the structure, for example, amino acids, proteins, catalysts and so on.

Natural technique utilizes microbial life forms, for example, microscopic organisms, parasites, yeast, and plants or plant removes increased extensive utilized as an option in contrast to concoction and physical strategies in the field of nanotechnology. One of the crucial procedure in nanoparticle combination includes bio-decrease. Both unicellular and multi-cell natural living beings can deliver inorganic materials either intra-or extra cell upto nanoscale measurements.

Plant-intervened amalgamation of nanoparticles have points of interest in light of their simple accessibility, green planning of nanoparticles by utilizing plant removes is most helpful examination subject in the field of nanotechnology. In the biogenic union plant extricates are utilized in the watery structure in the creation of respectable nanoparticles in light of the fact that the accessibility of lessening specialist is extremely high in the plant separate than the entire plant [55]. Besides plant-intervened amalgamation of nanoparticles is less complex and simpler to be led without need of a particular conditions concerning the physical and substance strategies are required. This method is increasingly ecological agreeable as the incorporated results of the procedure including waste items are come about because of plant extricates. Hence, this green amalgamation

strategy for the manufacture of nanoparticles is generally appropriate for huge scope creation with compelling cost venture, eco-accommodating, ok for individual as well.

Aside from strength and reproducibility, the pace of bio decrease of metal particles utilizing natural specialists is demonstrated to be a lot quicker and furthermore conceivable at typical pressue and temperature conditions. The bio-decrease union capability of the plant separate is than the microbial culure. Likewise, the waste items came about because of the microbial-based technique is progressively destructive to our condition. Subsequently plant extricate union have less or nearly pollution zero consequently does nor influence our condition. Along these lines the biosynthesis strategy utilizing plant remove is basic, powerful and practical method [56].

#### **1.7 Different Properties of Silver Nanoparticles:**

#### 1.7.1 Diameter, Surface Area and Volume:

Silver nanoparticles have wide variety of biomedicine utilities. In general, Ag NPs are smaller when compared with size of 100nm and comprises of 15-20,000 Ag atoms which keep different electrical, physical, optical, chemical and biological attributes in comparison to their large volume substances. Ag nanoparticles' melting, insentive and optical belongings are greatly affected with their dimension and morphology (shape).

#### **1.7.2 Shape and Crystallinity:**

Ag nanoparticles' physical and volatility attributes, together with superficies chemistry, dimensions, size administering, structure, particle geo-morphology, particle configuration, overlay/limit, accumulation, coating or capping, termination rate, dissolution rate, solution particle susceptibility, coherence, ion release efficiency, enclosure type etc. [26]. By applying biological reductant like as the cultivation of bouncy of different species of microorganism like Bacillus, Ag nanoparticles may be prepared in different forms like round, bar, octave, hexagon, triangular, floral.

The assertion that particles of miniature dimension could much toxic as compared to larger dimension particles since they have greater superficies area [26]. Diverse kinds of family of nanostructures have been used in life science or medical field, together with nanocubes, nano-plates, nanorods, spherical nanoparticles, flower-like etc. [27]. The

toxicity of Ag nanoparticles basically be dependent on presence of chemical or organic coatings on the superficies of nanoparticles.

Ag nanoparticle superficies charge can decide the toxicity result in cells. For illustration, positive facet charge of Ag nanoparticles contributes more suitably permitting them to continue for extended time in blood streamlet as contrast to the negatively-charged Ag Nanoparticles [27], that is the most important way for the management of antiturmor agent.

Operating biological reduction factors like culture supernatants of the different kind of Bacillus, Ag NPs can be prepared in different mixed forms like cylindrical, round, bar, octagon, hexagon, triangular, flower-type etc.

#### **1.7.3 Optical Properties:**

As Ag NPs are revealed to a different wavelength of illumination, the vibrating electromagnetic area of illumination involves a set of the liberated electrons' coherent oscillation, that induces a detachment of charge in view respect of the ionic network that creates a dipole vibrating through the light control electric field. Amplitude of oscillation exceeds at the maximum individual(distinct) frequency well known as SurfacePlasmonResonance(SPR). Controlling the particle dimension, morphology & refractive index close to the particle superficies could change the immersion and dispersive properties of Ag nano-particles. Tiny nano-particles imbibe most of the light and having peaks about 400 nanometers, whereas for large nano-particles there is enlarge scattering and peaks that extend and move towards wavelength side [27]. Optical attributes of Ag NPs may also change when particles accumulate & when the conduction electrons close each facet of the particles is delocalized.

#### **1.7.4 Antibacterial Effect:**

The Ag nanoparticles have used antimicrobial or antibacterial influence to monitored bacterial growth in various usage, including dentistry work, surgical usage, wound and burn treatment, and biomedical instruments [28]. As we know, compounds based on Ag ions and Ag is very toxic towards micro-organisms. Introduction to the silver NPs, Ag nano-particles into bacterial cells may bring about a high level of anatomical & morphological variations that cause cell death. Ag nanoparticle antibacterial effect is mainly because of continued liberation of free Ag ions from the nano-particles that acts like a vehicle for the Ag ions.

