

Synthesis and characterization of silver nanoparticles

(Project – Report)

Submitted in partial fulfillment of the requirements for the award of

Bachelor of science degree in Biotechnology

By

S.JESSYFIN (38770007)



DEPARTMENT OF BIOTECHNOLOGY

SCHOOL OF BIO AND CHEMICAL ENGINEERING

SATHYABAMA INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

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JEPPIAAR NAGAR, RAJIV GANDHI SALAI, CHENNAI - 600 119

April - 2021



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

Accredited with "A" grade by NAAC

Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai –
600119

www.sathyabama.ac.in



DEPARTMENT OF BIOTECHNOLOGY

BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of S.JESSYFIN(38070003) who carried out the project entitled "**SYNTHESIS AND CHARACTERISATION OF SILVER NANOPARTICLES**" under our supervision from October 2020 to March 2021.

INTERNAL GUIDE
Dr.RENUGADEVI.K ,M.tech,ph.D

Head of the Department
Dr. V Ramesh Kumar

Submitted for Viva voce Examination held on .

Internal Examiner

External Examiner

DECLARATION

S. JESSYFIN (38770007) hereby declare that the Project Report entitled **“SYNTHESIS AND CHARACTERISATION OF SILVER NANOPARTICLES”** done by me under the guidance of **Dr. RENUGADEVI** at **Sathyabama institute of Science and Technology, Chennai** is submitted in partial fulfillment of the requirements for the award of Bachelor of Science degree in Biotechnology.

DATE:

PLACE:

SIGNATURE OF THE CANDIDATE

ACKNOWLEDGEMENT

I am pleased to acknowledge my sincere thanks to Board of Management of SATHYABAMA for their kind encouragement in doing this project and for completing it successfully. I am grateful to them.

I convey my thanks to Dr.V.Ramesh kumar, Head of the Department, Dept. of Biotechnology for providing me necessary support and details at the right time during the progressive reviews.

I would like to express my sincere and deep sense of gratitude to my Project Guide Dr.K.RENUGADEVI for her valuable guidance, suggestions and constant encouragement paved way for the successful completion of my project work.

I wish to express my thanks to all Teaching and Non-teaching staff members of the Department of Biotechnology who were helpful in many ways for the completion of the project.

ABSTRACT

Silver Nano particles (AgNPs) have emerged as an important tool for diverse applications in medical fields. The functionalization of biologically active molecules on the surface of AgNPs have been used for the detection, diagnosis and treatment of diseases. Silver Nano particles were prepared by PEG (Poly ethyl glycol) in the absence of other chemicals. Silver nano particles treated with PEG is the another choice for various application. PEG act as a reducing and capping agents. The synthesized NPs were characterized using UV-Visible spectroscopy; FT-IR; DLS. This study aims at synthesis and characterisation of silver nanoparticle and pegylated siver nanoparticle using chemical reduction method.

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LIST OF ABBREVIATIONS AND SYMBOLS:

Computed tomography-	CT
Dynamic Light Scattering-	DLS
Ferrous carbonate-	FeCO ₃
Fourier Transform Infrared Spectroscopy-	FT-IR
Hydrochloride-	HCL
Hydrodynamic diameter-	dH
Localised Surface Plasmon Absorption-	LSPR
Magnetic resonance imaging-	MRI
Millivolt-	Mv
Nanometer-	Nm
Polyethylene glycol silver nanoparticles-	PEG-AgNPs
Polyvinyl Pyrrolidone -	PVP
Positron emission tomography-	PET
Potassium bromide pellet-	KBr pellet
Potassium dichromate -	K ₂ Cr ₂ O ₇
Revolutions per minute-	Rpm
Silver cation -	Ag ⁺
Silver nanoparticles-	AgNPs
Sodium Borohydride.-	NaBH ₄
Sodium dodecyl sulphate -	SDS
Sodium hydroxide-	NaOH
Super-critical carbon dioxide -	SCCO ₂
Surface - enhanced Raman imaging-	SERS
The polydispersity index-	PdI
Magnetic resonance imaging -	MRI
Inducible Nitric Oxide Synthase-	iNOS

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CHAPTER 1: INTRODUCTION

Nanotechnology is becoming an innovative area of increasing research and industrial interest in 1980's. Nanotechnology can be define as the unique properties of atomic and molecular assemblages which are used for drug delivery, image contrast agents , and for diagnostic purpose .The advantage of nanotechnology is to relates with the biological system which has the ability to control the size of the particle and devices. Nano - scales are smaller than human cells (10,000 - 20,000)in diameter .Nano - particles are smaller than 20nm which can traits through blood vessels walls[Gabriel A *et .al.,*] (2004).

The research in nanotechnology breaks through in areas such as materials and manufacturing, nano - electronics, medicine and information technology. It is widely felt that nanotechnology will be net Industrial revolution .When the dimension of a material is reduced from a large size, the properties remains same the small changes occur. A quantum dots has all three dimension in nano range. The term quantum is associated with these three types of nano structure, because of change in properties arise from the quantum - mechanical nature of physics in the domain of the ultra small. [Frederick *et.al.,*](2005) .

The biotechnology has an endless opportunities for molecular diagnosis and therapy. The nano carriers can be designed in the way to facilitate them to act as imaging probe using variety of techniques such as ultrasound (US), X-ray, computed tomography (CT), positron emission tomography (PET), magnetic resonance imaging(MRI), surface - enhanced Raman imaging (SERS). Nano particles such as magnetic nano particles (iron oxide), silver nano-particles, gold nano-particles, silver nano-particles, nano shells and nano cages have been modified to the use of diagnostic and therapeutic agents [Bharat Bhushan *et.al.,*](2007)

1.1 TYPES OF NANOPARTICLES:

There are different types of nano particles such as gold, silver, magnetic, silica,titanium, and chitosan which has wide application in bio medical, catalysis and environmental remediation.

SILVER

Silver nano particles (AgNPs) is the most effective because of its good antimicrobial effect against bacteria, virus, and other eukaryotic micro - organisms. They are most widely used nano - materials among all, thereby it is used as antimicrobial agents in textile industry, water treatment, etc., the silver nano-particles have been successfully synthesized in the plants such as *Azadirachta indica*; *Capsium annuum* and *Carica papaya*.

