

Experimental Studies on Selected Nano-Particles Using Infrared Spectrometry Analysis

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ABSTRACT

The synthesis of nanoparticles can be classified basically into three (3) different methods which are: reduction of some chemical reagents, attrition and pyrolysis. In this research work the reduction method was employed where silver nanoparticles was synthesized and studied based on some certain criteria's. The synthesis of the silver nanoparticles was carried out in different days so as to have samples that will be different from one another and as well to have variation of life span. A freshly prepared silver nanoparticles is pale yellow in colour which gradually changes into yellow, violet and finally grey which confirms that the sample is no longer in nanoscale meaning it has aggregates. Aggregation is one of the major problems associated with nanoparticles and for that reason the features or certain properties of these nanoparticles are been studied using Fourier transform infrared (FT-IR). The FT-IR spectrum converts the vibration as the infrared ray passes through the sample from interferogram to a spectrum of absorbance and transmittance of different peaks. The spectrum gotten from the samples shows that silver nanoparticles has a wave number ranging from about 4000 to 400. The spectrum also shows that the values of the absorbance and transmittance of the samples decrease as the samples stays longer. Another property that was observed is that the bonds existing between the particles are mostly doubles and singles. The number of vibrational peaks within each and every spectrum reduces as the samples stays longer; the number of peaks present within a spectrum speaks for the amount of particles present in the sample.

KEYWORDS: Synthesis, Nanoparticles, IR Spectrometry, Fourier Transform, and Life Span

1.0 INTRODUCTION

Matter can be placed into broad categories according to size. Macroscopic matter is visible with the naked eye and microscopic ones are invisible. Atoms and (most) molecules are microscopic with dimensions less than 1nm. Mesoscopic particles, such as bacteria and cells that have dimensions on the order of micron(s), can be observed with optical microscopes. Falling into the gap between the microscopic and the mesoscopic is another class of matter, the nanoscopic particles, Poole and Owens (2003).

Nanotechnology deals with processes that take place on the nanometer (nm) scale, that is, from approximately 1 to 100 nm. Nanoscale materials possess a unique property which requires research studies. Advances are occurring in synthesis of isolated nanostructures because of its usefulness. This opens the possibility for creating a new generation of advanced materials with designed properties, not just by changing the chemical composition of the components, but by controlling the size and shape of the components. For example, the melting point of nanosized metal particles depends upon the size of the particles. The smaller a particle becomes, the more the proportion of surface atoms increases, which in turn increase the reactivities of the materials. As particles decrease in size the number of surface atoms

becomes equal to or even exceeds the number of inner-core atoms. For a typical bulk material the surface is negligibly small in comparison to the total volume. Surface atoms are more easily rearranged than those in the center of the particle, and so the melting process, which depends on destroying the order of the crystal lattice, which get started at a lower temperature. The melting point of gold metal is 1064°C. For 11-12 nm gold particles it is about 1000°C, then begins to drop dramatically to 900°C for 5 to 6 nm particles and to 700°C for 2 to 3 nm particles because of the decrease in the particle sizes.

In this research work, effort is made to synthesize nanoparticles using available silver materials as the possible materials available to us presently.

Nanoparticles have now clinched the heart of many researchers, the world manufacturer are busy making things look smaller and even faster in processing. Chips are no longer in micro scale but in nanoscale in the development. This work is target to see if at all from the grass root level nanoparticles can be produce and as to study it under infrared ray.

2.0 BACKGROUND LITERATURES

Although nanoparticles are generally considered a discovery of modern science, they actually have a very long history. Nanoparticles were used by artisans as far back as the 9th century in Mesopotamia for generating a glittering effect on the surface of pots (Hayashi et al., 1997). Even these days, pottery from the middle ages and renaissance often retains a distinct gold or copper colored metallic glitter. This luster is caused by a metallic film that was applied to the transparent surface of a glazing. The luster can still be visible if the film has resisted atmospheric oxidation and other weathering. The luster originated within the film itself, which contained silver and copper nanoparticles dispersed homogeneously in the glassy matrix of the ceramic glaze. These nanoparticles were created by the artisans by adding copper and silver salts and oxides together with vinegar, ochre and clay, on the surface of previously-glazed pottery. The object was then placed into a kiln and heated to about 600°C in a reducing atmosphere. In the heat the glaze would soften, causing the copper and silver ions to migrate into the outer layers of the glaze. There the reducing atmosphere reduced the ions back to metals, which then came together forming the nanoparticles that give the colour and optical effects (Hayashi, et al., 1997).