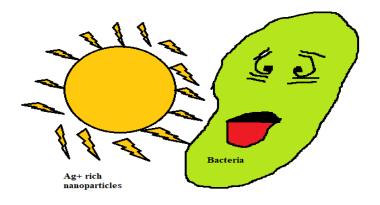


Figure 1.4: Antibacterial effect of Ag ions.

#### **1.7.5 Particle Stability:**

With their application the aggregation of nanoparticles is very difficult. Ag nanoparticles can be stabilized to charge. Zetapotential defines measure of particle stability factor for charging stabilized particle. Nanoparticles with zeta potential in excess of 20milivolt or less than -20milivolt keep sufficient electrostatic repulsion to remain stable in solution. Zeta potential is strongly liable in solution for certain molecules or contaminants [29]. Ag NPs are as well susceptible towards illumination and can be collected in the dark (especially UV light). Intensely acidic or basic solutions also enhance the disintegration rate of nano-particles to ionic shape on the container sides or re-deposit on existing nanoparticles resulting in a alter in the mean diameter and distribution of size[30]. With the help of UV- Visible spectroscopy, particle stability can be accurately determined due to the unique optical properties of Ag nanoparticles.

#### 1.7.6 Surface Chemistry of AgNPs & Functionalization:

With a large range of materials Ag nano-particles may be functionalised. Polymers like polyvinyl pyrroiodine(PVP) and tannic acid are widely employed as capping agents for material usages of Ag nanoparticles. In general, Ag NPs employed in life science or medical usage are coated by polyethylene glycol, bovineserum or various other proteins and peptides [31]. Particles may be operationalized by the molecules in order that superficies charge of the negatively polarity Ag nano-particles can be easily flipped to a positively charged area.

#### **1.7.7 Nanoparticle surface:**

The most common question that comes up is "what's on the surface of your nanoparticles? ". Response is that the molecules interact with facet of NPs, & that surface will bind molecules that build a two layer of charge which Inhibits agglomeration of NPs. Silver nanoparticles together with citrate, tannic, and polyvinyl pyrrolidone were capped. Citrate is feebly associated with the facet of the nanoparticles, frequently used because the feebly tighten capping agent can provide long lasting stability & is easily disploged by certain alternative molecules inclusively with thiols, polymers, anticorps & proteins[32].

Tannicacid like a multidentate capping agent, which may be substituted together with molecular thiols . Tannic acid is mainly used in usages where large concentrations of particles are requisite like a capping ligand-..

Polyvinylpyrollidone is a kind of substance or polymer which can bind tightly to the facet of the Ag nanoparticles. PVP can provide more stability than tannic acid or capping agents with citrate acid, but it's very difficult to displace.

Most nanoparticles have a dynamic surface, and are forcefully affected by the confined environment. Distinct state affects the particle in distinct ways. Excessive salt environment allows the twin layer to collapse easily and cause gathering or accumulation of nanoparticles. The stable particles would also be correlated with proteins and other biomolecules. For example, when adding nanoparticles to mixture used in vitro and in vivo investigations, it is better to use low-salt particle containing the medium's protein components, & then put on salt to conduct the mixture up to isotonic conditions. The protein tie to the facet of the particles, and the emulsion will remain steady. When nano-particles are mixed straight to an excessive salt buffer then the particles can gather before fixation of the protein can happen.

#### **1.8 Applications of Silver Nanoparticles:**

#### **1.8.1 In Biological Sensors:**

In recent years' peptide capped Ag nanoparticles have been mainly studied for colorimetric sensing. It concentrate on the character of interaction between peptide & Ag, and impact of the peptide on Ag nanoparticles creation. Efficiency of fluorescent

sensors based on Ag nanoparticles can be very high, and the detection limit can be overcome[33].

#### **1.8.2 Optical Probes:**

Ag nanoparticles are employed primarily as surface enhanced Raman scattering & metal enhanced fluorescence probes [37]. Ag nanoparticles have more advantage over other noble metal nanoparticles for the probe like excessive elimination coefficients, sharp elimination bands and large field enhancement.

#### **1.8.3** Antibacterial property of silver nanoparticles:

The **Antibacterial effects** of AgNPs have been used to control bacterial growth in a variety of applications, including dental work, surgery applications, wounds and burns treatment, and biomedical devices. Silver ions and Silver based compounds are highly toxic to microorganisms. Introduction of AgNP into bacterial cells can induce a high degree of structural and morphological changes, which can lead to cell death. Ag nanoparticles have an antibacterial effect caused by the continued liberation of free Ag ions by NPs[34].

#### **1.8.4 Catalytic properties:**

Ag nanoparticles were prepared to display redox catalytic attributes for biological factors like colorants, chemical agents like benzene, etc. Nanoparticle's chemical surrounding shows a major part in its catalytic attributes. It's also vital to be aware that complexed catalysis occurred by absorbing the reactant species into catalytic substratum [35]. As complicated factors, polymers, surfactants are account as the stabilizer to inhibit nanoparticles from coalescing, catalyst property is typically reduced because of decrease in absorption power. Commonly, Ag NPs are mainly practiced as the catalyst for chemical response(Rx) with titanium dioxide.