1.2 SILVER NANOPARTICLES:

Silver has been in use since time immemorial in the form of metallic silver, silver nitrate, silver sulfadiazine for the treatment of burns, wounds and several bacterial infections. Silver is used to make water potable. Silver nitrate was used in its solid form and was known by different terms like, “ Lunar caustic “ in English, “ Lapins infernal,” in Latin and “Pierre infernal “ in French. In the 1940s after penicillin was introduced the use of silver for the treatment of bacterial infection minimized [Mahendral Raj *et.al.*,] (2009)

Silver is widely known as a catalyst for the oxidation of methanol to formaldehyde and ethylene to ethylene oxide. In the United States, more than 4×10^6 tons of silver were consumed in 2000. Chemical reduction is the most frequently applied method for the preparation of silver nanoparticles (AgNPs) as stable, colloidal dispersion in water or organic solvents. Commonly used reductant are borohydride, citrate, ascorbate and elemental hydrogen. The reduction of silver ions (Ag^+) in aqueous solution generally yield colloidal silver with particle diameter of several nano meter [Virender K. Sharma *et.al.*,](2009)

The antibacterial effect of Ag salts have been noticed since antiquity and Ag is currently used to control bacterial growth in a variety of applications include dental work, catheters and burn wounds. With the emergence and increase of microorganisms resistance to multiple antibiotics, and the continuing emphasis on health - care costs, many researchers have tried to develop new, effective

antimicrobial reagents free of resistance and cost. Such problems and needs have led to the resurgence in the use of Ag - based antiseptics that may be linked to

broad-spectrum activity and far lower propensity to induced microbial resistance than antibiotics. [Jun Sung Kim *et.al.*,](2007).

he antimicrobial activities of silver - containing materials are often studied in terms of their Ag⁺ content. Ag⁺ has been reported to interact with cytoplasmic components and nucleic acids, to inhibit respiratory chain enzymes and to interfere with membrane permeability. The bio activity of nano - sized metal particles and their uses as molecular probes are research areas of growing interest. Owing to their large surface area and high reactivity compared with the bulk solid, nano sized metal particles exhibit remarkable physical, chemical, and biological properties. In particular, the optical properties of silver nano particles which vary according to their size and shape, as well as their chemical environment, have been intensively studied.[Chun - Nam Lok . *et.al.*,](2007).

Several salts of silver and their derivatives are commercially employed as antimicrobial agents. Thus, nanoparticles of silver have aptly been investigated for their antibacterial property. Nano particles of silver have thus been studied as a medium for antibiotic delivery and to synthesize composites for use as disinfecting filters and coating materials. The bacterial property of these nanoparticles depend on their stability in the growth medium, since this imparts greater retention time for bacterium - nanoparticles interaction [Siddhartha Shrivastava *et.al.*,](2007). The toxicity of AgNPs has also been shown by a number of *in vitro* studies. Toxicological investigations of NPs imply that, e.g. size, shape, chemical composition, surface charge , solubility, their ability to bind and affect biological sites as well as their metabolism and excretion influence the toxicity of NPs. Although the high toxicity of silver ions is known, measurement of the amount of silver ions within the investigated AgNPs suspensions has been rarely done and thus knowledge about the degree to which silver ions contribute to the toxicity to the toxicity of AgNPs and how much toxicity is related to the AgNPs themselves. Besides its antimicrobial effect, AgNPs are known to induce toxicity in many different species and chronic exposure to silver is known to cause argyria and / or argyrosis in humans. [Sista Kameswara Srikar *et.al.*,](2016).

1.3 SYNTHESIS OF SILVER NANOPARTICLES

CHEMICAL SYNTHESIS:

Currently, many methods have been reported for the synthesis of Ag-NPs by using chemical, physical, photochemical and biological routes. Each method has advantage and disadvantage with common problems being costs, scalability, particle size and size distribution. Among the existing methods, the chemical methods have been mostly used for production of Ag- NPs. Chemical methods provide an easy way to synthesize Ag-NPs in solution. The chemical synthesis process of the Ag-NPs in solution usually employs the following three main components (i) metal precursors, (ii) reducing agents and (iii) stabilizing / capping agents. The formation of colloidal solutions from the reduction of silver salts involves two stages of nucleation and subsequent growth. It is also revealed that the size and the shape of synthesized Ag-NPs are strongly dependent on these stages.

CHAPTER 2 : REVIEW OF LITRATURE

Nanotechnology is the science that deals with the matter at the scale of 1 billionth of a meter (i.e. $10^{-9}\text{m} = 1\text{nm}$), and is also the study of manipulating matter at the atomic and molecular scale .It plays a major role in development of innovative methods to produce new products, to substitute existing products equipment and to reformulate new materials and chemicals with improved performance resulting in less consumption of energy and materials and reduced harm to the environment. [Xi -Feng Zhang *et.al.*,] (2016)

The NNI (National Nanotechnology Initiative) defines nanotechnology as the research and development at the atomic, molecular, or macro molecular level in the sub -100nm. It contributes to the technologies in the field of health care and their impact on diagnosis, prevention and treatment. Nano biotechnology leads to the multifunctional devices such as biological and chemical analysis with better sensitivity and specificity. Nano biotechnology also involves in drug delivery system, imaging, bio -sensor, pathogens, electronics and in - vitro diagnostics [Scoot E.Mc Neil *et.al*] (2005).

2.1 NANOPARTICLES:

The term “nano” is derived from the Greek word “nanos” which means small.It is used as a prefix for one billionth part (10^{-9}). The major goals in nano particles is to control the size , surface properties and surface characteristics of nano particles can be easily achieve by both active and passive drug targeting . Nano particles are prepared by variety of materials such as polysaccharides, proteins, and synthetic polymers. [Michele Greque de Morials *et.al*] (2014).

Nano particles are proved to be promising multi-functional platform because they can be used for many imaging and therapeutics function. The synthesis of silver occurs by reduction of soluble salts with the reduction agents such as citrate or sodiumhydroxide. Physical synthesis are typically carried out in the gas phase, such particles are not surface-conventionalized, in water but are highly agglomerate. [Jae Yong Song Hyeon-Kyeong *et.al.*,](2009). Nano - particle is commonly used for

molecular assembly and composed of inorganic or polymeric materials. It is also can be in the form of nano - vesicle and the shape is often spherical but cylindrical, plate like shapes. [Svitlana Chernousova *et al.*,](2013). The transition from micro particles to nano particles leads to number of changes in physical properties that increase the ratio of surface area to volume and the particle size in quantum effect predominate. Carbon black is the nano - particulate material has been produced in quantity of decades. Nano particles are made of variety of materials which are split into metal oxides ceramics, such as zinc, aluminium, titanium and silicate nano -particles in the form of nano scale flakes of clay. It can be incorporated in a polymer during polymerization. Pure nano- particle metals at lower temperature can be merge into solids, without melting. Ceramics nano -particles can be used to create thin layers, or amorphous [O.V. Salata *et.al.*,](2004). Nano-particles has the ability to accumulate in the cell without P- glycoprotein, which is one of the mediator of multi-drug resistance. Nano- particles are used as used as drug delivery system to kill cancer cells. Nano- particles are applied as drug system are sub micron - sized particles (3-200) nm devices or system that include variety of materials including polymers, lipids, viral nanoparticles [Paul Holister *et.al.*,](2003).