2.1 Origin of Nanoparticles

In nanotechnology, a particle is defined as a small object that behaves as a whole unit with respect to its transport and properties. Particles are further classified according to diameter. Coarse particles cover a range between 10,000 and 2,500 nanometers. Fine particles are sized between 2,500 and 100 nanometers. Ultrafine particles or nanoparticles are sized between 1 and 100 nanometers. The reason for this double name of the same object is that, during the 1970-80's, when the first thorough fundamental studies with 'nanoparticles' were underway in the USA (by Granqvist and Buhrman) and Japan, (within an ERATO Project) they were called 'ultrafine particles' (UFP). However, during the 1990s before the National Nanotechnology Initiative was launched in the USA, the new name, 'nanoparticle' had become fashionable. Nanoparticles may or may not exhibit size-related properties that differ significantly from those observed in fine particles or bulk materials. Although the size of most molecules would fit into the above outline, individual molecules are usually not referred to as nanoparticles (Panigrah et al., 2004).

2.2 Synthesis of Nanoparticles

There are several methods for creating nanoparticles, including reduction of some chemical reagents, attrition and pyrolysis. In attrition, macro or micro scale particles are ground in a ball mill, a planetary ball mill, or other size reducing mechanism. The resulting particles are air classified to recover nanoparticles. In pyrolysis, a vaporous precursor (liquid or gas) is forced through an orifice at high pressure and burned. The resulting solid (a version of soot) is air classified to recover oxide particles from by-product gases. Pyrolysis often results in aggregates and agglomerates rather than single primary particles. But here the nanoparticles were synthesized using the reduction method (Solomon et al., 2007).

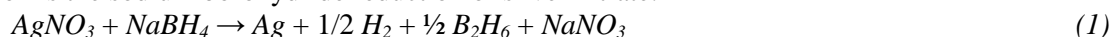
2.3 Reduction Method of Synthesizing Nanoparticles

Here basically three different nanoparticles can be synthesized which are the gold, silver and copper nanoparticle. But in the laboratory only one out of the three were studied due to unavailability of some reagent. Silver nanoparticles has been synthesis while gold and copper nanoparticles are been exempted due to the non availability of reagents not available.

2.4 Synthesis of Silver Nanoparticles and Protocol

2.4.1 Silver Nanoparticles Synthesis

Yellow colloidal silver has been reported upon reaction with ice-cold sodium borohydride and is the basis for the method used in this work described next. The determination of an optimal set of conditions for the synthesis of silver nanoparticles is described in the sections that follow. An excess of sodium borohydride is needed both to reduce the ionic silver and to stabilize the silver nanoparticles that form. The chemical reaction is the sodium borohydride reduction of silver nitrate:



2.4.2 Protocol

Reactions were done in clean dry 125-mL Erlenmeyer flasks. A 10-mL volume of 1.0 mM silver nitrate was added drop-wise (about 1 drop/second) to 30mL of 2.0 mM sodium borohydride solution that had been chilled in an ice-bath. The reaction mixture was stirred vigorously on a magnetic stir plate. The solution turned light yellow after the addition of 2 mL of silver nitrate and a brighter yellow when all of the silver nitrate had been added. The entire addition took about three minutes, after which the stirring was stopped and the stir bar removed. The silver nanoparticle is stable at room temperature stored in a transparent vial for as long as several weeks or months. Upon aggregation the colloidal silver solution turns darker yellow, violet and finally gray as aggregation proceeds (Solomon et al., 2007).

