EFFECT OF REDUCING AGENTS TYPES - ON THE SYNTHESIS OF SILVER NANOPARTICLES

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SUPERVISOR'S DECLARATION

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of degree of Bachelor of Chemical Engineering.

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STUDENT'S DECLARATION

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged. The thesis has not been accepted for any degree and is not concurrently submitted for award of other degree.

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Special Dedicated to my beloved parents, family,
my friends, my fellow colleague
and all faculty members.

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EFFECT OF REDUCING AGENTS TYPES - ON THE SYNTHESIS OF SILVER NANOPARTICLES

ABSTRACT

In recent years, metal nanoparticles such as silver have been studied extensively due to their unique properties that are significantly different from those of bulk material. In this study, silver nanoparticles were synthesized by chemical reduction method with different type and concentration of reducing agents which is glucose and Cetyl trimethylammonium bromide (CTAB). The effect of the reducing agent on the size and morphology of the silver nanoparticles has been investigated. In this study, the formation and morphology of nanosized silver nanoparticles has been characterized by using UV-VIS spectroscopy, and Field Emission Scanning Electron Microscopy analysis (FESEM), respectively. Meanwhile, the purity of element on the synthesized silver nanoparticles has been carried out by Energy-dispersive X-ray spectroscopy (EDX). From the results of UV-Vis, silver nanoparticles that used glucose as a reducing agent showed narrow size distribution compared to CTAB. The maximum absorbance of silver nanoparticles for glucose as reducing agent is 0.481 while for CTAB is 0.831. The average size of the resulting silver nanoparticles for the concentration were determined by Image J software and result for the size is 10 nm with the high purity (91.95 % weight) using glucose and (77.78 % weight) when used CTAB as a reducing agents. During sample preparation, glucose showed a slow reaction as reducing agents compare with CTAB, which is more suitable to control a size and morphology of silver nanoparticles. The synthesis of silver nanoparticles has remains a formidable challenge in order to find a simple way to generate monodisperse silver nanoparticles with small size at high concentration.

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ABSTRAK

Dalam tahun-tahun kebelakangan ini, nanopartikel logam seperti perak telah dikaji secara meluas kerana sifat-sifat unik yang ketara berbeza daripada bahan pukal. Dalam kajian ini, nanopartikel perak telah disintesis melalui kaedah pengurangan kimia dengan jenis yang berbeza dan kepekatan agen penurunan yang glukosa dan cetyl trimethylammonium bromida (CTAB). Kesan agen penurunan kepada saiz dan morfologi nanopartikel perak telah disiasat. Dalam kajian ini, pembentukan dan morfologi nanopartikel perak nanosized telah disifatkan dengan menggunakan UV-VIS spektroskopi, dan analisis Field Pelepasan Imbasan Elektron Mikroskop (FESEM), masing-masing. Sementara itu, berhadas elemen pada nanopartikel perak disintesis telah dijalankan oleh Tenaga serakan X -ray spektroskopi (EDX). Daripada keputusan UV- Vis, nanopartikel perak yang digunakan glukosa sebagai agen penurunan menunjukkan taburan saiz sempit berbanding CTAB. Absorbans optimum nanopartikel perak untuk glukosa sebagai agen penurunan adalah 0,481 dan bagi CTAB mengurangkan ejen adalah 0.831. Saiz purata nanopartikel perak yang terhasil untuk kepekatan ditentukan oleh Imej perisian J dan menyebabkan untuk saiz ialah 10 nm dengan keaslian yang tinggi (91.95 % berat) menggunakan glukosa dan (7.78 % berat) apabila menggunakan CTAB sebagai agen penurunan . Semasa penyediaan sampel, glukosa menunjukkan reaksi yang perlahan mengurangkan ejen membandingkan dengan CTAB, yang lebih sesuai untuk mengawal saiz dan morfologi nanopartikel perak. Sintesis nanopartikel perak telah kekal sebagai cabaran yang besar untuk mencari cara yang mudah untuk menjana nanopartikel perak monodisperse dengan saiz kecil pada kepekatan yang tinggi.

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^{o}C	Degree Celsius
%	Percentage
ml	Mililiter
O	Oxygen
C	Carbon
Cl	Chlorine
nm	nanometer
mM	mili Molar
keV	Kilo electron Volt
AA	Ascorbic Acid
kHz	Kilohertz
g	Gram
Ag	Silver
$AgNO_3$	Silver Nitrate
Q	Amount of Material
AgNPs	Silver Nanoparticles
SPR	Spectrum Plasmon Resonance
CTAB	Cetyl Trimethylammonium Bromide
UV-Vis	Ultraviolet Visible
EDX	Energy Dispersive X-Ray
FESEM	Field Emission Scanning Electron Microscopy
ANOVA	Analysis of Variance

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF RESEARCH

Nanomaterial is one of essential building in nanotechnology field that emerge from physical, chemical and engineering sciences with the techniques and procedures materials at nanoscale level (Mahendran et al., 2013). The historical development of nanoparticles starting with Paul Ehrlich and then first attempts by Ursula Scheffel and colleagues in the late 1960s and early 1970s which described from a personal point of view (Kreute, 2006). In general, nanoparticles are defined as particulate dispersions or solid particles with a size in the range of 10 to 100 nm (Sally et al., 2007). It is accepted in the context of nanoscience and nanotechnologies, the units should only be those of dimensions, rather than of any other unit of scientific measurement (Kholoud et al., 2009). The scientific interest of nanoparticles is depends on the effectively a bridge between bulks materials and atomic or molecular structures regarding to its size at nanoscale and observations of size dependent properties (Kandarp & Mihir, 2013).

The characteristic of silver nanoparticles has a unique electronic, optical, thermal and catalytic property. The nanometer-size particles show unique because it can considerably change physical, chemical and biological properties compare to the macro scaled counterparts, due to their high surface to volume ratio. While the optical characteristic of nanoparticles which is including transparency, absorption, luminescence and scattering is depended on their particle size as it will determined their surface properties (Christina et al., 2011). The characteristic of silver

nanoparticles can be obtained by analyzing the spectral properties at absorbing and scattering of light. The silver nanoparticles will delocalized and shared amongst neighbouring particles when the particles are aggregate and the conduction electrons are near between the each particles surface (Kandarp & Mihir, 2013).

Moreover, the high electrical conductivity, stability and the low sintering temperature usually included in conductive inks, pastes and filler. Additional applications include molecular diagnostics and photonic devices also show the advantages of the optical properties of the silver nanoparticles. However, in spite of these advantages, nanoparticles do have limitations. For example, their small size and large surface area can lead to particle aggregation, which making physical handling of nanoparticles difficult in liquid and dry forms. Different parameters such as time, temperature and the concentration of the reducing agent and the surfactant can be manipulated in order to control the particles size distribution (Mohanraj & Chen, 2006). In all synthesis method, it is vital to obtain uniform, narrow particles distribution as well as low particles agglomeration.

Furthermore, silver nanoparticle is also commonly used as antibacterial application such as wound dressing, medical catheters, and bone prostheses (Sahoo et al., 2009). In the treatment of wounds, silver nanoparticles can be replaced with the silver sulfadiazine as an effective agent (Kandarp & Mihir, 2013). Recently, many new industries involves in production of antibacterial gels using silver nanoparticles. Nanoparticles also play a crucial role in inhibiting bacterial growth in aqueous such as in water treatment because of it high reactivity due to the large surface to volume ratio. It also contains material that can be used in water treatment. The reduction of silver ions to silver metals can be observed as silver nanoparticles display intense colour due to surface Plasmon resonance which were depending on the size of the particles. It has been discovered by previous work (Maribel et al., 2009) that at room temperature, the reaction would take several weeks to reach completion but at reflux the reaction would be finished within seconds. At the higher temperatures, the particles become more disperse and the nanoparticles also were formed at room. The dispersions of silver nanoparticles display intense colors due to the plasmon resonance absorption (Maribel et al., 2009).

Furthermore, silver nanoparticles also can use as a conductive applications for the conductive inks to enhance thermal and electric conductivity. This product bringing a changing in the electrical technology field. Besides that, silver nanoparticles also has been used in home application such as for washing machine, refrigerator and aqua guards (Felba., 2011). In this study, the reduction of silver nanoparticles will be carried out by organic and inorganic reducing agents. Glucose and cetyl trimethylammonium bromide (CTAB) will be used as reducing agent while ascorbic acid as a surfactant to control the dispersion of silver nanoparticles. It is very important to stabilize dispersive nanoparticles during the course of silver nanoparticle preparation, and protect the nanoparticles that can be absorbed on or bind onto nanoparticle surface.

1.2 PROBLEM STATEMENT

The synthesis of silver nanoparticles also is one of the challenging processes because the most important in this synthesis is to get narrow size distribution which has a range from 10 to 100 nm. This study only focused on the effect of reducing agent concentration on the size and morphology of silver nanoparticles. The challenge in synthesis of silver nanoparticles is to find a simple way to generate monodisperse silver nanoparticles. To achieve the narrow size distribution, the parameter such as of concentration should be controlled using chemical reduction method. Therefore, the method of synthesize silver nanoparticles remains a formidable challenge in order to find a simple way to generate monodisperse silver nanoparticles (Jiping et al., 2011). To keep the size of nanoparticles small, the initial concentration of silver salt in a reaction system must be lower and the reducing agents should be strong. If the initial concentration is high, then silver particles may grow too large. This study only focused on the effect of reducing agent concentration on the size and morphology of silver nanoparticles.

The other challenging in synthesis of silver nanoparticles is due to the difficulty to control the stability of the size and shape of particles in aqueous solution for the long term (Amadeus et al., 2012). It is depends on the medium of silver nanoparticles are immersed in because when the nanoparticles are applied and stored

in aqueous condition, the silver nanoparticles will interact with biological matter and living cells and will affected to the aggregation, size and shape of the nanoparticles and long-term stability (aging). In view of the rapidly progress, silver nanoparticles is growing need in the different fields for many applications.