2.2 PROPERTIES OF NANOPARTICLES:

The properties of nanoparticles are generally categorized into 3 namely:

2.2.1 OPTICAL PROPERTIES OF SILVER NANO PARTICLES:

The nanoparticles depends on their size, shape, environment, chemical composition and other parameters. Surface plasma resonance have an intimate relationship with the shape of each nanoparticles. The main optical features depends on geometry, size and the optical resonance of spherical, spheroidal, cubic and other geometrical shapes . In optical response structural techniques such as Transmission Electron Microscopy (TEM) shows the image of small piece of sample that gives the information about local properties and characterization of nanoparticles. On the other hand, optical are no - destructive and they can be used to perform in situ and real time measurements. Optical measurements were performed on solution of nano particles suspended techniques in deionized water . The optical properties of individual particles are determined by electro dynamic interaction between them. The

resulting optic spectrum depends on inter particle separation and the dielectric environment.

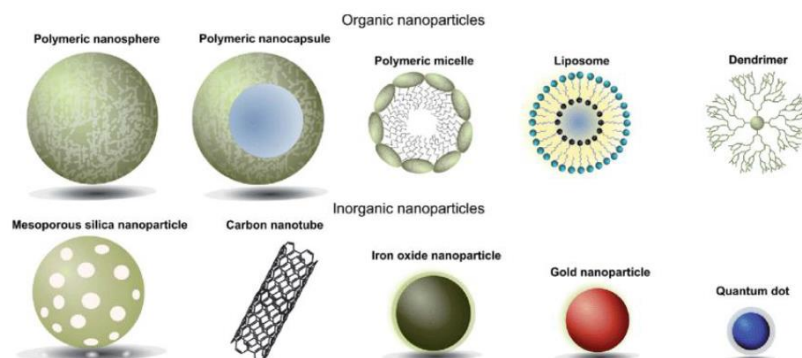


Fig 2.2.1 : Properties of Nano particles

2.3 METHODS FOR SYNTHESIS OF NANO PARTICLES:

2.3.1 CHEMICAL METHOD:

Chemical method is the most common method. It controls the growth of metal nano particles to obtain nano articles of small size with a spherical shape and narrow distribution in diameter. Silver nano particles can be synthesized by chemical reduction at low cost with high yield. The chemical synthesis process of AgNPs has the following components namely: (1) reducing agents; (2) stabilizing /capping agents. The chemical synthesis process of AgNPs used as reducing agents and stabilizing or capping agents and this is because of the sample size and their distribution [A.I.Gonzalez and Cecilia Noguez *et.a.l.*,](2007). Chemical reduction method is the common method for the synthesis of AgNPs using organic and inorganic reducing agents. The synthesized AgNPs appears as coloured solution, due to the surface of metals that contains free of charged electron and the positively charged nuclei. In general , the reduction of AgNO₃ using different reducing agents such as PEG (Poly ethyl glycol), Tri sodium citrate , Sodium borohydride (NaBH₄) are applied for reduction of silver ions (Ag⁺) in the aqueous or non aqueous solution [Jun Natsuki *et.al.*,](2015). The PEG act as reducing agents and it is very sensitive. The reducing agents of Ag⁺ to Ag⁰ - was results with the increasing in the length of polymer chain of PEG.

2.3.2.SURFACE PLASMON:

Nobel metal nano particles displays unique optical properties that differentiate them from their bulk counterparts. The most fascinating finding, regarding this peculiar optical performance, is that they often exhibit strong extinction bands in the visible spectrum, and therefore bright and gaudy colour, that are not present in the spectrum of the bulk metals. Moreover, metal nano particles scatter light with high efficiency and , unlike molecular dyes, their extinction spectra are really a combination of both absorption and scattering. The first theoretical approach for modelling the optical properties of nano particles proposed by Mie, According to Mie theory, the resonance condition is achieved when the real part of the dielectric function of the metal equals the dielectric function of the surrounding medium (Mie ,1908). Therefore , the LSPR(*Localized Surface Plasma Resonance*) is tunable and its frequency can be modified through change in the nano particles are sensitive to their local environment.[Carlos Caro .*et.al.*,](2007)

2.3.3. SILVER NANO PARTICLES -SURFACE CHEMISTRY:

When nano particles are , in solution, molecules associate with the nano particles surface to establish a double layer of charge that stabilizes the particles and prevents aggregation. Aldrich Materials Science offers several silver nano- particles suspended in a dilute aqueous citrate buffer, which weakly associates with the nano particles surface. Silver nano particles are extraordinarily efficient at absorbing and scattering light and, unlike many dyes and pigments, have a color that depend upon the size Silver nano particles have already been extensively studied for their unique optical and sensing properties. Solutions of spherical silver nano particles are known to have a strong yellow color and an extinction UV-Vis spectrum featuring a sharp and the shape of the particles.[Elham Abbasi.. *et.al.*,](2014)

2.4. SENSING PROPERTIES :

peak at 400nm. The colour of silver nanoparticles solution is due to the so –called localised surface plasmon absorption (LSPR) which arise influence of known electric field.

2.4.1 Fourier transform infrared spectroscopy (FT-IR):

The Fourier transform infrared (FTIR) spectroscopy measurements were carried out to identify the bio molecules for the synthesis of silver nano particles. It is based on the measurement of the absorption of electromagnetic radiation with wavelength within the mid - infrared region (4000 - 400 cm^{-1}) .If a molecular absorbs IR radiation, the dipole from the coupled oscillation of the conduction electron in the metallic nano particles induced by the incident light electric field. The properties of nano particles the most often used in the sensing application is based on the change in dielectric constant of the surrounding medium by changing the solvent quality or by complex formation at the surface of the nano particles. Silver nano particles present therefore great interest for sensing applications in both gas and liquid phase.[Stephan T.Dubas *et.al.*,](2008)

2.5.CHARACTERIZATION OF NANO PARTICLES

Nano particles are generally characterized by their size, morphology, and surface charge using advanced microscopic techniques such as Scanning electron microscopy (SEM), Transmission electron microscopy (TEM); Dynamic light scattering (DLS). The average particle diameter, size distribution and charge affect the physical stability and then vivo distribution of nano particles.