2.5 Application of Silver and Gold Nanoparticles

Compared with organic dyes the absorption of light by gold and silver nanoparticles is 5–6 orders in magnitude higher. This implies that a metal nanoparticle is over 1 million fold more likely than the comparable dye to absorb light, such as near-Infrared (NIR) light and convert it into thermal energy. As a result, these nanoparticles have recently been used in thermal therapy and optical imaging of tumors. Silver and gold nanostructures have been studied as important colorimetric sensors due to their high extinction coefficient. The sensitivity of surface plasmon peaks to environmental changes can lead to optical detection of binding materials on the silver surface. The sensitivity of such an optical sensor is strongly dependent on the size, shape, structure of the nanoparticles, and the surrounding medium. For example, when the refractive index of the surrounding medium changes, the intensity and position of the surface plasmon peak changes. An increase in the refractive index often causes the surface plasmon peak to shift to the longer wavelength. The red shift of the surface plasmon peak can also be caused by the aggregation of the particles and has been used for detection of glucose and protein. One example is use of boronic acid-capped silver NPs for detection and monitoring of blood sugar. The interactions of boronic acid-capped silver NPs and glucose resulted in aggregation of the nanoparticles and were accompanied by a shift in the plasmon peak of silver NPs from 397 nm to 640 nm (Yonzon et al., 2005).

The ability of gold and silver nanostructures to enhance local electric fields has led to their applications as Surface Enhanced Raman Scattering (SERS). The enhancement factor can be as much as 10¹⁴-10¹⁵, which enables the detection of individual molecules absorbed on metal particles. The maximum signal amplification in SER spectroscopy (10¹⁵) can be achieved with silver particles of about 50 nm (Curry et al., 2006).

Silver has been known for antibacterial activity since the ancient Greece times. Currently, the investigation of this phenomenon has gained more attention due to the increase of bacterial resistance to antibiotics, caused by their overuse. Silver nanoparticles can be used in medicine to reduce infections in burn treatment, to prevent bacteria colonization on dental materials, stainless steel materials, to eliminate microorganisms on textile fabrics, or they can be used for water treatment. Antimicrobial activity of colloid silver particles is influenced by the dimensions of the particles. The extremely small size of

nanoparticles results in the particles having a large surface area relative to their volume. This allows them to easily interact with other particles and increases their antibacterial efficiency (Pearce et al., 2007).

In heterogeneous catalysis, it is well known that the increasing proportion of surface atoms with decreasing particle size makes small metal particles highly reactive. Silver has been utilized as an oxidation catalyst especially for production of ethylene oxide from ethylene.

3.0 MATERIALS AND METHODS

3.1 Materials

During the characterization of the nanoparticles the following materials are used; (i) Sample of nanoparticle, (ii) Fourier transmitter infrared spectrometry (FT-IR), (iii) a computer system and (iv) printer.

3.2 Methodology

The process of characterizing the sample of nanoparticle was carried out in the following way;

The cover of the FT-IR spectrometry was open to bring out the cuvette which was rinse with distilled water, about 1.5ml of a sample was poured into the cuvette, a cover was used to cover it and then the cuvette was place in its holder in the FT-IR spectrometry.

The scanning process was conducted using the computer system which has an application that is been used to characterize such samples. While scanning the sample a platform for naming the sample will pop out, this allows one to name the sample with the name of his choice.

After the scanning is over a chromatograph of transmitters and frequency is displayed which is printed using the printer. On the chromatograph display on the window one can switch the nature of the graph from that of transmitters against frequency to absorbance against frequency which can also be printed out. The same procedure was followed for the samples only that the naming differs in order to distinguish them from one another.



Figure 2 Colloidal silver in various stages of aggregation label a to d from left to right, a) clear yellow sol, b) dark yellow, c) violet, and d) grayish, as aggregation proceeds.

3.3 Function of the FT-IR Spectrometry

Fourier Transform Infrared (FT-IR) spectrometry was developed in order to overcome the limitations encountered with dispersive instruments. The main difficulty was the slow scanning process. A method for measuring all of the infrared frequencies simultaneously, rather than individually, was needed. A solution was developed which employed a very simple optical device called an interferometer. The interferometer produces a unique type of signal which has all of the infrared frequencies 'encoded' into it. The signal can be measured very quickly, usually on the order of one second or so. Thus, the time element per sample is reduced to a matter of a few seconds rather than several minutes (Pearce et al., 2007).