The technological and environmental also is one of challenges where in the areas of solar energy conversion, catalysis, medicine and waste treatment also consider in order to control the size particles and composition of silver nanoparticles (Rupiasih et al., 2013). In addition, the silver nanoparticles have unique catalytic, optical, electrical and antimicrobial properties. Silver is a nontoxic inorganic antimicrobial agent, which is inhibiting the microbe's growth. So, by using the concentration of reducing agents in synthesis silver nanoparticles can control the size and shape distribution in long term.

1.3 OBJECTIVE

The main objective of this research is to study the optimum concentration of reducing agent on the synthesis of silver nanoparticles by determine:

a) Effect of concentration of glucose as a reducing agent.
b) Effect of concentration of Cetyl trimethylammonium bromide (CTAB) as a reducing agent

1.4 SCOPE OF RESEARCH

In synthesis of silver nanoparticles, the main scope of this study is to determine the optimum concentration of reducing agents with the narrow size distribution which has a range from 10 to 100 nm. The chemical reduction method will be used to synthesize the silver nanoparticles because it is the most commonly methods used, simplest, and easiest. Glucose and cetyl trimethylammonium bromide (CTAB) will be used as a reducing agent with difference concentration, while ascorbic acid as a surfactant.

In order to get the results, UV-Vis absorption spectroscopy is used to monitor the formation of the silver nanoparticles and the change color of solution also can illustrate from visible observation. The optimum absorbance for the sample can showed the optimum concentration for the sample. Therefore, the Field emission scanning electron microscopy *(*FESEM*)* will be used to get the structural properties of silver nanoparticles with high-resolution surface images and image J software is used in order to determine the particles size distribution, where the size distribution can illustrated from the shape of histogram. After that, the elemental analysis of sample also has been performed using energy dispersion X-ray analysis (EDX). This analysis also to detect the other impurity contains in the sample except the pure silver, with no oxide.

1.5 RATIONAL AND SIGNIFICANCE OF RESEARCH

Silver nanoparticles such as silver have been studied extensively due to their unique properties that are significantly different from those of bulk material. These unique properties could be attributed to their small size and large surface area which is in many applications including electronic, catalyst and photonic. Currently, nanoparticles based on silver nanoparticles are applied in antimicrobial coatings, and many textiles, keyboards, wound dressings, biomedical devices, ink-jets, inks, safety labels, pigments, conducting strips. Based on this research, the variable parameter is the concentration of glucose and CTAB as reducing agents. Each different concentration of reducing agents will give the different result of morphology and the size of distribution. The narrow size distribution will produce from the difference concentration of glucose and CTAB, which the results from UV-Vis spectroscopy for the different trends of graph. When the wavelengths for the concentration of reducing agents increase, the size of particles also increase. After that, the increasing concentrations of reducing agent, the color of the solution will changes. According this study, the selected for the best result is depending on the uniform shape and narrow size distribution with determine the optimum concentration reducing agents.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

A reducing agent is the element or compound in a reduction oxidation reaction that donates an electron to another (John, 2003). The surfactants are to control the morphology and particles size of silver nanoparticles. If the surfactant molecule is dissolved in a nonpolar medium, it can intersect with other part from the nonpolar dispersant (Palash et al., 2010). The important role of this process is the value of pH, temperature, stabilizer and concentration. From the previous study (Sahoo et al., 2009), the optimum pH is found between 8.5 – 9.0 as it a uniform particles size and narrow distribution. Meanwhile, when the value of pH is higher, the silver nanoparticles are more stable.

During synthesis process, the stabilizer adsorbs on the solid-liquid interface and forms a layer of molecular. It also can reduce surface tension by reducing the absorption capability. Therefore, when the concentration of reducing agent is increase, the solutions become more cluster but it does not affect the particles size distribution. Besides that, it also reported that the reduction process of silver ions is higher in alkaline solution (Manjeet et al., 2009). Furthermore, the large amount of the stabilizer also can prevent nanoparticles reactions from the other compound. It is also become more entanglement in the dense network of the stabilizer (Malina et al., 2012).

According the previous study (Chang et al., 2009), the chemical reduction method is used for reduction of $AgNO_3$ by sodium borohydride ($NaBH_4$) as a reducing agent and sodium dodecyl sulfate (SDS) as a surfactants. From previous work, it shows that when the concentration of AgNO3 increased, the color of solution changed from yellow to brown. Meanwhile, a weak absorption maximum of surface Plasmon peaks was observed at 400 nm, indicating the formation of silver nanoparticles. The intensity of the maximum Plasmon peak was reported to be increased as concentration of AgNO3 increase, indicating that more silver nanoparticles were formed.

2.2 SILVER NANOPARTICLES

Shape and size control of silver nanoparticles is a promising strategy to tailor their physical and chemical properties for various applications in the field of photonics, catalysis, medical research and surface enhanced Raman spectroscopy (SERS). Superior physical and chemical properties were observed for the metal nanostructures with complex shapes (Etacheri et al,. 2010). A typical example is the shape and size dependent optical properties exhibited by triangular silver nanoplates. Triangular plates show three surface plasmon resonance (SPR) bands corresponding to dipole and quadruple plasmon resonance, but only one SPR band is observed for spherical silver nanoparticles. A large number of aqueous and non-aqueous methods were reported in the past decade for the synthesis of silver nanoparticles having different size and shapes such as rods, prisms, cubes, wires and disks (Etacheri et al,. 2010).

Colloidal silver nanoparticles synthesized in a polymer matrix have wide applications as biosensors, antimicrobial agents, catalysts and in new generation light weight electronic devices. A battery of techniques is available in the literature to synthesize silver nanoparticles in aqueous as well as in non aqueous medium. The general philosophy of the synthesis of metal nanoparticles from its salt solution is based on using a reducing agent in presence of a capping agent. Capping agents keep the nanoparticles away from agglomeration besides modifying their morphology as well (Patakfalvi1 et al., 2006).

2.3 OPTICAL PROPERTIES OF SILVER NANOPARTICLES

UV-VIS absorption spectra have been proved to be quite sensitive to the formation of silver colloids because silver nanoparticles exhibit an intense absorption peak due to the surface Plasmon excitation. The absorption band in visible light region (350 nm – 550 nm, Plasmon peak at 445 nm) is typical for silver nanoparticles. The Plasmon peak and the full-width of half-maximum (FWHM) depend on the extent of colloid aggregation. To monitor stability of the silver nanoparticles, we have measured the absorption of the nanoparticles after different periods of time (Sally et al., 2007). In this research, the stability of particles size distribution has determined by different concentration of reducing agents.

There was no obvious change in peak position, except for the increase of absorbance. As the particles increase in size, the absorption peak usually shifts toward the red wavelengths. Increase of absorption indicates that amount of silver nanoparticles increases. The stable position of absorbance peak indicates that new particles do not aggregate. One can understand that since the silver colloidal particles possessed a negative charge due to the adsorbed citrate ions, a repulsive force worked along particles and prevented aggregation (Sally et al., 2007).

To determine the particle size we performed several calculations changing the radius of particles and compared absorption dependencies on wavelength with the experimental UV-Vis absorption spectrometry results. In theoretical calculations the radius of particle was changed from 5 nm to 100 nm. We have chosen scattering from many spheres, because there was possibility that particles in colloidal solution are not uniform (Sally et al., 2007).

2.4 REDUCING AGENTS

The reduction of silver cations at different reducing agent/loading agent molar ratios, when the reducing agent is increased in the same mixture of AgNO3. The maximum absorption band is shifted to shorter wavelengths. UV–vis absorption bands when the reducing agent concentration is increased, an increase of the reducing agent will produces an absorption band shift to shorter wavelengths. The position of the maximum absorption bands shifted to shorter wavelengths when concentration was increased, and the resulting colors are formed in a different order (from violet to orange) during the synthesis process.

However, when the molar was increased, the maximum absorption band shifted to short wavelengths with a corresponding change of color (brown or green). Furthermore, when higher molar was added to the solution (with orange color only), a new intense absorption band appeared at 100 nm which was indicative of the formation of nanoparticles with spherical shape. These same spectral absorption variations in both regions have been observed with higher concentrations (Perdo et al., 2013).

2.5 STABILITY

Stability of the silver nanoparticle formed in a solution is a highly challenging area when considering the size and shape dependant properties. The shape of silver nanoparticles undergo etching and finally convert to spherical particles resulting in a blue shift of the in-plane dipole resonance. The large blue shift, which can be up to 100 nm in magnitude, is a direct measure of particle instability and a small blue shift value usually indicates higher particle stability. An etching study of the green sol up to 1 hr after the final addition of Ag+ has been performed to examine the stability of the formed nanoparticles. In the present case, the blue shift observed for triangular nanoparticles were very small (5 nm), which clearly indicate the formation of stable silver nanoparticles (Vinodkumar et al., 2010).

2.6 GROWTH OF SILVER NANOPARTICLES

Growth of the nanoparticles was controlled by only varying the concentration such as ascorbic acid. A single Plasmon resonance around 400 nm was formed without ascorbic acid which indicates the presence of only spherical nanoparticles (18 nm size). Crystallization of triangular nanoparticles and a corresponding second Plasmon resonance at a higher wavelength were observed as a result of increasing the amount of ascorbic acid. A gradual red shift of second Plasmon resonances were also observed on increasing the concentration of ascorbic acid and a green coloured sol containing biggest particles were obtained using 50 mM ascorbic acid. Absorption band associated with spherical nanoparticles was also found in all other nanoparticles colloids. Intensity ratios of these peaks were found to be highly dependent on the concentration of ascorbic acid. The red coloured sol containing 10 mM ascorbic acid was found to have a higher intensity peak around 400 nm and a lower one around 500 nm (Vinodkumar et al., 2010).