#### **1.8.5 Supported By Silica Spheres As Reduction of Dyes:**

Ag nano-particles were prepared on an inactive Silica spheres sustain [36]. The help actually plays no part in catalytic capacity and is used like a tool that is used in colloidal solution to prevent coalescence of the Ag nanoparticles. Thus Ag nanoparticles were synthesized and their ability to act as an electron relay for sodium borohydride reducing dyes. There will be no reaction in the middle of sodiumborohydride & specific dye such as methyleneblue without the catalyst of Ag nanoparticles.

#### **1.8.6 Selective Oxidation of Benzene:**

Ag nanoparticles are beneficial because of the higher number of active sites that can be easily assisted on aerogel [38]. The largest selective property of benzene to  $C_6H_5OH$ (phenol) was take noticed in the aerogel matrix at a low weight percentage of Ag (1 percent Ag). Due to higher monodispersity within the aerogel matrix of 1 per cent silver sample, this selectivity is supposed to result. Every weight percent sol. forms various size particles with a distinct size range breadth.

#### 1.8.7 Silver Alloy:

Nanoparticles made from gold-silver alloy have a synergistic effect on CO oxidation. Every impeccable metal nano-particle exhibits very low catalytic bonding for CO oxidation; side by side, the catalytic attributes have been largely increased. The Au behaves like a strong combining factor to O2 atom, & Ag acts like a powerful catalyst for oxidation [39]. For Au / Ag proportion of 3:1 with 10:1 synthesizes Silver alloys, nano-particles alloy shows entire modification if one per cent of CO is sustain in the air at surrounding temperature. The size of prepared nanoparticles has played no large part in catalytic efficiency.

#### **1.8.8 Chemotherapy:**

Ag NPs may go through coating approach which provide a constant functionalized facet that may be mixed to substrates. Substrates may be mixed through steady ester & ether bonds which are not immediately decreased by inherent metabolicenzymes. Under metabolic attack low toxicity nanoparticles will remain viable for required time to spread along the body systems [40]. If tumor is aimed for investigation, then UV illumination may be applied across that region. The light's EM energy affects the photo responsive connector to tear the drug & the substrate of nanoparticles. Now, the drug liberated in an undeviated active configuration that acts at cancer tumor unit cells [40].

# Chapter 2 Characterization techniques

In this part, different strategies have been utilized to describe the integrated silver nanoparticles. The portrayal of Ag nanoparticles tests arranged by neem, tulsi and mint, are required so as to comprehend the relationship between the properties and molecule size. The crystallite size, microstructure, cross-section boundaries and strain were investigated using a X-ray diffractometer. Retention spectra of all the 3 distinct examples are researched by utilizing bright Absorption spectra (UV-vis). The above portrayal methods can affirm the physical qualities of the readied Ag-nanoparticles These portrayal strategies are talked about in subtleties in the accompanying area.

- X-ray diffraction technique
- Ultraviolet-Visible Absorption spectroscopy

#### 2.1 X-ray Diffraction:

XRD is an instrumental strategy that is utilized to examine/distinguish tests XRD can give the examination researcher a quick apparatus for routine example ID. X-beam diffraction is a long-run request procedure which is touchy to the intermittent structure of a strong material X-beam diffracting method is significant method that is accustomed to recognizing the clear stage presents inside the example and to compute the auxiliary boundaries (like grain size, stage organization, strain state ,deformity structure, and favored directions). A lattice of crystal is supposed to be made up of systematic layer of planes containing atoms of identical separation afar [57]. As x-ray wavelength is close to the inter-atomic spacing, Laue scientist in 1912 recommended that whichever crystal may acts like grating for x-ray diffraction. Thus if x-ray beam is permitted to drop on a crystal then a immense no. of different intensity images are created. whether the wave which are diffracted are in a similar phase as that of incident then they strengthen each other so that a pattern of bright shiny is formed on the photographic sheet, put down in their way.[1 2 3]

When X-ray radiations are moves through material, the radiations interact by electrons of atoms, which results in scattering of light radiations. And when these atoms are arranged in the planes (that is the substance is crystalline in nature) and distance in the middle of these atoms are of equal extent(value) as that wavelength of X-beams used, alternating progressive & reductive interferences may be produced [58] diffracted X-rays were emitted at particular angles, depended on interlayer spacing in the middle of

atoms present inside the crystal planes. Each and every atom belongs to numerous pairs of crystal planes. Every pair of plane has a particular inter-planar separation & may gave arise to a specific angle of diffracted X-rays. William Henry Bragg & William Lawrence Bragg in 1913, played out a scientific count to discover entomb nuclear good ways from the X-beam diffraction, the connection is called as Bragg's law[59].

#### $n\lambda = 2d \sin\theta$

Here,

- $\lambda =$  Incident x-ray wavelength
- n = Diffraction order
- d = Inter-planar distance
- $\theta$  (theta) = Bragg's angle

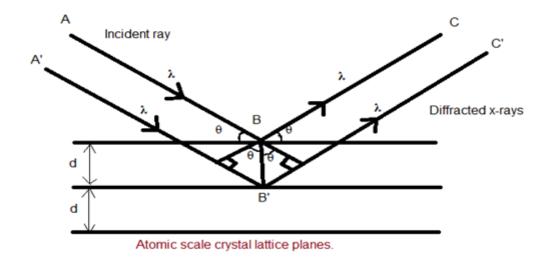


Figure 2.1: Bragg's Law.