2.5.1 UV Visible spectroscopy:

Ultraviolet spectroscopy is a technique used to quantify the light that is absorbed and scattered by a sample (a quantity known as the extinction, which is defined as the sum of absorbed and scattered light). The sample is placed between a light source and a photo detector and the intensity of a beam of light is measured after and before passing through the sample. Nano -particles have optical properties that are sensitive to size, shape, concentration, agglomeration and the refractive index near to the nano particles surface, which make UV Spectroscopy a valuable tool for identifying, characterization and studying of these materials. Nano particles with the specific wavelength of light and the unique optical properties of these materials is the foundation for the field of plasmonics.

2.5.2 Dynamic Light Scattering (DLS):

2.5.2.1. Particle size analyzer:

In order to find out the particles size distribution of the nano particles along with the poly dispersity was determined using the particles size analyzer. Then the experiment was carried out in computer controlled particle size analyzer to find out the particle size distribution. Particle size was based on the measuring of the time dependent fluctuation of scattering of laser light by the nano particles undergoing Brownian motion. Particle size distribution and morphology are the most important parameters of characterization of nano particles. Morphology and size are measured by electron microscopy. The major application of nano particles in drug release and drug targeting. It has been found that particle size affect the drug release. Smaller particles offer larger surface area.

2.5.2.2.Zeta potential measurement:

Zeta potential was determined by means of zeta analyzer. It describes the electrical potential in the double layer of ions surrounding of a particle surface and the absorbed ions in the diffusion layer. The measurement of the zeta potential mainly depend on the direction and velocity of particles under the moment is modified and the molecule becomes IR active. A recorded spectrum gives the position of bands related to the strength and nature of bond and functional groups, providing the molecular structure and interactions. These techniques helped to probe adsorbents electro chemically and detect volatile reaction products. Dry powder of the extract and Ag nano particles solution wee centrifuged at 5000 rpm for 15 min and resulting suspension was re -dispersed in sterile distilled water. The purified pellets were dried and analyzed by Thermo Nicolet Avator 300 instrument in the diffuse reflectance mode at a resolution of 4cm⁻¹ in KBr pellets.

2.6 SILVER NANOPARTICLES:

Silver has been used for thousand of years as a precious metal by humans in different applications as jewellery, tools, coins, photographic materials or explosives. One of the most significant use of silver is an disinfectant with hygienic for medical purpose. Since the ancient times, silver vessels were used for the storage of wines and water because it was believed that this metal preserved their conditions. Also Hippocrates described the use of silver powder for its application in wound healing and in the treatment of ulcers. In the 17th and 18th centuries, silver nitrate was used for ulcer treatment and its antimicrobial activity was established in the 19th century.

Silver salts and silver compounds have been used in different biomedical fields, especially in burn treatment. The optical properties of metal nanoparticles of group 11 are based on the collective oscillation of free conduction electrons resulting from the interaction with the electromagnetic radiation. The biomedical properties of silver nano particles also represent a relevant field of research. Several interesting applications related to this property have been developed, mainly focused on the detection of chemical species of organic, inorganic or biological origin. The antimicrobial properties of silver nanoparticles based on the studies of antibacterial, anti-fungal, and wound healing potential properties have also been reported.

In this sense most of the chemical methods for the synthesis of Ag nano particles are based on reduction reaction of silver (I) metal salts.[Jorge Garcia Barrasa *et.al.*](2010).

2.7 SILVER NANOPARTICLE SYNTHESIS:

2.7.1 CHEMICAL METHOD:

Chemical method are usually to synthesize silver nano particle and chemical reduction is the most frequent applied method for the preparation of stable, colloidal dispersion in water or organic solvents.

Chemical reduction:

Typical reducing agents include polyol NaBH_4 , sodium citrate and N-N-dimethyl formamide. Doubtless, in order to prevent aggregation of AgNPs, it needs stabilize with capping agents such as Sodium dodecyl sulphate (SDS) , polyvinyl pyrrolidone (pvp), tri-sodium citrate . Some of the chemical reaction can be carried out in the room temperature. Thermal method such as reduction of Ag^+ by dextrose or hydrazine as a reducing agents and the well known. Tollens reduction with reduction agents of m - hydroxy benzaldehyde are from chemical reduction method. Silver nano particles have been synthesized by the polyol process with the assistance of super-critical carbon dioxide (SCCO_2) , with silver nitrate used at the base material , polyvinyl pyrrolidone (PVP)used as a stabilizer and the ethylene glycol as the reducing agents and solvent.

CHAPTER 3: AIM AND OBJECTIVES:

- The synthesis and characterization of AgNPS and PEG AgNPs by citrate method or by Sodium borohydride method.
- Characterization of AgNPs and PEG AgNPs by UV -Vis Spectrophotometer ,FT-IR, DLS.

CHAPTER 4 : MATERIALS AND METHODOLOGY

4.1 MATERIALS:

4.1.1 Equipments used

1. Conical flask
2. Test tubes
3. Weighing balance
4. Shaker incubator
5. Millipore filter
6. Centrifuge
7. Eppendorf
8. Petri plates
9. Measuring cylinder
10. UV-spectrophotometer

4.1.2 Chemicals used:

Silver nitrate, tri sodium citrate and polyethylene glycol (MW:6000) were purchased from Sigma Aldrich (USA). 0.1 M NaOH solution was used to control the pH of the nanoparticles at 7.6. K₂Cr₂O₇ and HCL were brought from Merck and used for cleaning the glass ware.