Most interferometers employ a beam splitter which takes the incoming infrared beam and divides it into two optical beams. One beam reflects off of a flat mirror which is fixed in place. The other beam reflects off of a flat mirror which is on a mechanism which allows this mirror to move a very short distance (typically a few millimeters) away from the beamsplitter. The two beams reflect off of their respective mirrors and are recombined when they meet back at the beamsplitter. Because the path that one beam travels is a fixed length and the other is constantly changing as its mirror moves, the signal which exits the interferometer is the result of these two beams ‘interfering’ with each other. The resulting signal is called an interferogram which has the unique property that every data point (a function of the moving mirror position) which makes up the signal has information about every infrared frequency which comes from the source. This means that as the interferogram is measured; all frequencies are being measured simultaneously. Thus, the use of the interferometer results in extremely fast measurements. Because the analyst requires a frequency spectrum (a plot of the intensity at each individual frequency) in order to make identification, the measured interferogram signal cannot be interpreted directly. A means of ‘decoding’ the individual frequencies is required (Zhu et al., 2005). This can be accomplished via a well-known mathematical technique called the Fourier transformation. This transformation is performed by the computer which then presents the user with the desired spectral information for analysis.

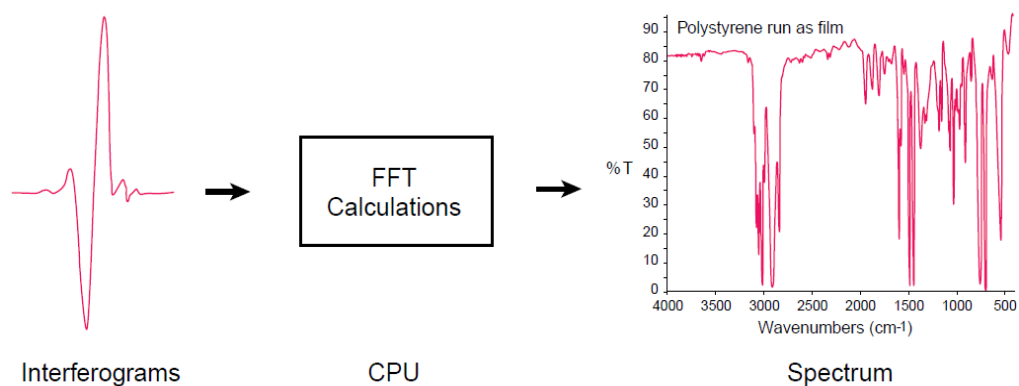


Figure 3 The above picture shows a path way through which a computer converts the interferograms using the FFT calculations in to spectrum NTC (2001).



Figure 4.1 (a) The Fourier transforms Infrared spectrometry

4.0 RESULTS

The results of the research work and discussion of the findings were presented in this chapter. The sample of freshly prepared silver nanoparticle was characterized immediately after synthesis we will be used as reference to the other samples which where characterize after certain days of their synthesis. Another reference chart from literature is also been used to check for the types of bonds existing within the samples.

4.1 Absorption Bands

The diagram below shows a reference diagram to be use in determining the nature of bonds existing between the components that make up the various samples.

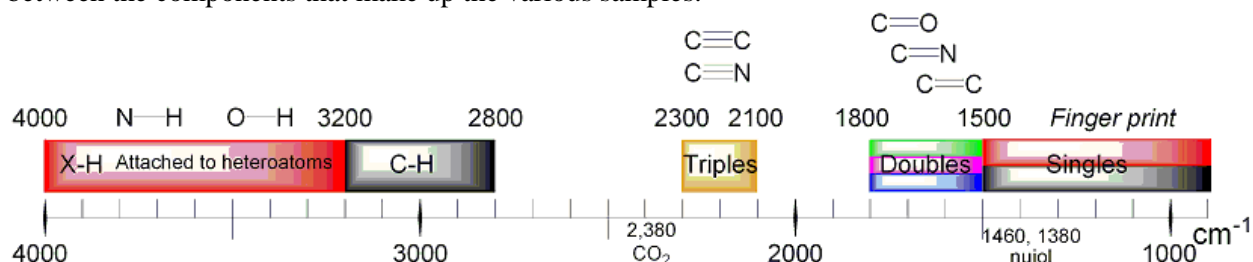


Figure 4.1(b) Absorption band showing the possible bonds at various wave number

The chromatographs of transmittance and absorbance spectrum obtain after characterizing the samples are shown below:

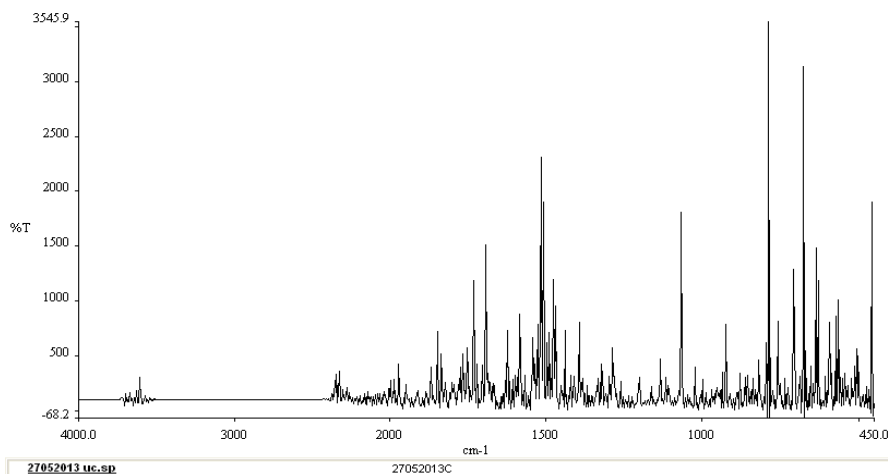


Figure 4.2.1(a) Chromatograph of T against F of freshly prepared silver nanoparticles of Day 2

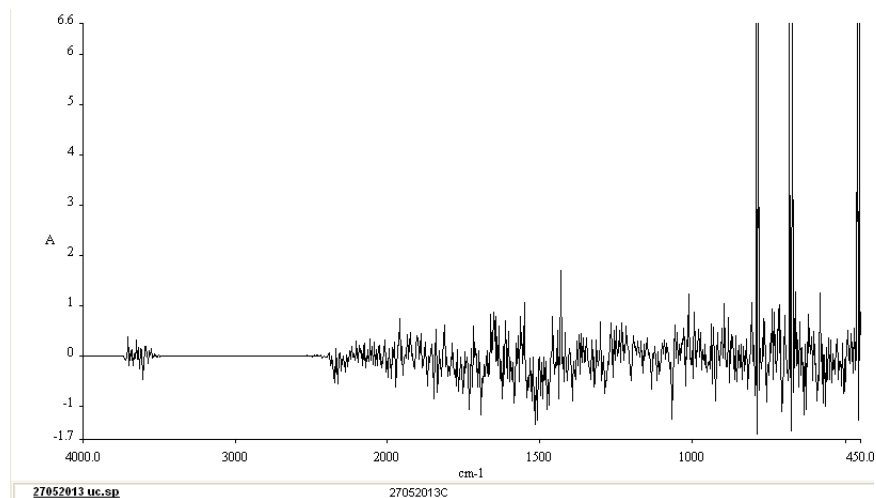


Figure 4.2.1(b) Chromatograph of A against F of freshly prepared silver nanoparticles of Day 2

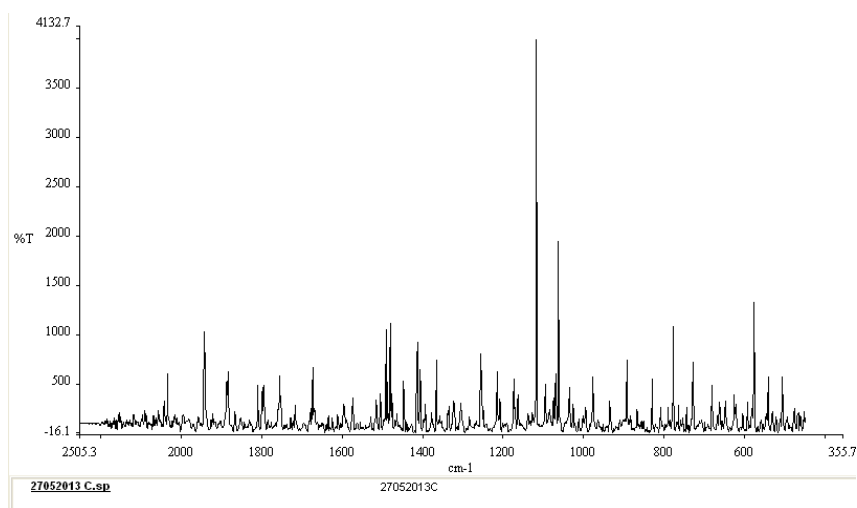


Figure 4.2.2(a) Chromatograph of T against F of freshly prepared silver nanoparticles using different method of synthesis