- ➤ X-rays diffracted from atoms inside crystalline plane follows the reflection laws.
- The 2-rays are reflected from the continuous planes, may be inside phase when the additional path covered by 2<sup>nd</sup>-ray is integral count of wavelengths. When the wavelength illuminated is familiar & the angle could be calculated, there the inter-planar spacing can be easily calculated. A set of inter-planer spacing will

be get through an individual compound, representing pair of planes which may go through from atom & it may be utilized for compare with the other pair of inter-planer spacing's get by the general results.

Condition for the reflection (that is Bragg's condition) can be fulfilled for some arrangement of planes whose between planer dispersing will be more prominent than half of the frequency of X-beams utilized (when  $d < \lambda/2$  at that point  $\sin \theta > 1$ ,which is absurd )[1 2 3].

This trial comprises of examining two X-beam films uncovered in a powdered diffraction camera. In the powder X-beam procedure, the x-beam falls on an aggregate mass of little precious stones having direction every which way, and the diffracted bar will frame h,k,l shaping a cone. Bends of these cones are caught by a film which is encircled by the specimen [60]. X-beams pillar goes through a collimator and strikes the example. A film strip which is put along the round and hollow camera divider would be uncovered by dispersed radiations and show, after adequate improvement and time, the diffraction design. The un-dispersed radiation will leave the camera through the leave port.

The Scherrer eqn., it is a equation which related with the dimension of nanoparticles, or crystallites, in a strong to widening of top in diffraction design. scherrer eqn. can be utilized for the assurance of the size of particle of the crystals in the powder structure.

It is broadening strip at middle of the maxim intensity, Subsequently removing instrumental broadening strip, measured in radians. It may sometimes have written as Delta  $\Delta = (2\theta)$ ,  $\theta = Bragg$ 's angle.

There are 3 basic parts of X-rays diffractometers: an Ex: - holder , x-beam tube, xbeam indicator . Number of X-rays are shaped in cathode flash tube by heating fiber to liberate number of electrons, these electrons are quickening towards an object with apply adequate measure of voltage, & the target material is bombarded by electrons. At the time where the electron has a sufficient measure of energy to break inner shell of electrons then x-ray range is delivered. This range comprises of different parts, k $\alpha$ 1 and k $\alpha$ 2 [61]. The k $\alpha$ 1 has insignificantly lower frequency yet force is twice as k $\alpha$ 2 has. These particular frequencies are relying upon the trademark target material. Normally  $k\alpha 1$  and  $k\alpha 2$  are adequately near the frequency with the final aim that usual load of the two is employed. Cu is normally utilized objective for individual-valuable stone diffraction with Cu k $\alpha$  illumination (light) equivalent to 1.54 angstroms [62] .The X-ray beams is coordinated & collimated on the Ex. At point when the example and locator are pivoted then The power of X-ray beams were recorded when the X-ray episode on the example fulfill the Bragg's condition then productive impedance design happens and a force top shaped.[1 2 3]Detector is utilized to record and to process these X-ray signals and it convert the sign to a tally rate that is yield of a gadget like printer or PC screen etc[63].



Fig 2.2: Bruker's X-ray Diffractometer [Model: D8 ADVANCED]

configuration of X-rays beam diffractometer is with the aim that the example pivots inside the direction towards particular point (collimated) X-ray beams pillar at a pillar of  $\theta$ , although X-ray beam indication device (detector) is linked with a branch for gathering the diffracted X-rays & thus turns at a point 2 $\theta$ . The apparatus or device which is utilized for maintaining the edge and used to pivot the example is called as Goniometer. [1 2 3] For the average powder XRD patterns, information is gathered onto point 2 $\theta$  ranges between 5degree to70degree [64].

# **Application of Powdered XRD Technique:**

- It is robust technique for the detection of an unknowable material in form of amorphous, polycrystalline and single crystalline.
- Recognition of fine crushed materials like clay that are rigorous to measure optically.
- Determine unit cell parameters.

# 2.2 UV Visible Spectroscopy:

It clarifies why natural mixes retain UV or noticeable light. It additionally clarifies why the ingested light frequency is diverse for various compound.

# **WORKING:**

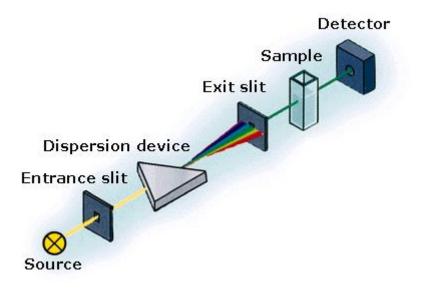


Figure 2.3: Diagrammatic representation of Spectrometer.