On increasing the ascorbic acid concentration, the lower wavelength peak intensity decreases with a corresponding intensity increase of the higher wavelength peak. Shifting of the plasmon resonances to higher wavelength associated with a decrease in absorption intensity of spherical particles indicates the growth of triangular silver nanoparticles on the expense of spherical nanoparticles. Thus spherical and triangular nanoparticles were found to be stabilized at lower and higher ascorbic acid concentrations respectively. From these results it is clear that the concentration of ascorbic acid play an important role in the growth of triangular silver nanoparticles (Vinodkumar et al., 2010).

2.7 CHEMICAL REDUCTION METHOD

In 1857, Micheal Faraday, for the first time reported a systematic study of the synthesis colors of colloidal gold using chemical reduction route (Asim et al., 2012). The chemical reduction method is one of the commonest methods to synthesize of silver nanoparticles because of its convenient operation, ease of control and simple. The chemical reduction method involves the reduction of $AgNO_3$ by a reducing agent in the presence of a suitable stabilizer, which is necessary in protecting the growth of silver particles through aggregation.

In the formation of silver nanoparticles by the chemical reduction method, the particle size and aggregation state of silver nanoparticles are affected by various parameters, such as initial $AgNO_3$ concentrations, reducing agent/$AgNO_3$ molar ratios, and stabilizer concentrations. Besides that, this method also can controlled the morphologies with the strongly during the synthesis because it is very strong depend on the temperature adopted (Reza et al., 2011). Based on this method also, the size of particles will achieve in range 10 to 100 nm.

CHAPTER 3

METHODOLOGY

3.1 MATERIAL AND SOLVENTS

The synthesis of silver nanoparticles was carried out using standard synthetic chemical procedure and commercially available reagents. Glucose and Cetyl trimethylammonium bromide (CTAB) were purchased from Sigma Aldrich with purity 96 % and 99%, respectively. Ascorbic acid with purity 99% is used as a surfactant which is a reagent grade also purchased from Sigma Aldrich. Besides that, silver nitrate ($AgNO_3$) with purity 99.9% in a crystalline form also purchased from Sigma Aldrich is used as a precursor for synthesis of silver nanoparticles. For analysis of sample, ethanol were used with purity 99.5% and also purchased from Sigma Aldrich. The entire chemicals were used as received without purification.

3.2 APPARATUS AND EQUIPMENT

For synthesis of silver nanoparticles, the main apparatus and equipments that have been used were 250 ml two-neck-flask, thermometer, magnetic bar, thermometer stand and hot plate with magnetic stirrer. The other apparatus in preparations of solutions is 600ml beaker, 250 beaker, stopper and 50 ml measuring cylinder. During the centrifugation process, the centrifuge tubes were needed for centrifuge the sample. After that, ultrasonic cleaning also used as an equipments for the preparation on synthesis of silver nanoparticles. For this part, a beaker is needed to cleaning the samples.

In this study, the samples obtained in synthesis process were characterized using UV-Vis adsorption spectroscopy, Field Emission Scanning Electron Microscopy analysis (FESEM) and Energy Dispersive X-ray spectroscopy (EDX). The freeze drying cuvette is used for UV-Vis adsorption and dropper for drop the sample on study for analysis of FESEM and EDX.

3.3 RESEACH DESIGN

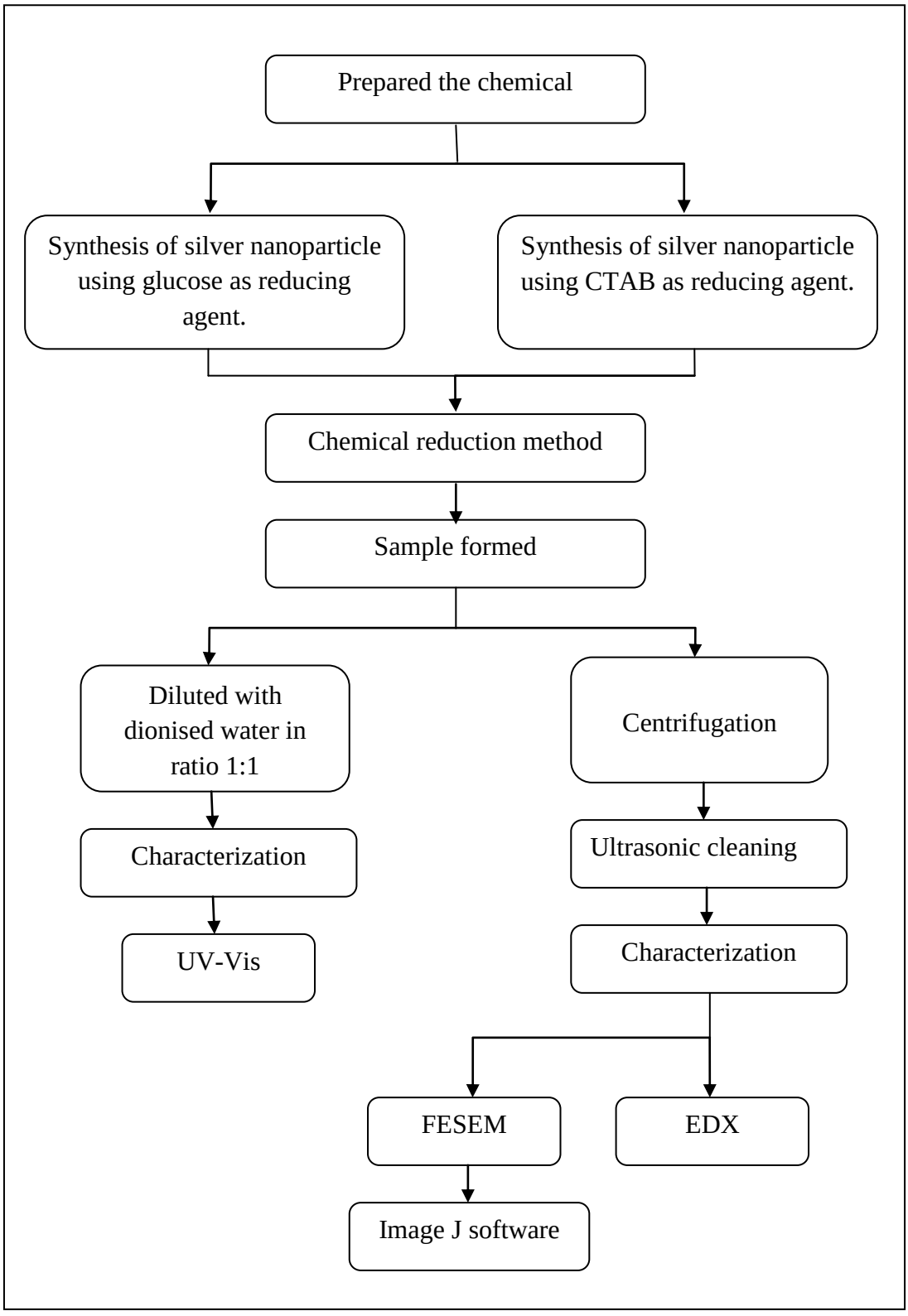

Figure 3.1: Methodology for Synthesis of Silver Nanoparticles

3.4 PREPARATION FOR SYNTHESIS OF SILVER NANOPARTICLES

The synthesis of silver nanoparticles was prepared using chemical reduction method for different concentration of reducing agents by the following step:

i. 1 mM of silver nitrate (0.085 g) was dissolved in 500 ml of dionised water and stirring until the fully dissolved.

ii. 0.5 mM of glucose (0.036 g) as a reducing agent also was dissolved in 400 ml dionised water for another beaker.

iii. After glucose is fully dissolved, added 2 mM of ascorbic acid (AA) (0.085 g) into the glucose solution. Make sure all the materials are fully dissolved with the effective stirring.

iv. Then, 80ml of silver nitrate solution was heated into conical flask with thermometer and 20 ml of glucose and AA also was heated into two neck flask with a stirrer and thermometer until the temperature for both solutions is reached to 60^{o}C.

v. When the temperature for both solution was reached to 60^{o}C silver nitrate solution was mixed into glucose and AA solution with maintain the temperature 60^{o}C for 20 minutes with vigorous agitate by magnetic stirrer.

vi. After 20 minutes of the reaction, the silver solution was left at room temperature and stored in a dark bottle.

vii. Step (i) to (vii) were repeated with different concentration of reducing agents and types of reducing agent as shown in Table 3.1.

The amount of material, Q has been calculated by using equation 3.1:

$$Q(g) = molar\ concentration \times \frac{volume\ water}{1000} \times molecular\ weight \qquad (3.1)$$

Table 3.1: parameter of the prepared samples silver nanoparticles at different concentration of reducing agents of glucose and CTAB respectively.

Experiment	Silver Nitrate (mM)	Reducing agents		Surfactant AA (mM)
		Glucose (mM)	CTAB (mM)	
S1	1.0			2.0
S2	1.0	0.5		2.0
S3	1.0	1.0		2.0
S4	1.0	1.5		2.0
S5	1.0	2.0		2.0
S6	1.0	2.5		2.0
S7	1.0		0.5	2.0
S8	1.0		1.0	2.0
S9	1.0		1.5	2.0
S10	1.0		2.0	2.0
S11	1.0		2.5	2.0

3.5 CENTRIFUGATION

The centrifugation is the purified process for silver nanoparticles process before analysis of FESEM and EDX. The aim of this process is to remove the excess of chemical or reagent in the samples solution. The method for centrifugation process by the following step:

i. 40 ml sample of silver nanoparticles was poured in 50 ml centrifuge tube and placed into the centrifuge machine.

ii. 8 samples can run at the same time. After placed the sample into centrifuge machine, set the setting of centrifuge with 3500 rpm of rotary and 15 minutes at the room temperature.

iii. After finish the centrifugation process, remove the solution at the top of centrifuge tube until solution reached to 15 ml. Then, 15 ml of ethanol (99.5%) solution was added in the silver solution for washed the silver nanoparticles.

iv. Step (i) to (iii) were repeated for the entire sample.