4.2.METHODOLOGY:

4.2.1.CHEMICAL PREPARATION:

4.2.1.1.Synthesis of citrate capped silver nanoparticles:

To synthesis citrate capped silver nanoparticles, 1mM of silver nitrate was dissolved in 25 ml of distilled water, to this solution 5ml of 1% tri sodium citrate was added drop by drop by placing it on the stirrer and allow it to boil for few minutes. Few minutes later, the solution turned to pale yellow colour indicating the presence of silver nanoparticles. Then it was removed from the heating device and allowed it to cool at the room temperature..(Bijoy Sankar Boruah *et.al.*,](2017)

4.2.1.2.Synthesis of PEGylated silver nano particles:

To synthesis PEGylated silver nanoparticles, 50 mg of PEG (Poly ethylene glycol) was dissolved in 25 ml of distilled water and 1mM of silver nitrate was added to the solution and heated till boiling on a magnetic stirrer for about 20 min. Few minutes later, the solution turned to yellow colour indicates the presence of silver nanoparticles. Afterwards, it was cooled to room temperature and stored at 40 C before use. (Bijoy Sankar Boruah *et.al*);(2017)

4.2.2. CHARACTERIZATION OF SILVER NANO PARTICLES:

4.2.2.1.UV-Vis spectroscopy :

UV-Vis spectroscopy is an important technique used to obtain information on the formation and stabilization of aqueous nanoparticles. It is one of the most widely used techniques for structural characterization of silver nanoparticles. It is used for measuring absorption spectra of sample and it has intense peak of 416nm.

4.2.2.2.Dynamic Light Scattering (DLS):

Dynamic Light Scattering (DLS), scattering of light intensity of AgNPs, PEG-AgNPs were measured to study the average particle size. The synthesized AgNPs and PEG-AgNPs were given for DLS analysis. The average particle size and zeta potential of prepared AgNPs and PEG-AgNPs were studied in a computer controlled particle size analyser (HORIBASZ-100for windows [Z type] Ver2.20).

4.2.2.3.FT-IR Spectroscopy analysis:

The synthesized PEGylated AgNPs were centrifuged at 10,000 rpm for 10 minutes, then collected pellet were dried and get powder form. This powder was used for PEG-AgNPs synthesis. FT-IR was performed for AgNPs, PEG - AgNPs using (SHIMADZU, JAPAN). The powder form prepared as KBr pellet. The prepared pellet were used for FT-IR spectra of samples were recorded for the wave number 4000cm^{-1} to 400cm^{-1} .

CHAPTR 5: RESULT AND DISCUSSION

5.1 SYNTHESIS OF SILVER NANOPARTICLES:

1% of tri-sodium citrate was added to the solution containing AgNPs in distilled water. During this process the slow color changes from white to yellow color. Fig 5.1 (a) . When 1% of tri-sodium citrate was added to the solution which contains 50mg of PEG AgNPs in distilled water. During this process the slow change from white to yellow color. Fig 5.2 (b).



Fig 5.1 (a) Synthesis of silver nanoparticles.



Fig 5.2 (b) Synthesis of PEGylated silver nano particles.

5.2 CHARACTERIZATION OF SILVER NANOPARTICLES:

5.2.1. UV -VIS SPECTRA of PEGylated Silver nano particles:

The synthesized silver nano particles were characterized prior to the study of anti-tumour efficacy of the silver nano particles. A plasmon band at 416 nm was observed in the absorption spectrum of the silver colloids synthesized by reduction method using PEG (Fig 5.2.1), indicating the incidence of Ag nano particles.

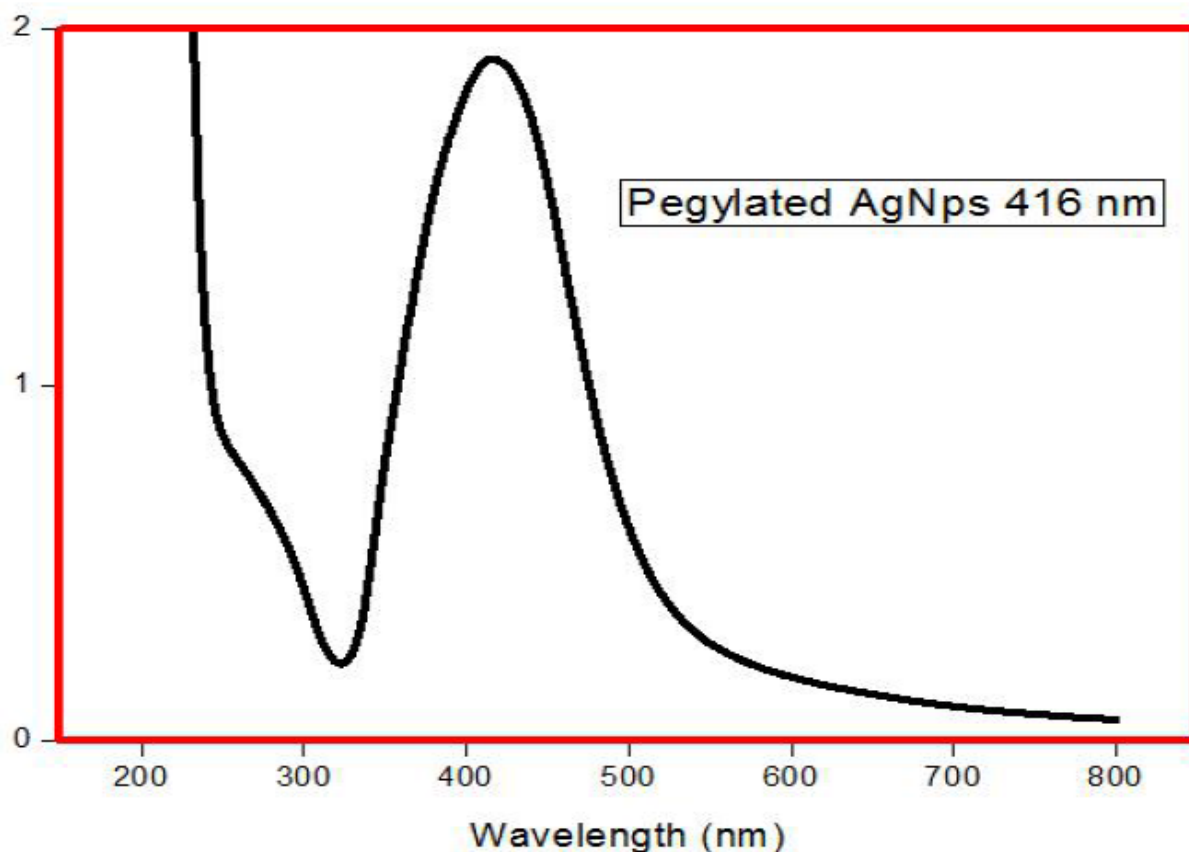


Fig.5.2.1. UV -Vis spectra of PEG functionalized AgNPs

5.2.2 Dynamic Light Scattering :

5.2.2.1. Particle Size distribution :

The colloidal properties of the synthesized nano particles were estimated from the intensity distribution of dynamic light scattering (DLS) (Fig 5.2.2.1 and 5.2.2.2). The major scattering peak at 7.3 nm of AgNPS and 12.0 of PEG-AgNPS indicates the hydrodynamic diameter (dH) of the particles. The polydispersity index (Pdl), a dimensionless parameter, was calculated from the photon correlation spectroscopic analysis to arrive at the particle size distribution. The intensity distribution graph indicates that the silver nano particles are reasonably well monodispersed with the polydispersity index of AgNPS is 1.438 and PEG-AgNPS is 1.576.