By using UV- visible absorption spectroscopy qualitative measurements can be done easily [65]. But it is ineffectual for qualitative measurements. Fundamental principle of UV Spectroscopy is Beer Lamberts Law. The concentration of analyte in mixture is detected by evaluating the absorbance of EMR at distinct wavelengths. UV visible characterization cures in measuring absorption & transmittance of various samples like coatings, windows, filters and pigment. The illuminating object used commonly a deuterium discharge flash light for detecting ultra violet field & tungston halogen flash light is used for identify visible field (region). Depending on type of instrument the discharging material is employed to isolate wavelength of light may be an absorption filter, prism monochromator / diffraction grating. If measurements is done, and the source should be captured to avoid any stray of light dropping upon the recorder. This radiation affects the quantification value & therefore measurements are introduced in the readings. The recorders are employed to record the intensity of light [66].



Figure 2.4: UV- VIS Spectrometer [Model: Carry UV 300].

A single light beam and a sample holder is present with the facility for selecting a fixed wavelength for a range of EMR. The measurements of absorption for different wavelengths performed here simultaneously. The sample is in general in liquid state. This sample is placed in a test tube, a cuvette which is kept inside the sample holder of instrument. The measurement using a photometer requires a standard reference sample.

SPECTROMETER

The concentration for the standard reference must be known. The reading for the standard are noted down as displayed by the photometer. After that the unknown sample is placed in test tube holder and readings for the same are noted down.

#### **Principle :**

It clarifies why natural mixes assimilate UV or obvious light. It additionally clarifies why the consumed light frequency is diverse for various compound[67].

At the point when the occurrence light goes through the example then the vitality given by the illumination is utilized for energize an  $e^-$  by ground (bonding/non-bonding) orbital into energized void (a - antibonding) orbital.

In each and every chance, an e<sup>-</sup> bounces from a complete orbital state to an unfilled enemy of holding orbital express .Each excitation takes vitality from the episode light. Clearly a major hop needs more vitality than that of little jump[68].

Each frequency of illumination has a certain vitality related by light .when that certain measure is equivalent to the vitality required for any of these hops then that frequency will be ingested .

As we know,  $\underline{\mathbf{E}} = \mathbf{h}\mathbf{v}$ 

Here : E =Incident ray energy

h = Planck's constant

 $\mathbf{v} = \text{Light freq.}$ 

For a high energy jump we need that the e<sup>-</sup> absorbs light of lighter frequency .Greater the frequency, greater will be the energy.

But in UltraViolet-visible absorption spectroscopy we are using  $\lambda$  of light rather than the freq.

As, <u>λ=c/v</u>

Here,

 $\lambda = Light's$  wavelength

$$c = Light's speed$$

v = freq.

For Higher frequency, lower will be the wavelength. Hence for bigger energy jump, light of high frequency will soak up light with a lesser wavelength.

In Absorption spectroscopy, spectrometer works in between range of 200nm to 800nm approx. (ie. From near ultra-violet to near IR region) .Very Finite number of the e<sup>-</sup> jumps are may occur in that range[69].

The significant jumps are :-

- By pie-bonding orbital towards pie\* anti-bonding orbital.
- By non-bonded n orbital towards pie\* anti-bonding orbital.
- By non-bonded n orbital towards  $\sigma^*$  anti-bonded orbital.

Therefore we can say that in sequence to soak light in the region of 200-800nm, molecule should have either  $\pi$  bond or atoms with non-bonded orbitals.

Absorbance is a Scale of the quantity of light absorbed higher the amount of absorbance, greater specific wavelength is being absorbed.

If wavelength is towards 200nm then the compound is in ultra-violet range. therfore colourless for example  $\lambda = 217$ nm for Buta-1,3-Diene. Therefore there is no non-bonding electrons. Peak is observed because e- jumps from  $\pi$  to  $\pi^*$  anti-bonding orbitals.

## **Applications of Ultraviolet-Visible Absorption Spectroscopy:**

- With the help of UV- vis. absorption spectroscopy qualitative measurements can be done easily. But it is ineffective for the qualitative measurements.
- Basic principle of UV Spectroscopy is Beer Lambert Law. The conc. of analyte in a mixture detected by estimating the absorbance of EMR onto distinct wavelengths.

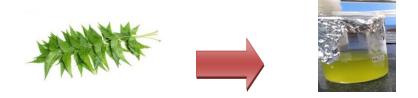
• UV visible characterization supports in measuring absorption & transmittance of distinct samples like coatings, windows, filters and pigments.

# Chapter 3 Results and Conclusion

## 3.1 Aqueous Neem leaf extract mediated AgNPs synthesis:

### **3.1.1 Preparation of Neem Leaf extract:**

- Leaves of Azadirachta Indica (NEEM) were first rinsed with tap water and then distill water to remove all the dust and unwanted visible particles.
- About 1 gm of finely incised wet Neem leaves were boiled in 50 ml distill water at 50°C for about 15 min.
- The supernatant was filtered using Whatman filter paper No.1 to remove the particulate matter. A pale yellow clear solution is obtained and stored at 4-8°C.