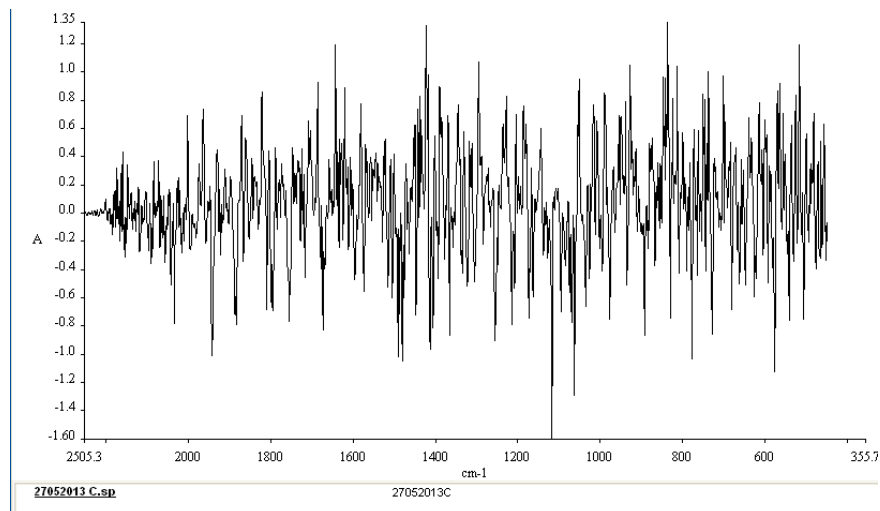


Figure 4.2.2(b) Chromatogram of T against F of freshly prepared silver nanoparticles using different method of synthesis

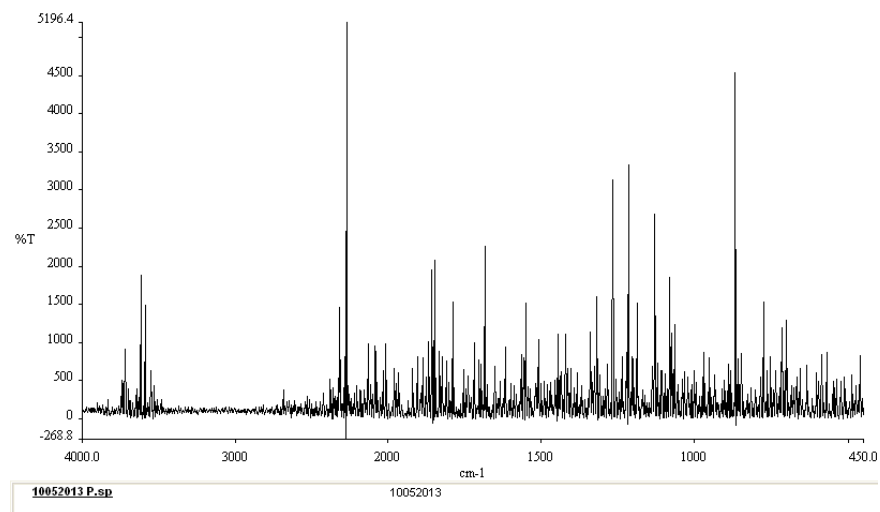


Figure 4.2.3(a) Chromatogram of T against F of freshly prepared silver nanoparticles of Day 1

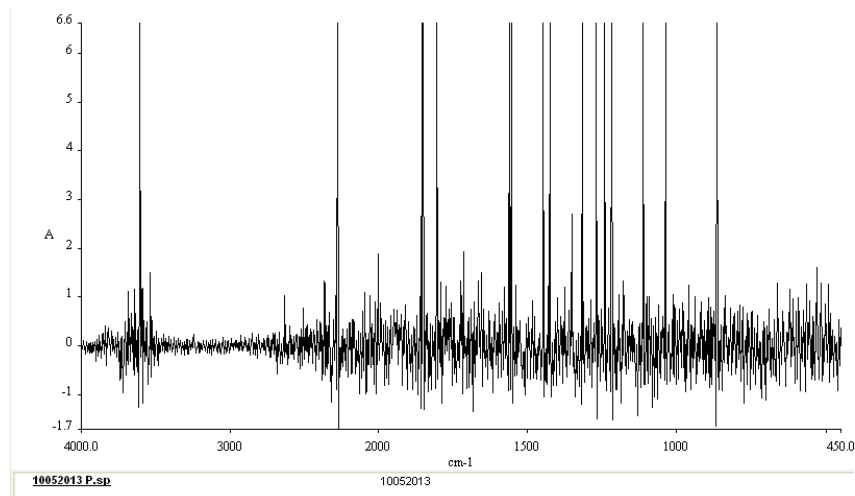


Figure 4.2.3(b) Chromatogram of A against F of freshly prepared silver nanoparticles of Day 1