3.6 ULTRASONIC CLEANING

After finish the centrifugation process, the samples will continue for the cleaning process using ultrasonic cleaning. The aim of this process is to clean the silver nanoparticles at the wall of the centrifuge tube. The method for doing this process by the following method:

i. 400 ml of dionised water was added into 600 ml beaker. Then, put the sample in the centrifuge tube into the beaker.

ii. Open the ultrasonic cleaning and placed the beaker into the ultrasonic cleaning. After that, set the setting for operation with 9 kHz and 15 minutes at room temperature.

iii. After 5 minutes, the machine will stopped and open the sample from the ultrasonic cleaning. Lastly, the samples were stored into the dark bottle. Make sure the ultrasonic cleaning was stopped before take the samples.

iv. Step (i) to (iii) were repeated for the other samples.

3.7 CHARACTERIZATION

3.7.1 UV-VIS ABSORPTION SPECTROSCOPY (UV-VIS).

UV-visible measurements were carried out on Jasco V-570 UV/VIS Spectrophotometer at the resolution of 10 to 100 nm. UV-Vis absorption spectroscopy was used to monitor the formation of the silver nanoparticles. It is revealed the formation of silver nanopartícles by exhibing the typical surface plasmon absorption from the UV–Vis spectrum. The UV-Visible spectrophotometer uses two light sources, a deuterium (D_2) lamp for ultraviolet light and a tungsten (W) lamp for visible light.

The grating can be rotated allowing for a specific wavelength to be selected. At any specific orientation of the grating, only monochromatic (single wavelength) successfully passes through a slit. A filter is used to remove unwanted higher orders of diffraction. The light beam hits a second mirror before it gets split by a half mirror (half of the light is reflected, the other half passes through). One of the beams is allowed to pass through a reference cuvette (which contains the solvent only), the other passes through the sample cuvette. The intensities of the light beams are then measured at the end.

The sample were recorded the value of absorbance between silver nanoparticles and the dionised water with the wavelength from 300 nm to 800 nm. The total of samples can run are 4 samples at the same time but the results will move from one sample to another sample. The time interval for this analysis is 1 minutes for 200 nm of wavelength. The entire sample will carry out for this analysis and the result have been plotted in chapter 4.

3.7.2 FIELD EMISSION SCANNING ELECTRON MICROSCOPY (FESEM).

Field-emission scanning electron microscopy (FESEM) was used in a technical feasibility study to formation of structural of silver nanoparticles in nanometre into the internal morphology and size of silver nanoparticles distribution. FESEM observations were carried out using magnitude X20000 for the control process, X50000 for 0.5 Mm of glucose, X40000 for 2.5 mM of glucose, X25000 for 0.5 mM CTAB and X40000 for 2.5 mM CTAB.

The samples were dropped on the carbon tape at the stud. The solution should be fully dried before doing this analysis to get the good result. The Image J software was using to confirm the size of distribution of the sample. The results of structural and size distribution are shown in chapter 4.

3.7.3 ENERGY DISPERSIVE X-RAY (EDX).

Energy dispersive x-ray analysis **(**EDX) analysis was used to confirm the presence of elemental silver without any further impurities in the prepared of silver nanoparticles. The silver nanoparticles were prepared in the nanopowder form to this compositional analysis. The EDX profile showed the presence of elemental silver peak along with small carbon peak due to the adhesion of carbon tape on to the aluminium stud. The identification lines for the major emission energies for silver nanoparticles in the range 0 – 10 keV are displayed. After that, the reduction of silver into elemental silver and absence of other impurities also has been confirmed from EDX studies.

The EDX observations were carried out using magnitude X17000 for concentration 2.5 mM of glucose and X10000 for concentration 2.5 mM of CTAB as reducing agents in the synthesis of silver nanoparticles. The results of elemental silver peak for each element and the impurities for all the elements from the results are shown in chapter 4.

3.8 STATISTICAL ANALYSIS

All experiment is conducted with a new concentration of silver nanoparticles was repeated triplicate. Statistical analyses were performed using Data Analysis in Microsoft Excel 2010. Two ways ANOVA without replication was utilized to compare the optimum absorbance from different silver concentrations and *t* test was used to analyze the particles size distribution. The samples were considered statistically significant when $p < 0.5$. The results are shown in chapter 4.

CHAPTER 4

RESULT AND DISCUSSION

4.1 VISIBLE OBSERVATION OF SILVER NANOPARTICLES

The silver nanoparticles synthesized were prepared by a chemical reduction method in aqueous solution in the presence of ascorbic acid as a surfactant, while glucose and CTAB were used as a reducing agent. In synthesis of silver nanoparticles, glucose is known as one of mild reducing agent while CTAB is the strong reducing agents. During synthesis process, the colourless solution of silver nitrate went through a number of colour changes from colourless to pale yellow, yellow, brownish or greenish before it stabilized. This final colour solution which is after the synthesis process has been stopped is depending on the concentration of reducing agent as well as surfactant. In this study, different concentration of both reducing agents; glucose and CTAB resulted in different colour of silver solution. Besides that, the changing colour for different concentration of CTAB shows the faster rate of changes compare with the different concentration of glucose.

The colours of silver solutions at various concentration of glucose gradually change from yellowish to yellow-green and turn to pale brown, whereas for silver solutions at various concentration of CTAB, the colour change from yellowish to dark green and then to brown. The changing of colour of the solution was preliminarily confirmation of silver nanoparticles formation. This indicates the formation of Ag-NPs in both of silver solutions. The comparisons color between two reducing agents (glucose and CTAB) with concentration 0 mM, 0.5 mM, 1.0 mM,

1.5 mM, 2.0 mM and 2.5 mM were observed and shown below in Figure 4.1 and Figure 4.2.

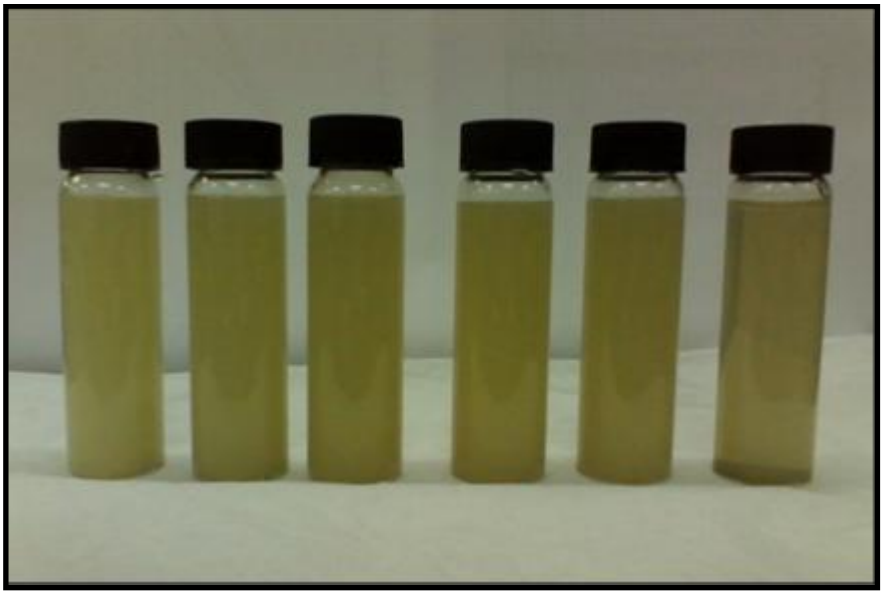

Figure 4.1: Color change indicates the formation of silver nanoparticles by different concentration of glucose.

From the results of synthesis of silver nanoparticles using 0.0 mM of glucose, the solution colour that formed is pale yellowish. However, when the concentration of glucose is increase, the silver solution has started to change from yellowish to greenish. The occurrence of colour changes from yellowish to greenish for glucose at concentration of 1.0 mM to 1.5 mM. After that, the solution changes to brownish at 2.0 mM of glucose, where the molar concentration of reducing agent is same with the molar concentration of surfactant (2.0 mM of ascorbic acid). Furthermore, increasing concentration of glucose at 2.5 mM, did not show any changes and the silver solution is remain brownish eventhough the molar concentration of reducing agents are larger than the molar concentration of surfactant.

The result at different concentration of CTAB is shown in Figure 4.2. The colours of the solution represent the existence of silver ions in the solution. From Figure 4.2, it can be seen that at 0.0 mM to 0.5 mM, the solution has changes from yellowish to cloudy greenish and turn to the dark green at 1.0 mM of CTAB. After that, the silver nanoparticles started to changes the colour to brown at 1.5 mM to 2.5

mM. From the observation during the synthesis process, the rate of changes of colour using CTAB as reducing agents is faster than glucose.

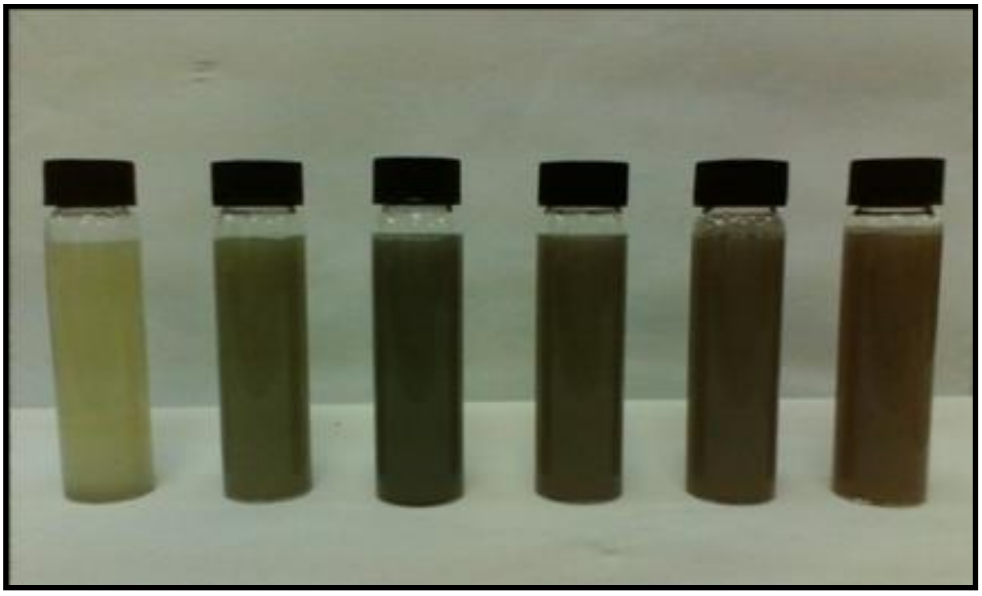

Figure 4.2: Color Change Indicates the Formation of Silver Nanoparticles by different concentration of CTAB.