### **3.1.2 Steps involved in nanoparticle synthesis by plant extract:**

A 1mM Sol. of AgNO3 is prepared by adding 0.0085gram of silver nitrate in 25ml of DW(distilled water)[56]. Presently, 0.5ml aqueous extract of neem was blended in 5ml of 1mM AgNO3 solution. The color of the solution changes from light-yellow color to brown, demonstrating the creation of silver NPs. The green synthesis plant extract method is better than the chemical method because it is pollutant-free, cheaper than other methods, eco-friendly, and simplest. The result shows that neem, tulsi and mint extract did a significant job in the stabilization and reduction of Ag ion into Ag-NPs.

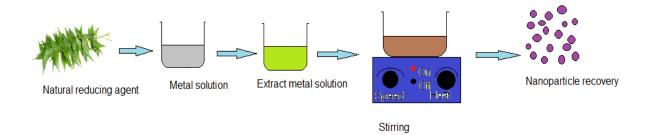


Figure 3.1: Steps involved in the silver nanoparticle synthesis.

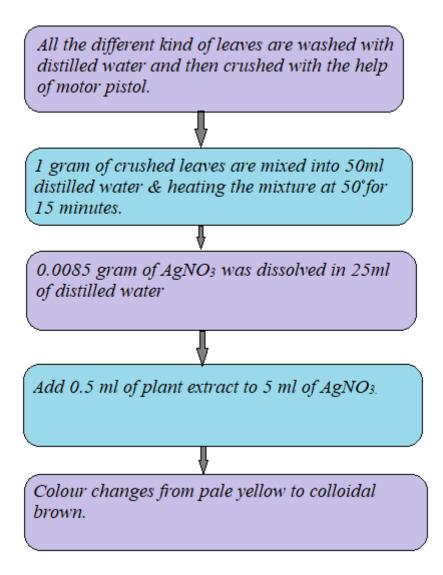


Figure 3.2: Flow chart for preparation of silver nanoparticles.

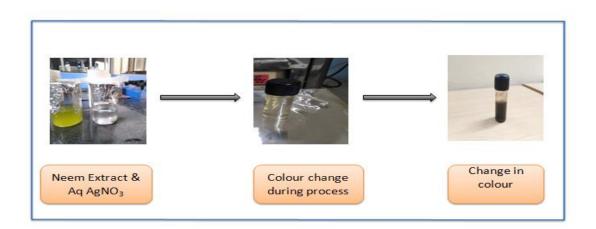
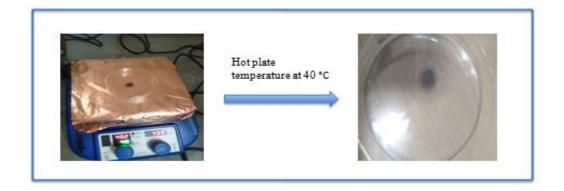


Figure 3.3: Change in color after mixing of neem extract and aq. silver salt.



**Figure 3.4**: Thin film of silver nanoparticles prepared by neem sample using Drop caste method.

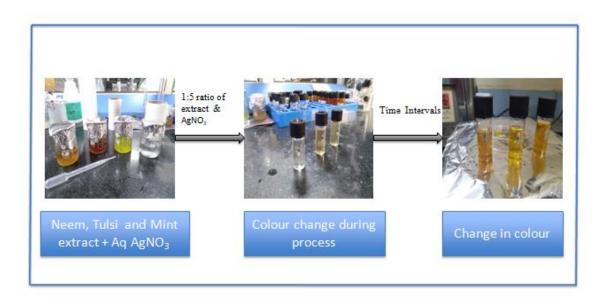


Figure 3.5: Diagrammatic Representation of Fabrication Process of Neem, Tulsi and Mint Capped AgNPs.

## Advantages

- It produces material with high purity.
- The method is a cost-effective method.
- It provides the facility to modify the particle surface state and overall homogeneity.
- It is also used in chemical analysis and in radiochemistry.

#### **3.2 XRD Analysis:**

X-ray diffraction of neem capped silver nanoparticles is used to analyze the crystalline size in the range of 20 to 80 degrees. All the XRD diffraction pinnacles can be filed to a converse spinel structure of Ag nanoparticles, which is in acceptable concurrence with the writing esteem JCPDS card number 19-0629. The intense peak of neem-coated AgNPs were observed at 38.08<sup>o</sup> and 44.24<sup>o</sup> relative to (1 1 1) and (2 0 0) F.C.C silver, which shown the crystalline behavior of silver nanoparticles.

All the structural parameters can be calculated from the peak represented in figure 3.6.

The mean size of the AgNPs formed was calculate by the FWHM of (1 1 1) diffraction reflection adopting Scherrer equation.

 $D = k\lambda/\beta cos\theta$ 

Where, D=particle size,  $\lambda$ =wavelength of x-rays (1.54Å),  $\theta$  = Bragg's angle corresponding to a peak,  $\beta$ = full-width half maxima measured (radians), k=Scherrer constant (0.94), and the particle size of Ag nanoparticle was found around 13.58 nm as observed from the main peak shown in Table 1.

 $d = n\lambda/2\sin\theta$ 

By using the above formula, the inter-planer spacing between the atomic layers are found to be 0.24 nm shown in Table 2(as observed from the main peak 1 at  $2\theta = 38.08$ )

and the lattice parameter 'a' is determined from the equation:

 $a = d\sqrt{h^2 + k^2 + l^2}$ 

So for the main peak 1 the value of the lattice parameter comes equal to 4.15 Å, which is given in table number 2.