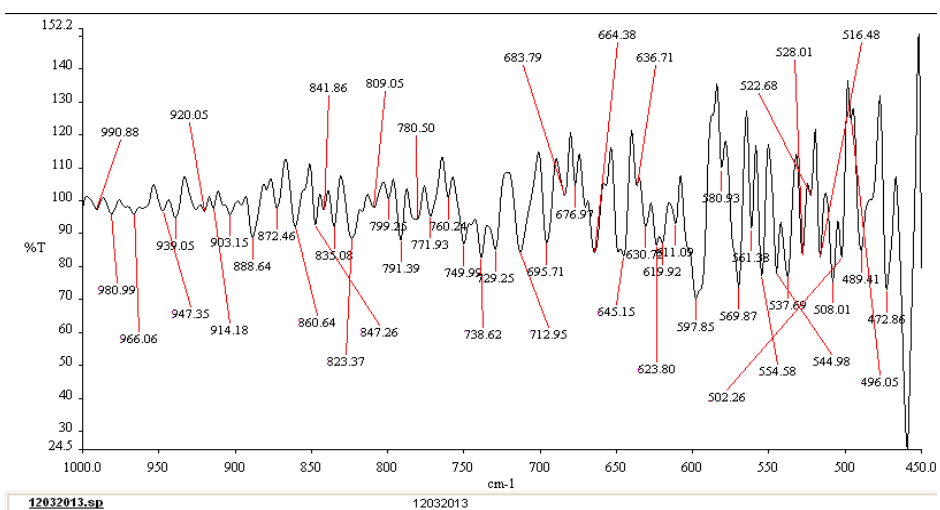


Figure 4.2.4(a) Chromatogram of T against F of 3 weeks prepared silver nanoparticles

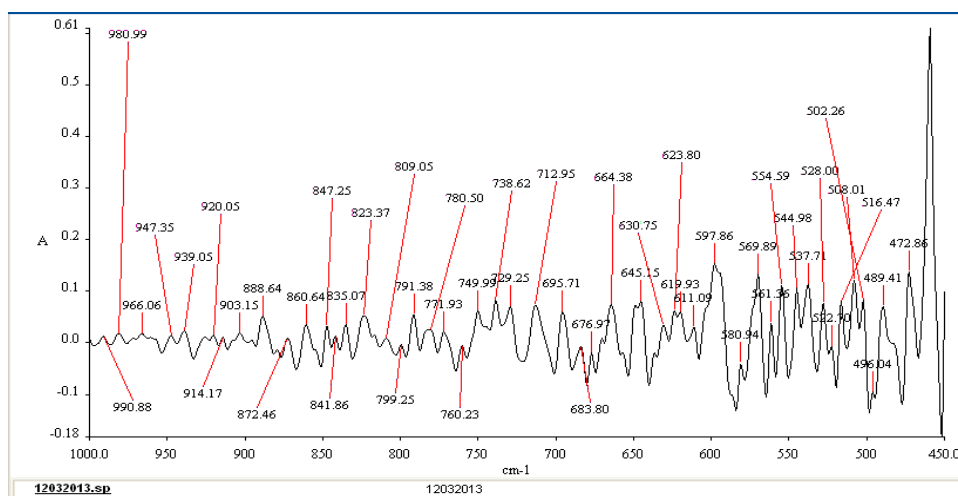


Figure 4.2.4(b) Chromatogram of A against F of 3 weeks prepared silver nanoparticles