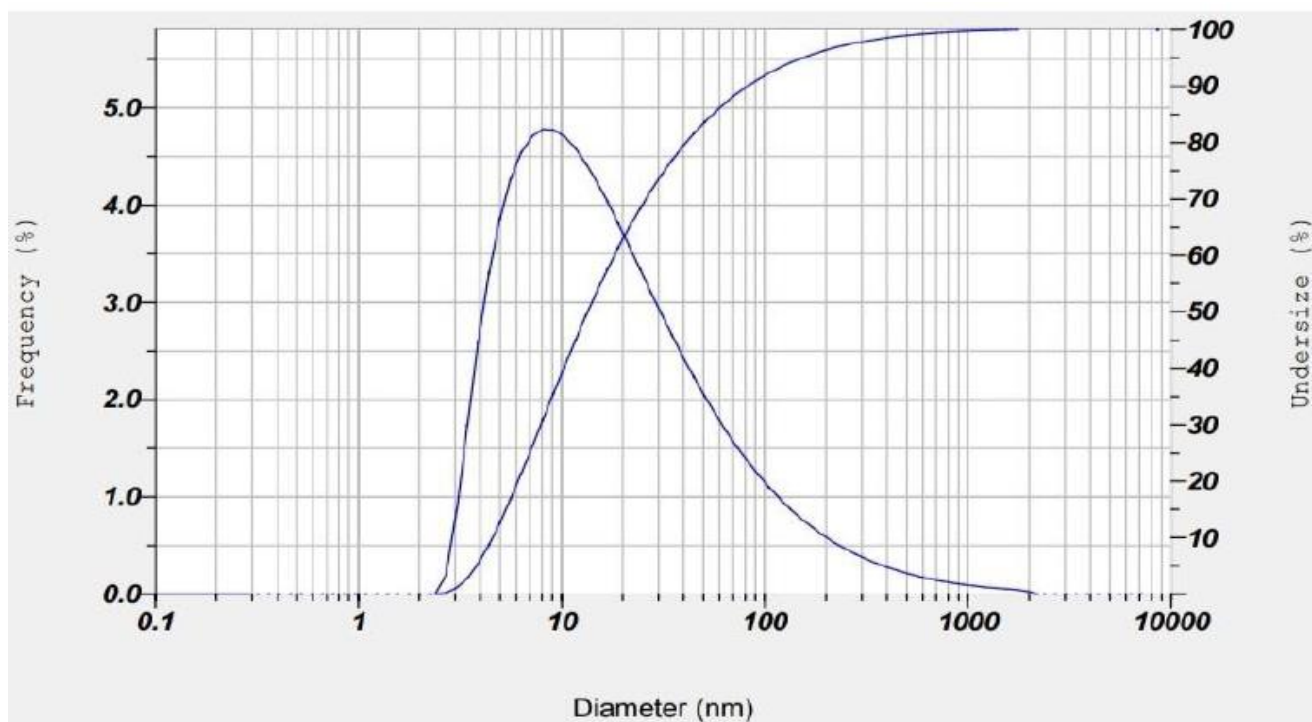


Fig 5.2.2.1: Particle size analysis of AgNPS Z- Average : 7.3 nm and PI: 1.438

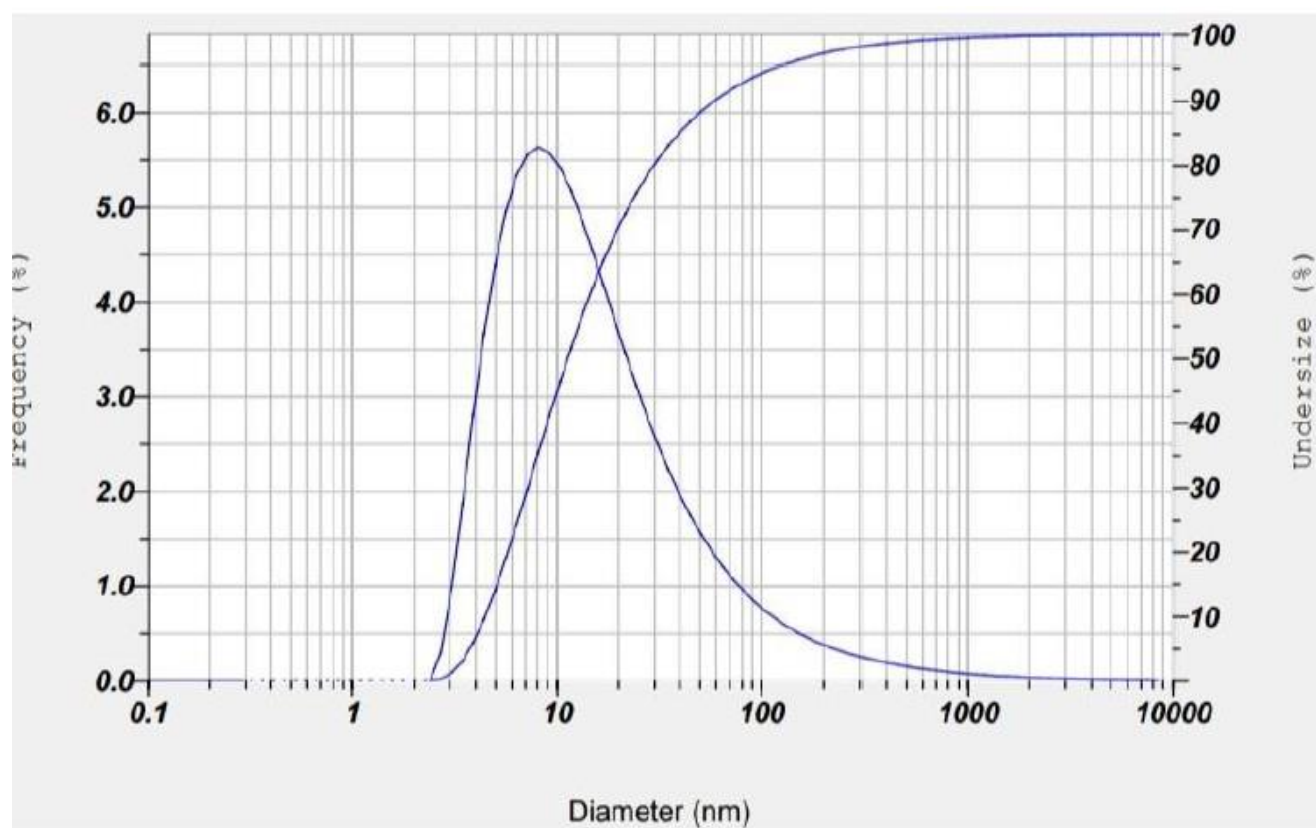


Fig 5.2.2.2 : Particle analysis of PEG-AgNPS Z - Average 12.0nm and PI : 1.576

5.2.3.Zeta potential:

Zeta potential measurements was utilized to study the surface charge of silver nano particle. Generally, it was used to study the physical stability, cellular uptake, bio-distribution and interaction of bio molecules. The zeta potential of AgNPs, PEG AgNPs were analyzed in this study, those are -28.4mv and -26.4 mv respectively. Thus the result shows particles possess negative charges on the surface.

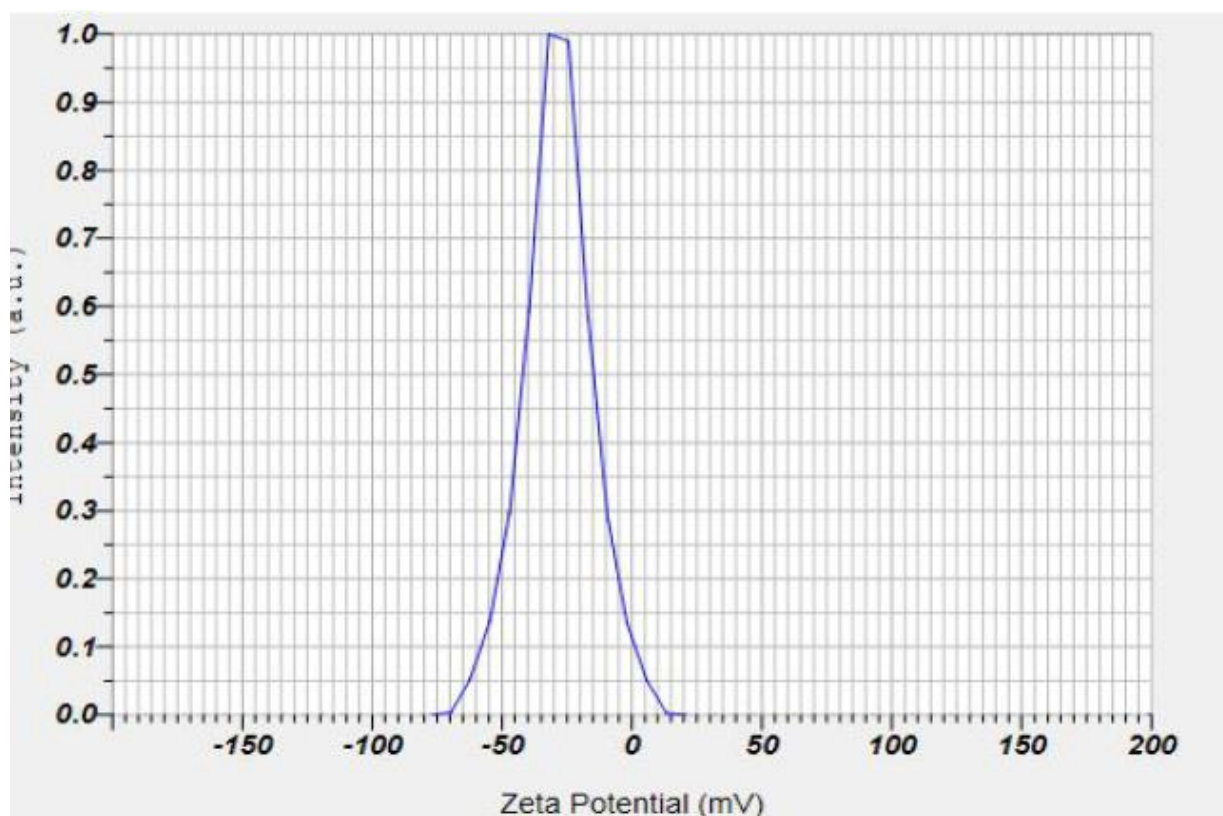


Fig 5.2.3.1:Zeta potential measurement shows the over all surface charge of AgNPS - 28.4 mv.