The atomic radius of the FCC structure is calculated from:

$$r = a / 2\sqrt{2}$$

So the value of atomic radius of FCC structure is calculated from the above relation and it is equal to 0.146 nm given in table 2.

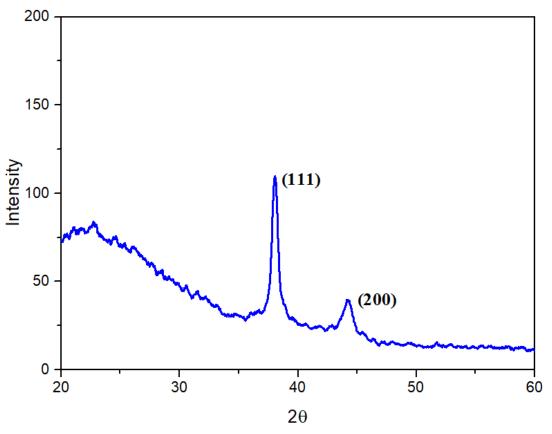


Figure. 3.6: XRD pattern of AgNPs made using neem extract.

All the structural parameters can be calculated from the peak represented in figure 3.6. A slight variation in the diffraction peak positions with the concentration represents the strain in the lattice of crystal. The XRD pattern shows in figure 3.6 exhibits a face-centered cubic structure. Both the peaks exactly match with the hexagonal symmetry of Ag nanoparticles having a face-centered cubic (FCC) structure. The diffraction pattern of prepared sample Ag nanoparticle from neem leaves shows broad peaks centered around  $2\theta \sim 38.08$  and, 44.24 (Fig 3.6) corresponding to the (111) and (200) planes and are summarized in Table 1.

Sample	Peak No.	2θ (degree)	θ (degree)	h k l (planes)
AgNPs	1	38.08	19.04	(1 1 1)
	2	44.24	22.12	(200)

Sample	Ag
FWHM (radian)	0.64675
D (nm), Crystalline size	13.58
-	
a (Å), lattice parameter	4.15
2	
r (Å), atomic radius	1.46
0	
d (Å), inter-planer spacing	2.4
a <sup>3</sup> (Å <sup>3</sup> ), Volume	71.47

**Table 2:** Various parameters of AgNPs obtained using Neem leaves.

Overall, from the calculation, it has been observed that for Ag nanoparticles(prepared by neem leaves) exhibit two peaks at angle  $2\theta = 38.08^{\circ}$ , and  $44.24^{\circ}$ . For the main peak at  $2\theta=38.08$  the miller indices is (111) and the corresponding value of FWHM is 0.64675. From this data, we calculate different parameters like particle size, lattice parameter, atomic radius, inter-planer spacing, etc. and the corresponding value of these are 13.58nm, 4.15Å, 1.46Å and 2.4Å, respectively.

#### **3.3 Optical Analysis:**

UV-Visible spectroscopy is one of the reliable & useful characterization techniques to monitor the stability & formation of Ag-nanoparticles. UV-vis spectroscopy is simple, straightforward, quick, delicate, requires a short time period for estimations. In Agnanoparticles the valance band and conduction band lie near one another (little bandhole vitality), in which free electrons can without much of a stretch moves. These free electrons are answerable for surface plasmon reverberation assimilation band. This retention band happens on the grounds that the aggregate swaying of free  $e^-$  of Ag nanoparticles are inside reverberation by influx of illumination (light). For the Ag nanoparticles assimilation top relies upon the dielectric medium, molecule size and concoction encompassing. The Ag-nanoparticles synthesized from each extract solution were analyzed by using a UV-visible spectrum. It is useful to determine the characteristic peaks spectrum of Ag nanoparticle wavelength prepared from neem, tulsi and mint leaves. Normally the characteristic peak of Ag-nanoparticles are found in the wavelength range between 400-600nm. The absorption peaks centered around 438nm, 421nm, and 425nm for the Ag nanoparticles made from neem, tulsi and mint leaves, respectively.

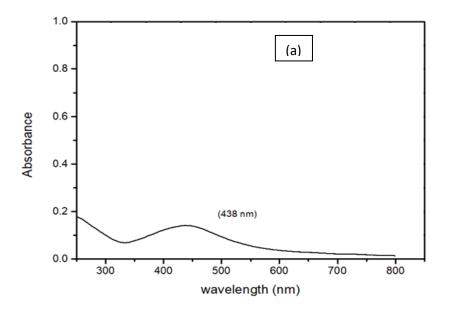


Figure. 3.7 Absorption Spectra of AgNP using neem extract.

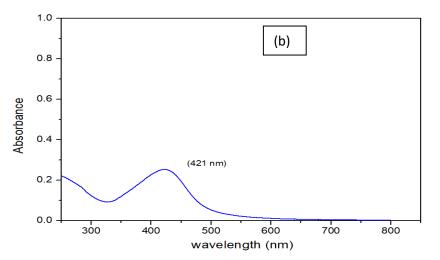


Figure. 3.8 Absorption Spectra of AgNP using tulsi extract.