The changing color of solution also can represent the interaction between ascorbic acid and reducing agents with the silver nitrate solution. From the observation, the CTAB have a strong interaction compare than glucose because the change colour for different concentration of CTAB show the significant of change colour from yellowish to dark green and then to brown. It is also the reaction when using CTAB as a reducing agent is fast reaction occurs than glucose as a reducing agent. The change of color silver solution can indicate the different size, shape and wavelength for each solution.

From the results, the changing color of glucose is more stable from the change color for CTAB because the change color for glucose is almost same. According the previous study (Zaheer et al., 2011), when silver nanoparticles prepared with mild reducing agents, it was relatively more stable compared to strong reducing agents. The dispersions of silver nanoparticles also display intense colors due to the plasmon resonance absorption and the formation of silver nanoparticles in the solution was confirmed by UV-Vis absorption spectroscopy.

4.2 FORMATION OF SILVER NANOPARTICLES

The spectrum Plasmon resonance (SPR) wavelength of silver nanoparticles synthesized under different conditions varies with their size and shape. According to Mie theory, spherical nanoparticle of silver will exhibit a single SPR band, whereas an anisotropic particle has showed the multiple SPR bands. When the amount of reducing agent is increase, the position of maximum absorption wavelength is highly shifted to a larger wavelength. The maximum absorbance and wavelength for different molar concentration of glucose is shown in Figure 4.3.

From the results for different concentration of glucose, the Plasmon resonance band centered was shifted from 420 nm to 440 at the concentration 0.5 mM to 2.5 mM. Meanwhile, the values of absorbances are increasing from 0.423 to 0.481 at 0.5 mM to 2.0 mM with the constant wavelength. However, from this observation, the optimum concentration is obtained at 2.0 mM with the higher absorbance was at 0.481. Further increasing of glucose concentration at 2.5 mM, somehow show a decrement on the value of absorbance which is 0.428. The decrement on the value of absorbance at 2.5 mM was believed due to the slow reaction between the silver nitrate and glucose as well as increasing on size of the nanoparticles. The increasing of particles size then was confirmed by the spectrum of UV-Vis that shows red-shifted from 420 nm to 440 nm. The different molar concentration of glucose somehow not showed any significant different on the shape of graph between the each molar concentration with the differences in value absorbance for each molar concentration also small.

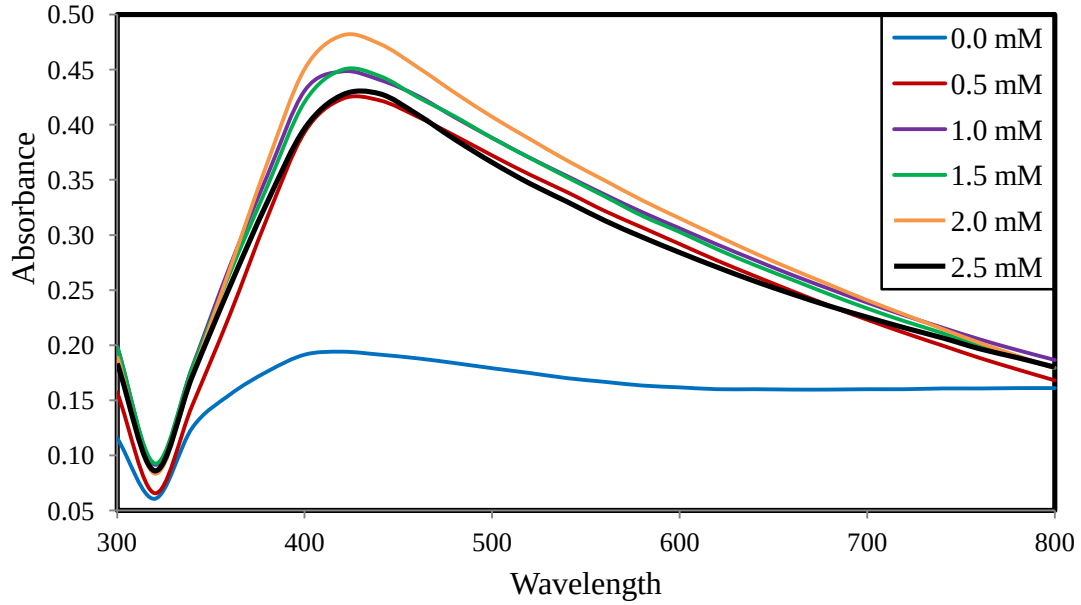

Figure 4.3: Spectrum Plasmon resonance for different molar concentration of glucose as reducing agents.

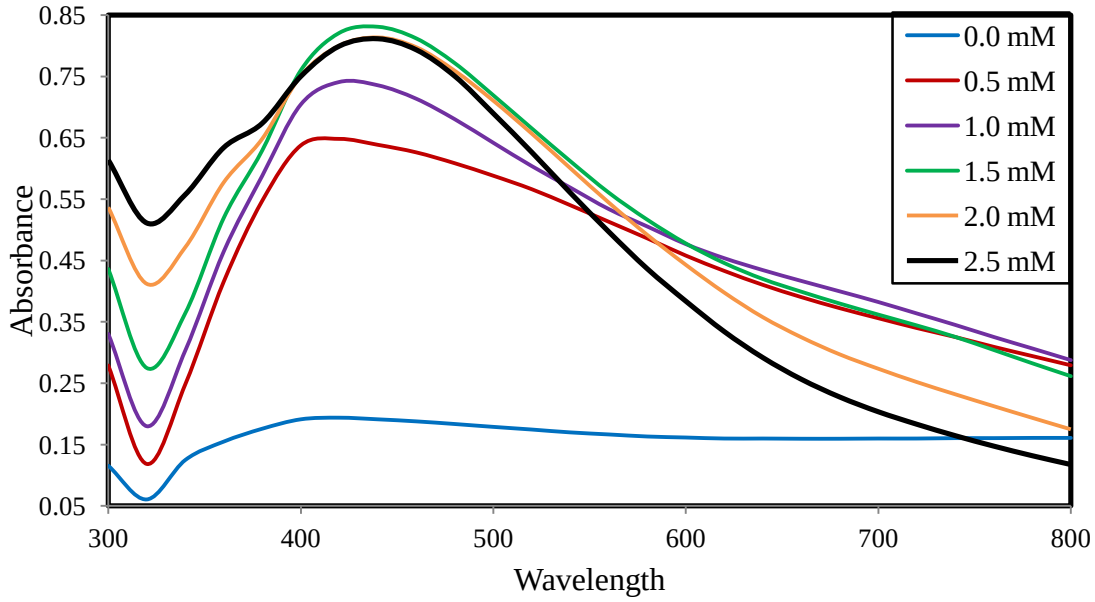

Figure 4.4: Spectrum Plasmon resonance for different molar concentration of Cetyl trimethylammonium bromide (CTAB) as reducing agents.

For results at different concentration of CTAB, the absorbance at 0.5 mM of CTAB is 0.648 at the wavelength 420 nm. The value of absorbance is found to be increased when the concentration of CTAB increase. From Figure 4.4, the optimum concentration of CTAB is at 1.5 mM with the value of absorbance is 0.831 and 440

nm of wavelength. After that, the absorbance decreased from 2.0 mM to 2.5 Mm and the wavelength is increase. The increasing wavelength from 420 nm to 440 nm indicated the red shift which also can be relates with the increasing of particles size. From this result also showed the CTAB can absorb the higher particles compared the value of absorbance for glucose. The formation of silver nanoparticles was found to be slow at lowest concentration and hence absorbance is also less. Once, the absorbance becomes constant, it – showed that the reaction is completed (Rimal et al., 2013).

4.3 PARTICLES SIZE DISTRIBUTION

The shape and particles size of silver nanoparticles that obtained from FESEM images shows the different size distribution of AgNPs with different concentration of reducing agents. From FESEM images, the average sizes of particles for glucose and CTAB from 0.5 mM to 2.5 Mm are same with the average 10 nm of size particles. From the Figure 4.4, it also can be seen that the particles tend to stick on the surface and form agglomerates. The particles of silver will increase when the concentration of reducing agents increased. This analysis was count for 100 particles for each concentration.

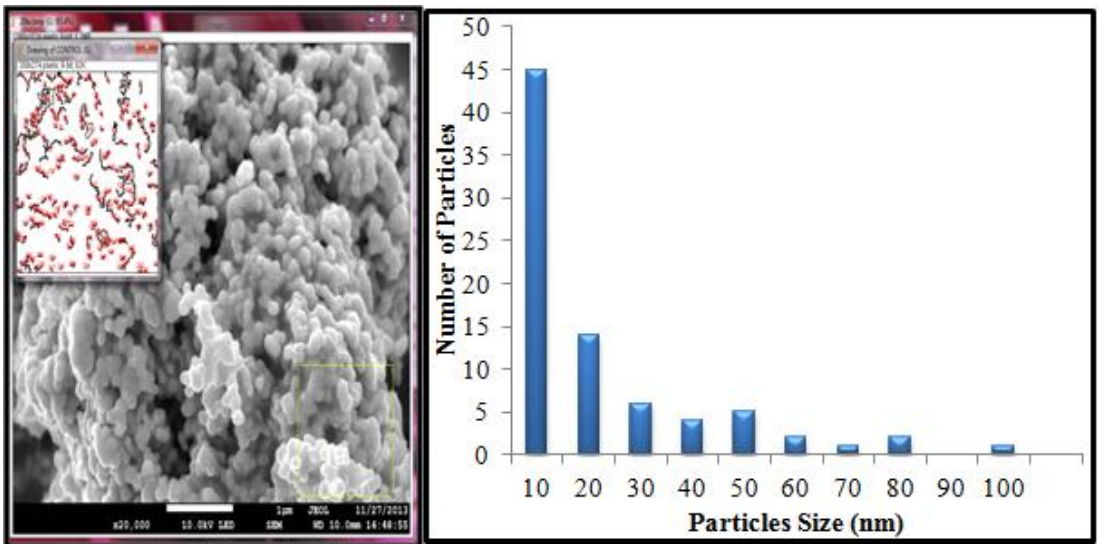

Figure 4.5: FESEM analysis and "Image J 1.34s" software for synthesis of silver nanoparticles without reducing agent.