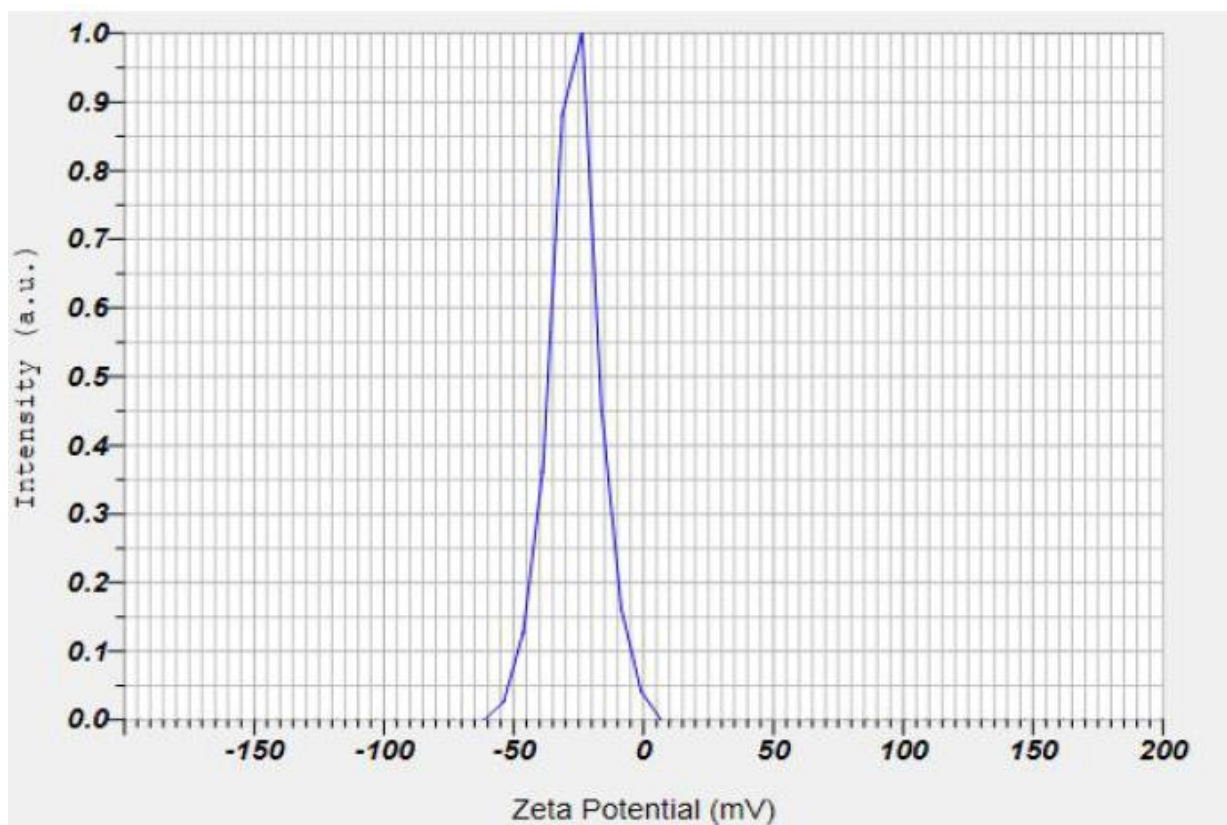


Fig 5.2.3.2:Zeta potential measurement shows the over all surface charge of PEG AgNPs -26.4mV .

5.2.4. Fourier Transform -Infrared Spectroscopy:

FTIR spectroscopy is an appropriate technique to confirm the chemical bonding, PEGylation to the surface of silver nano particles. The characteristic FTIR absorption band of AgNPS, PEG-AgNPS are presented in **Fig :7**. PEG-AgNPS shows strong IR absorption in 400-500 cm^{-1} region due to the presence of AgNPS. The characteristic band at 945.12 cm^{-1} , 1637.56, 1080.14 cm^{-1} and 1251.80 cm^{-1} correspond to the C-O out of plane bending vibration, the band at 1300.02 corresponds to the C-H alkene stretching band respectively, suggesting that the PEG is coated on the surface of AgNPs . The -OH stretching band around 3346.50 cm^{-1} indicates the physical adsorption of the PEG on the surface of Ag nano particles .

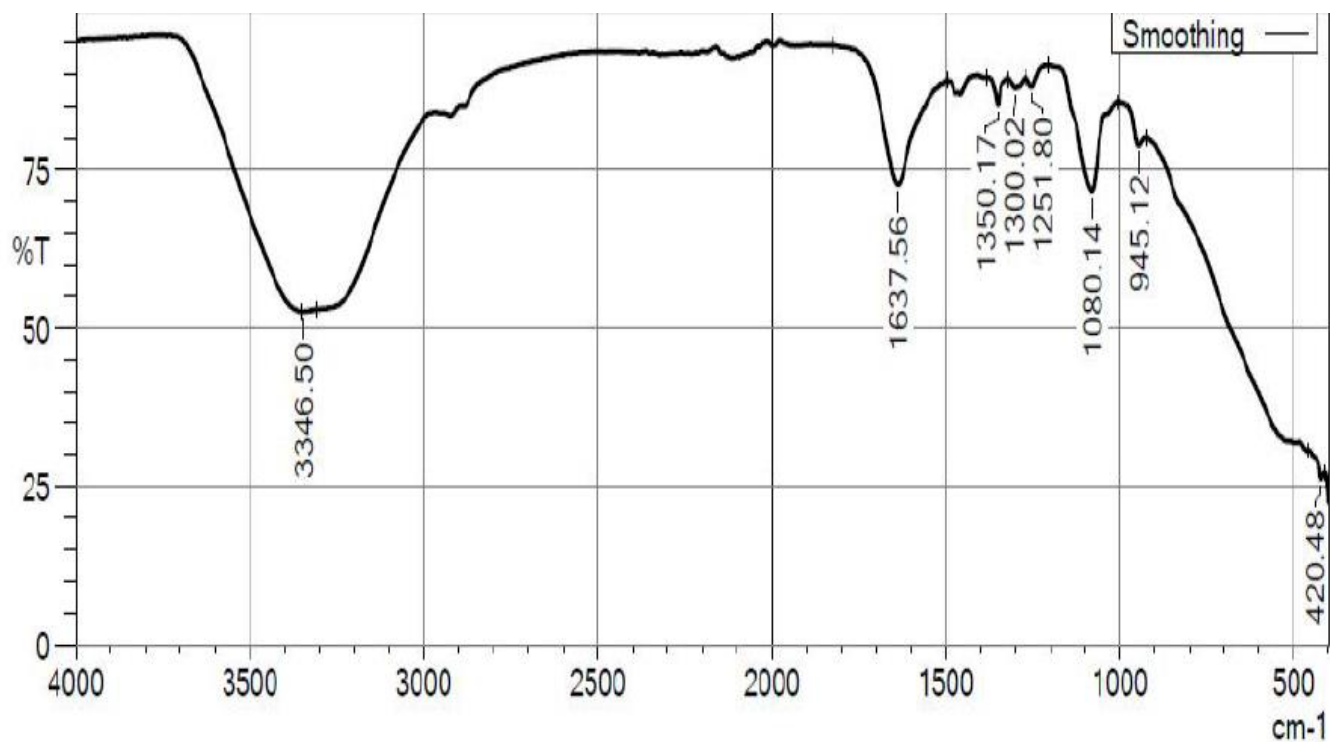


FIG 5.2.4.1 FT-IR analysis for PEG AgNPs.

Table 6.2.4.1:FT-IR analysis for PEG-AgNPs.

PEAK	BOND	FUNCTIONAL GROUP
420.48		
945.12	C-O	Carboxylic acid
1080.14	C-O	Carboxylic acid
1251.80	C-O	Carboxylic acid
1300.02	C-H	Alkene
1637.56	C=O	Carboxylic acid
3346.50	OH	Alcohol

CHAPTER 6 : SUMMARY AND CONCLUSION:

- Silver nanoparticles and PEGylated silver nanoparticles were synthesised and characterised using chemical reduction method.
- PEG act as a reducing and capping agents.
- The synthesized NPs were characterized using UV – Visible spectroscopy; FT-IR; DLS;
- The synthesised Silver nanoparticles and PEGylated silver nanoparticles will be used for various biological applications.

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