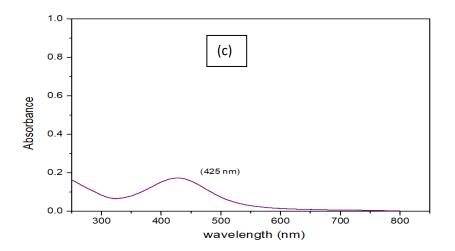
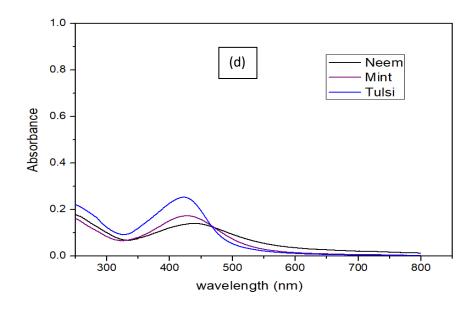


Figure. 3.9 Absorption Spectra of AgNP using mint extract.



**Figure 3.10**: Absorption spectra of silver nanoparticles prepared using neem, tulsi and mint extract are shown in (a),(b) and (c). The overlapped absorption spectra of all the three samples (d)

The absorption peak for Ag nanoparticles synthesized from neem, tulsi and mint were observed at wavelength 438nm, 421nm and 425nm, respectively. These absorption peaks are shown in figure 3.7 for neem(a), tulsi(b) and mint(c) & a comparative graph for these three is shown in (d).

Sample	Neem	Tulsi	Mint
Concentration (mol/ litre)	0.001	0.001	0.001
Wavelength,	438	421	425
λ (nm)			
Frequency,	0.685×10 <sup>15</sup>	0.713×10 <sup>15</sup>	0.706×10 <sup>15</sup>
$\mathbf{v} = \mathbf{c}/\lambda \; (\mathrm{Hz})$			
Energy,	0.4538×10 <sup>-18</sup>	0.4722×10 <sup>-18</sup>	0.4677×10 <sup>-18</sup>
E=hv (J)			
Absorbance, A	0.141	0.253	0.173
Molar absorptivity,	141	253	173
ε = A/cl (litre Mol <sup>-1</sup> cm <sup>-1</sup> )			

**Table 3:** Absorbance result of AgNP.

The characteristic peak of Ag-nanoparticles are found in the wavelength range between 400-600nm. The absorption peaks are centered around 438nm, 421nm, and 425nm for the Ag nanoparticles made from neem, tulsi and mint leaves, respectively (Table 3). And corresponding to these peaks intensity is maximum. For a high energy jump we need that the e<sup>-</sup> absorbs light of lighter frequency. The energy corresponding to neem, tulsi and mint is  $0.4538 \times 10^{-18}$ ,  $0.4722 \times 10^{-18}$  and  $0.4677 \times 10^{-18}$ J, respectively. As higher energy jump out of the three samples neem, tulsi and mint, is for tulsi, therefore electron absorbs lighter frequency of  $0.713 \times 10^{15}$ Hz as given in Table 3.Greater the frequency, greater will be the energy. For Higher frequency, lower will be the wavelength that is 421nm for tulsi given in Table 3. Hence for bigger energy jump, light of high frequency will soak up light with a lesser wavelength. Absorbance is a Scale of the quantity of light absorbed higher the amount of absorbance; greater specific wavelength is being absorbed. Therefore the absorbance value is maximum i.e, 0.253 for tulsi sample as its wavelength is also maximum out of the three samples. Also absorbance is given by the relation  $A = \epsilon lc$ , where  $\epsilon$  is the molar extinction coefficient, 1 is the path length and c is concentration of the specimen. So by finding the value of absorbance for all the samples from the graph we can easily calculate the concentration of the specimen. The value of absorbance is 0.141, 0.253 and 0.173 respectively for neem, tulsi and mint and the value of concentration for all the samples is 0.001mol/lt.

#### **3.4 Conclusion:**

We have successfully synthesized the silver nanoparticles mediated by using aqueous plant extract (neem, tulsi and mint leaves). XRD peaks shows crystalline nature of silver nanoparticles. This is the most common, economical and simple method. The neem, tulsi and mint extracts were specially prepared using wet leaves of the respective plant. This freshly prepared leaf extract using wet leaves were added to 1mM AgNO<sub>3</sub> solution in the separate 3 beakers for all the kind of extracts. AgNPs prepared from neem extract have particle size 13.58 nm calculated by using Scherrer's equation. The absorption peaks are 438 nm, 421 nm, and 425 nm shows good surface plasmon resonance behavior of the AgNPs prepared from neem, tulsi and mint leaves, respectively for the same concentration. The absorption maximum is at lower wavelength region when prepared from tulsi as compared to other two plants and is highest. The method is cheaper, non-toxic and less harmful for environment. The synthesized silver nanoparticles could be used in various applications such as for target drug delivery for cancer treatment, antibacterial effect, anti-fungicides, as an antioxidant, bio-imaging and biological sensors, etc.

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