From the result above, the silver nanoparticles showed the average size distribution is 6 nm with the standard deviation 1.7 for is 45 particles. When the size of particles increases, the number of particles is decrease. At these observations, the results showed no particles size larger than 100 nm.

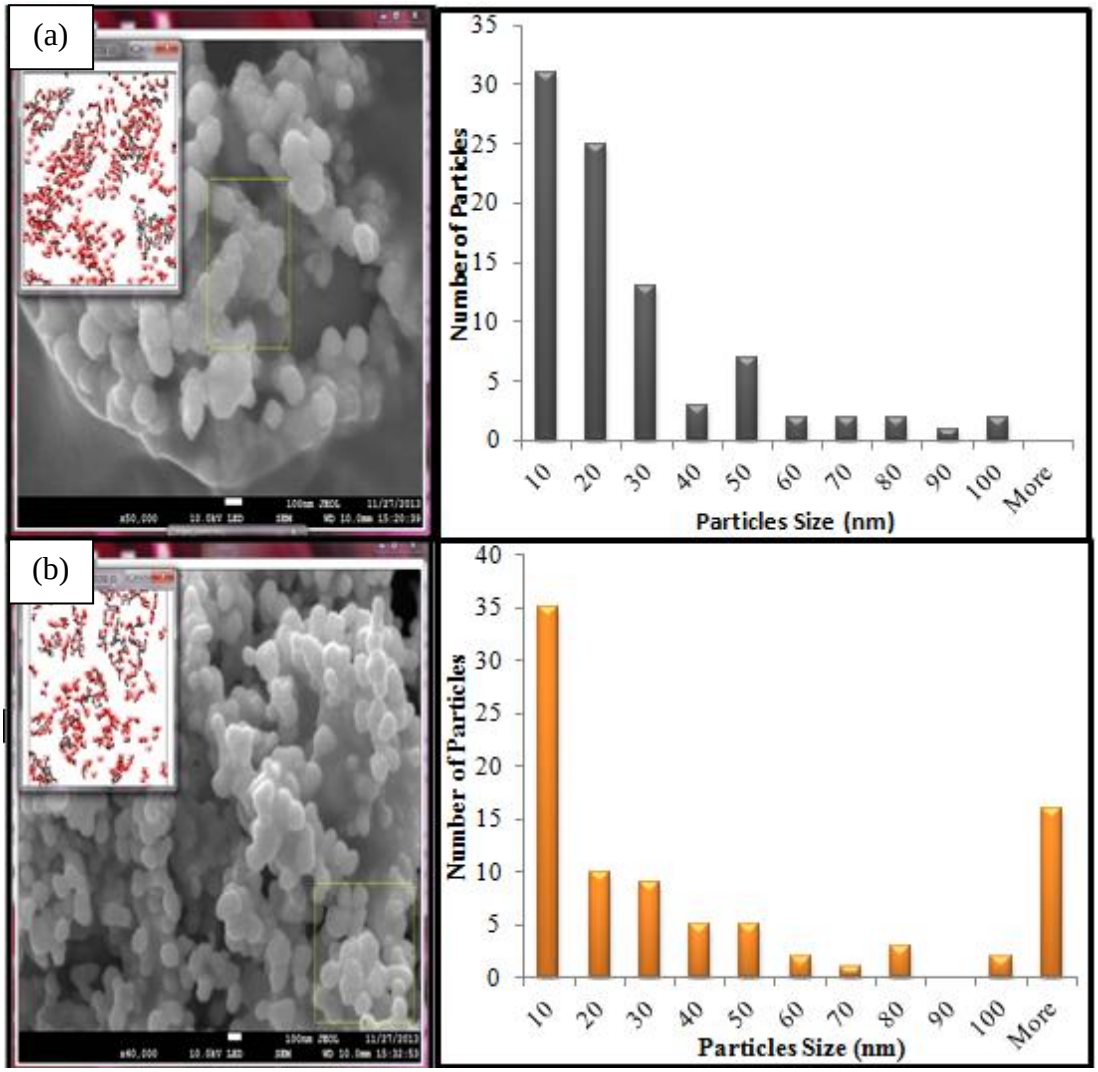

Figure 4.6: FESEM analysis and "Image J 1.34s" software for glucose as reducing agents (a) 0.5 mM Glucose (b) 2.5 mM Glucose

The results for 0.5 mM and 2.5 mM of glucose as reducing agents showed the different trends of graph and the particles size distribution of each concentration. The average size of particles for 0.5 mM of glucose is 7 nm with standard deviation 1.8 and for 2.5 mM of glucose is 6 nm with standard deviation 1.4. At 2.5 mM shows the narrow size distribution compare with 0.5 mM of glucose. From the different

concentration of glucose, 16 particles from 100 particles of analysis from 2.5 mM are appearing as a size of particles larger than 100 nm. Meanwhile, the concentration of glucose increase, the particles size distribution was more dispersed. Besides that, the optimum particles size also will increase from 0.5 mM glucose to 2.5 mM of glucose.

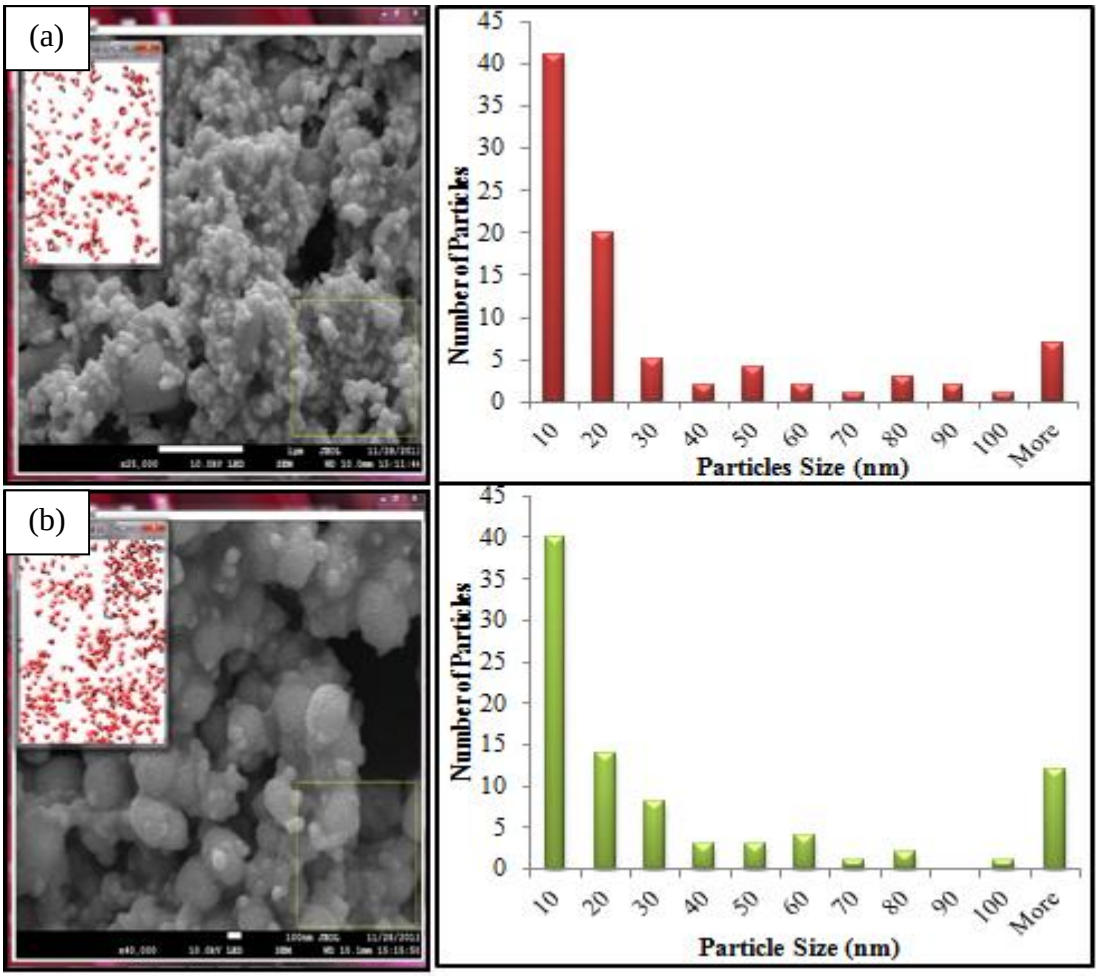

Figure 4.7: FESEM analysis and "Image J 1.34s" software for CTAB as reducing agents (a) 0.5 mM CTAB (b) 2.5 mM CTAB.

The average size for 0.5 mM of CTAB and 2.5 mM is 6 nm with the standard deviation 6.9 and 6.7. When the concentration of CTAB is increase from 0.5 mM to 2.5 mM shows the distribution of particles change from narrow distribution to board distribution. Both molar concentration of CTAB showed the size of particles larger than range 100 nm. From the comparison for all results from FESEM and Image J software, the glucose shows can be considered as good reducing agents in order to control the shape and size of silver nanoparticles and to achieve narrow size

distribution. When the concentrations of reducing agents on the synthesis of silver nanoparticles increasing, the agglomeration of small particles also increases (Steve & Rong., 2012).

4.3 THE PURITY OF SILVER NANOPARTICLES

EDX is an analytical technique used for the elemental analysis or chemical characterization of a sample. The aim of this analysis is to confirm the presence of elemental silver without any further impurities in the prepared of silver nanoparticles. The EDX observations were carried out using magnitude X17000 for concentration 2.5 mM of glucose and X10000 for concentration 2.5 mM of CTAB as reducing agents in the synthesis of silver nanoparticles. The results of elemental silver peak for each element and the impurities for all the elements from the results are shown in Figure 4.8.

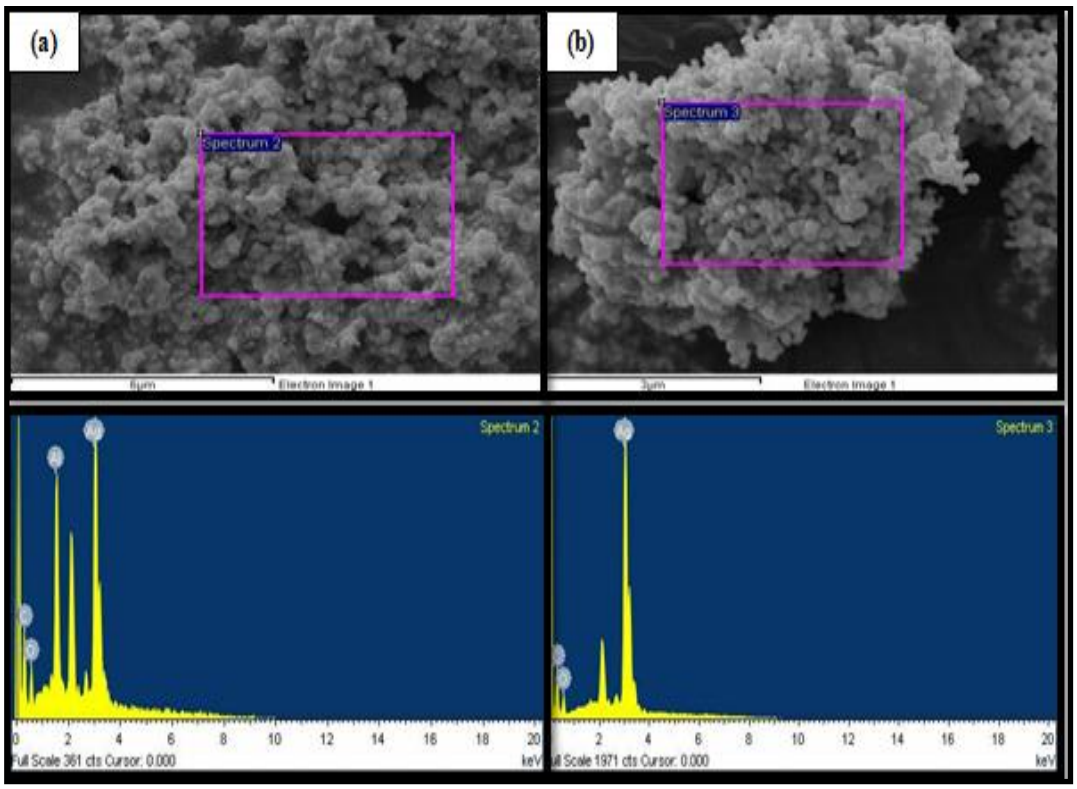

Figure 4.8: Typical EDAX spectrum (a) 2.5 mM CTAB (b) 2.5 mM Glucose

The results for 2.5 mM CTAB showed 77.78 % of silver, 9.44 % of aluminium, 7.38 % of oxygen and 5.40 % of carbon at the emission energy 17 keV. Meanwhile, Figure 4.7(b) also showed an EDX spectrum, emission energy at 10 keV for silver, and some of the elemental peak of Cl, C, and O were observed. The amount of Ag at 2.5 mM of glucose is 91.95 weight %, 5.00 % of oxygen and 3.05% of carbon. Through the EDX analysis, the percentages of atomic for each element also were showed in Table 4.1. The presence of element silver peak was confirming the reduction of silver and absence of the other impurities. The small carbon peak is absence due to the adhesion of carbon tape on the aluminium stud (Ajitha et al., 2013). From the results, the comparison of glucose and CTAB show the glucose is better than the CTAB due the impurities and the percentages of weight.

Table 4.1: Weight (%) and atomic (%) for each element

Element	2.5 mM CTAB		2.5 mM Glucose	
	Weight (%)	Atomic (%)	Weight (%)	Atomic (%)
C K	5.40	22.69	3.05	17.89
O K	7.38	23.26	5.00	22.03
AL K	9.44	17.66	0.0	0.0
AG K	77.78	36.39	91.95	60.09
Total	100.00		100.00	

4.4 STATISTICAL ANALYSIS

All experiment that was conducted was repeated triplicate for each concentration. Two ways ANOVA without replication was utilized to compare the optimum absorbance from different silver concentrations and *t* test was used to analyze the particles size distribution. The optimum absorbances for each concentration are shown in Table 4.2.

Table 4.2: Optimum Absorbance for Concentration of Glucose and CTAB

Concentration	Glucose	CTAB
	Absorbance	
0.0	0.194	0.194
0.5	0.423	0.648
1.0	0.448	0.741
1.5	0.45	0.831
2.0	0.481	0.813
2.5	0.428	0.811

The results from analyze the particles size distribution for glucose and CTAB are shown in Table A.1 and Table A.2. From the analysis, the p-value results for each concentration of glucose and CTAB is 0.03856 and p-value for different types of reducing agents is 0.006055. All the Samples were considered statistically significant because the p-value for these researches is lower than $p < 0.5$.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Silver nanoparticles were successfully prepared at different concentration of reducing agents with ascorbic acid as a stabilizer. UV-Vis absorption spectroscopy confirmed the formation of silver nanoparticles in the solutions. The UV-Vis absorption spectra showed the results for sample at optimum absorbance with the narrow distribution. The optimum concentration that gave highest absorbance when glucose is used as a reducing agent is at 2.0 mM meanwhile, the optimum concentration of CTAB is at 1.5 mM with 420 nm of wavelength.

However, with increasing of CTAB concentration, the aggregation was reduced, and the degree of dispersion improved considerably. The FESEM and EDX that were used to determine the shape and purities of particles – confirmed the spherical shape of the particles for the range of concentration. The glucose is considered as better reducing agents compared to CTAB because it can stable the size of silver nanoparticles. This was confirmed by using the Image J software of the FESEM image.

These results can be useful in preparing silver nanoparticles with narrow size distribution. As a conclusion, reducing agent which is glucose with ascorbic acid as surfactant gave uniform size distribution and shapes compared to concentration of CTAB.

5.2 RECOMMENDATIONS

There are some recommendations to improve the research on synthesis of silver nanoparticles as below:

i. Make sure the apparatus that need to use should wash and rinse with ionized water.

ii. The temperature and time for reduction of silver nanoparticles should be accurately so that the mixture is mixed completely

iii. The ice cube should be used and placed into beaker with the sample during ultrasonic cleanser to avoid the coagulation of sample.

iv. Make sure the samples are fully dry on the carbon tape at aluminium stud before the FESEM and EDX analysis to get the better image for particle morphology and size distribution.

v. Samples are recommended to be placed in the dark area or wrapped tightly the bottle samples with aluminium foil. It is to avoid the samples exposed with the light or sun.

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APPENDIX A

DATA POINT

Table A.1: t-Test (Two-Sample Assuming Equal Variances)

	Variable 1	*Variable 2*
Mean	0.404	0.673
Variance	0.011004	0.05966
Observations	6	6
Pooled Variance	0.035332	
Hypothesized Mean Difference	0	
df	10	
t Stat	-2.47874	
P(T<=t) one-tail	0.016307	
t Critical one-tail	1.812461	
P(T<=t) two-tail	0.032613	
t Critical two-tail	2.228139	

Table A.2: ANOVA (Two-Factor without Replication)

SUMMARY	*Count*	*Sum*	*Average*	*Variance*
Row 1	2	0.388	0.194	0
Row 2	2	1.071	0.5355	0.025313
Row 3	2	1.189	0.5945	0.042924
Row 4	2	1.281	0.6405	0.072581
Row 5	2	1.294	0.647	0.055112
Row 6	2	1.239	0.6195	0.073345
Column 1	6	2.424	0.404	0.011004
Column 2	6	4.038	0.673	0.05966

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Rows	0.301125	5	0.060225	5.769673	0.03856	5.05032
Columns	0.217083	1	0.217083	20.79698	0.006055	6.60789
Error	0.052191	5	0.010438			
Total	0.570399	11				

Table A.3: Summary of Absorbance for Different Molar Concentration of Glucose

Wavelength (nm)	Average Absorbance					
	Concentration of Glucose (mM)					
	0.0	0.5	1.0	1.5	2.0	2.5
300	0.116	0.157	0.199	0.198	0.190	0.183
320	0.061	0.066	0.092	0.093	0.083	0.086
340	0.125	0.145	0.180	0.179	0.173	0.172
360	0.155	0.227	0.271	0.265	0.269	0.253
380	0.176	0.316	0.354	0.344	0.365	0.330
400	0.191	0.393	0.431	0.421	0.451	0.404
420	0.194	0.423	0.448	0.450	0.481	0.412
440	0.191	0.422	0.441	0.444	0.473	0.428
460	0.188	0.407	0.426	0.425	0.452	0.409
480	0.184	0.390	0.407	0.408	0.429	0.387
500	0.179	0.372	0.388	0.388	0.407	0.366
520	0.175	0.355	0.370	0.370	0.387	0.347
540	0.170	0.339	0.353	0.352	0.367	0.330
560	0.167	0.322	0.337	0.335	0.349	0.313
580	0.163	0.307	0.321	0.317	0.331	0.298
600	0.162	0.292	0.306	0.303	0.315	0.284
620	0.160	0.277	0.291	0.287	0.299	0.271
640	0.160	0.263	0.277	0.273	0.283	0.258
660	0.160	0.249	0.264	0.259	0.269	0.246
680	0.160	0.236	0.251	0.246	0.255	0.235
700	0.160	0.223	0.239	0.233	0.241	0.225
720	0.160	0.211	0.227	0.222	0.228	0.216
740	0.161	0.200	0.216	0.211	0.215	0.207
760	0.161	0.188	0.205	0.199	0.202	0.197
780	0.161	0.178	0.196	0.189	0.190	0.189
800	0.161	0.168	0.187	0.179	0.179	0.180

Table A.4: Summary of Absorbance for Different Molar Concentration of Cetyl Trimethylammonium Bromide (CTAB)

Wavelength (nm)	Average Absorbance					
	Concentration of CTAB (mM)					
	0.0	0.5	1.0	1.5	2.0	2.5
300	0.116	0.279	0.330	0.436	0.537	0.614
320	0.061	0.119	0.180	0.275	0.412	0.511
340	0.125	0.248	0.303	0.364	0.472	0.557
360	0.155	0.416	0.465	0.520	0.577	0.634
380	0.176	0.547	0.588	0.630	0.648	0.674
400	0.191	0.637	0.704	0.760	0.751	0.750
420	0.194	0.648	0.741	0.821	0.800	0.799
440	0.191	0.638	0.735	0.831	0.813	0.811
460	0.188	0.626	0.713	0.812	0.797	0.793
480	0.184	0.608	0.680	0.772	0.759	0.750
500	0.179	0.588	0.642	0.719	0.710	0.690
520	0.175	0.566	0.604	0.665	0.657	0.626
540	0.170	0.540	0.569	0.612	0.600	0.560
560	0.167	0.513	0.534	0.561	0.544	0.497
580	0.163	0.486	0.505	0.517	0.492	0.436
600	0.162	0.458	0.477	0.478	0.443	0.384
620	0.160	0.433	0.454	0.446	0.398	0.334
640	0.160	0.410	0.434	0.420	0.358	0.292
660	0.160	0.390	0.417	0.399	0.325	0.256
680	0.160	0.372	0.400	0.380	0.297	0.227
700	0.160	0.356	0.382	0.362	0.274	0.203
720	0.160	0.340	0.364	0.344	0.252	0.183
740	0.161	0.325	0.345	0.326	0.232	0.164
760	0.161	0.309	0.325	0.304	0.213	0.147
780	0.161	0.294	0.307	0.283	0.194	0.132
800	0.161	0.279	0.288	0.262	0.175	0.117

APPENDIX B

GRAPH

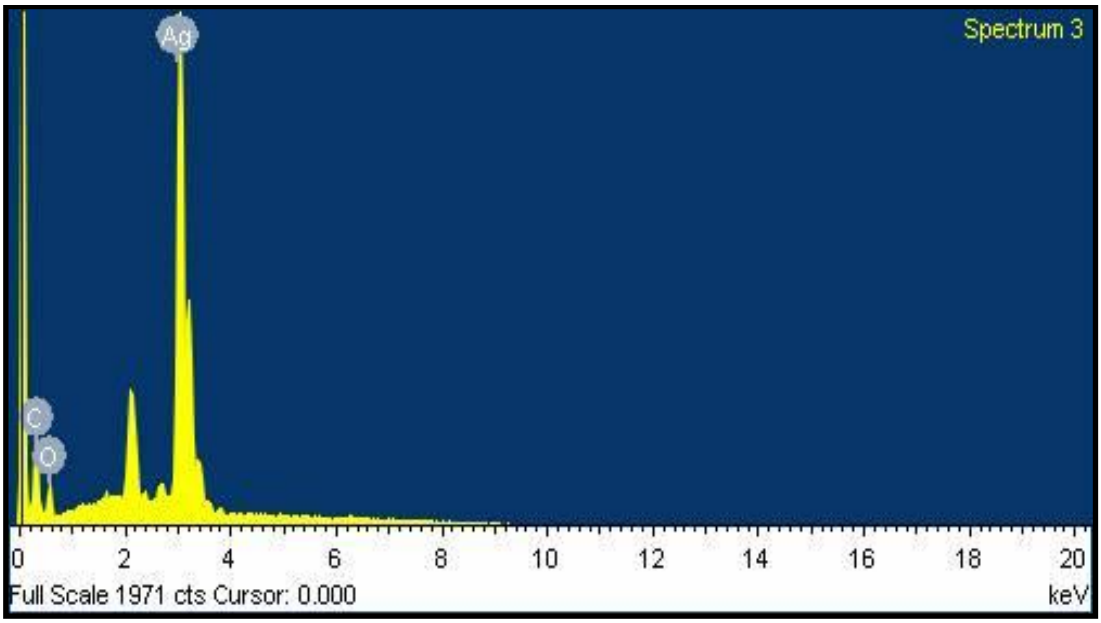

Figure B.1: Typical EDX Spectrum for 2.5 mM of Glucose

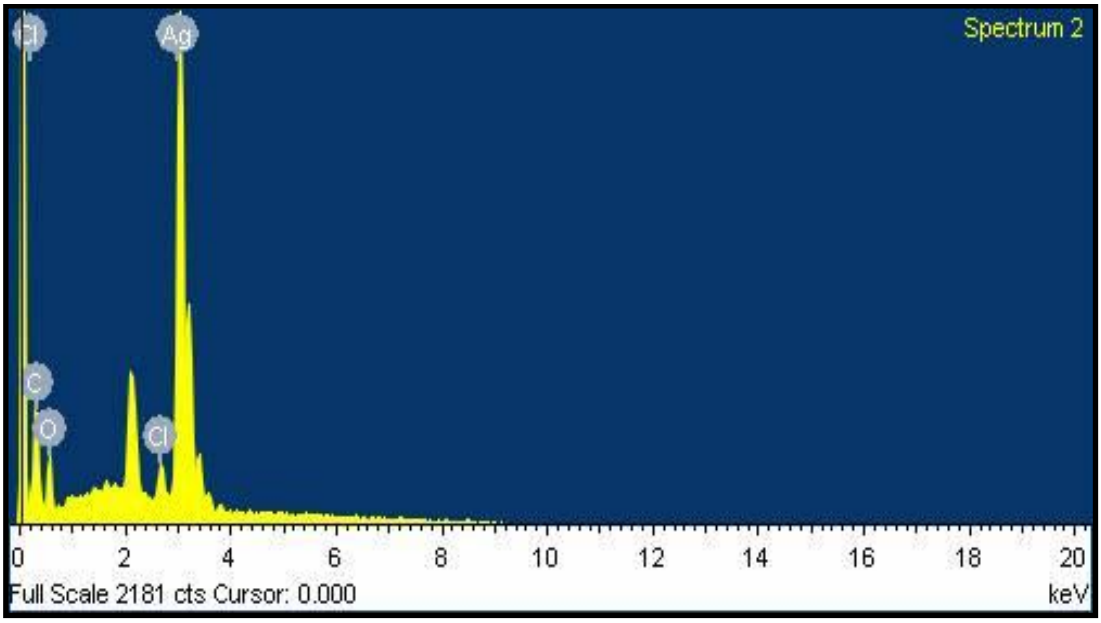

Figure B.2: Typical EDX Spectrum for 2.5 mM of CTAB

APPENDIX C

MATERIAL, APPARATUS AND EQUIPMENT

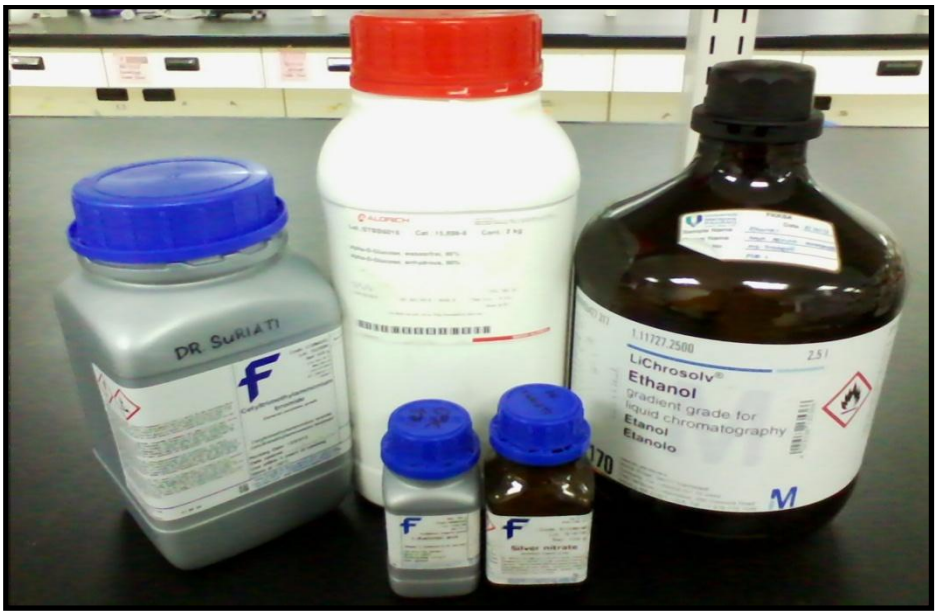

Figure C.1: Chemical Used In Synthesis of Silver Nanoparticles

Figure C.2: Ultrasonic Cleanser

Figure C.3: Weighing Machine

Figure C.4: Centrifuge Refrigerated

APPENDIX D

PRODUCT / SAMPLE

Figure D.1: Sample of Synthesis of Silver Nanoparticles from Molar Concentration of Glucose

Figure D.2: Sample of Synthesis of Silver Nanoparticles from Molar Concentration of CTAB

APPENDIX E

CHARACTERIZATION

Figure E.1: UV-Vis Absorption Spectroscopy (UV-Vis)

Figure E.2: Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray Analysis (EDX)