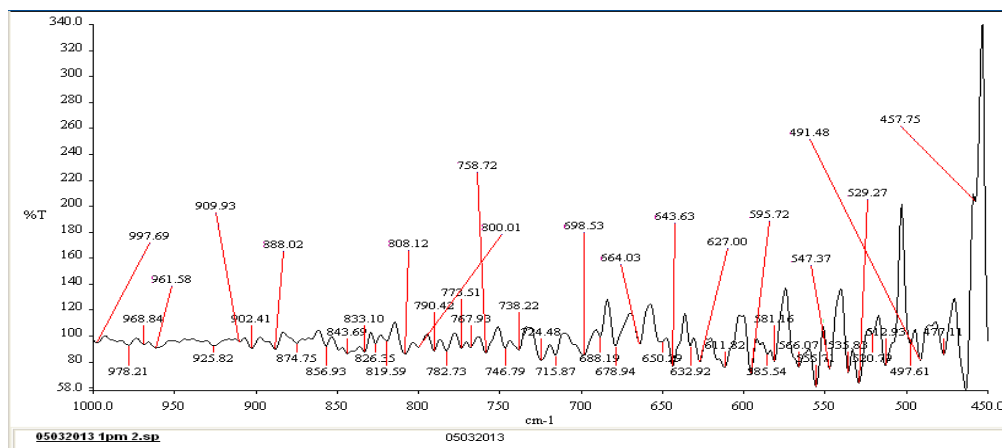


Figure 4.2.5(a) Chromatograph of T against F of 5 weeks prepared silver nanoparticles

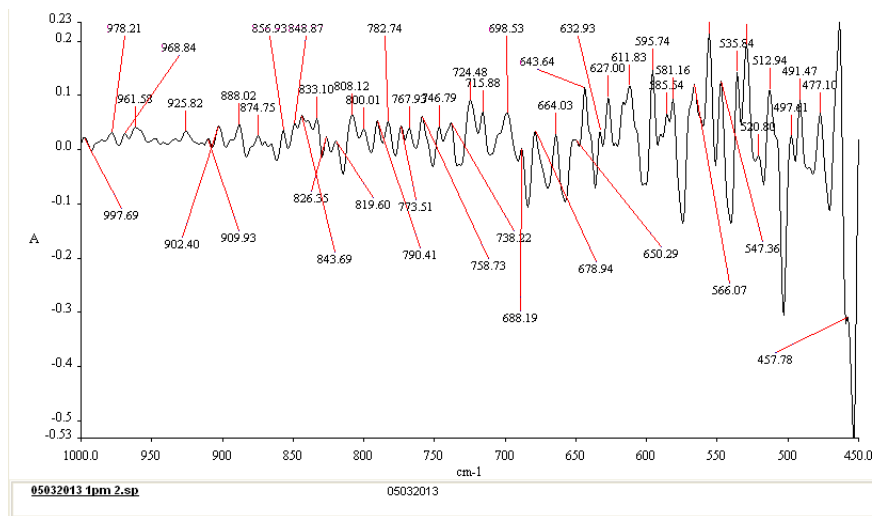


Figure 4.2.5(a) Chromatograph of T against F of 5 weeks prepared silver nanoparticles

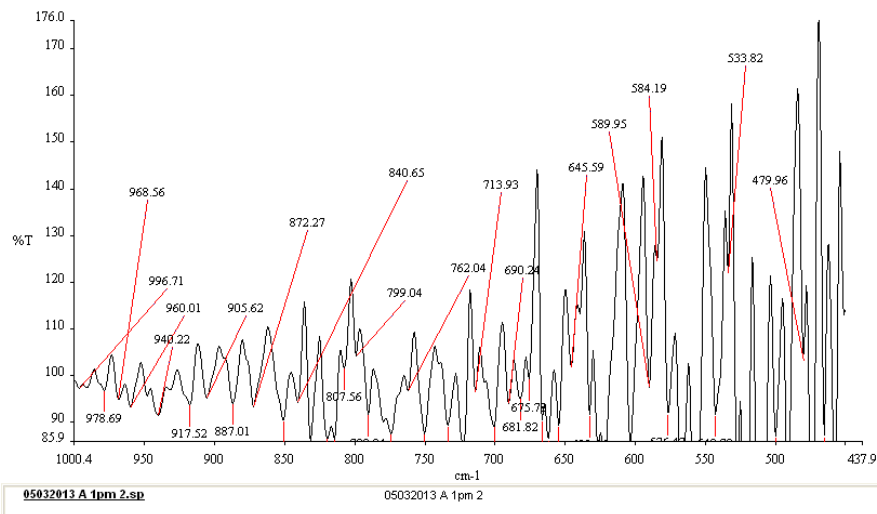


Figure 4.2.6(b) Chromatograph of A against F of an aggregated silver nanoparticle

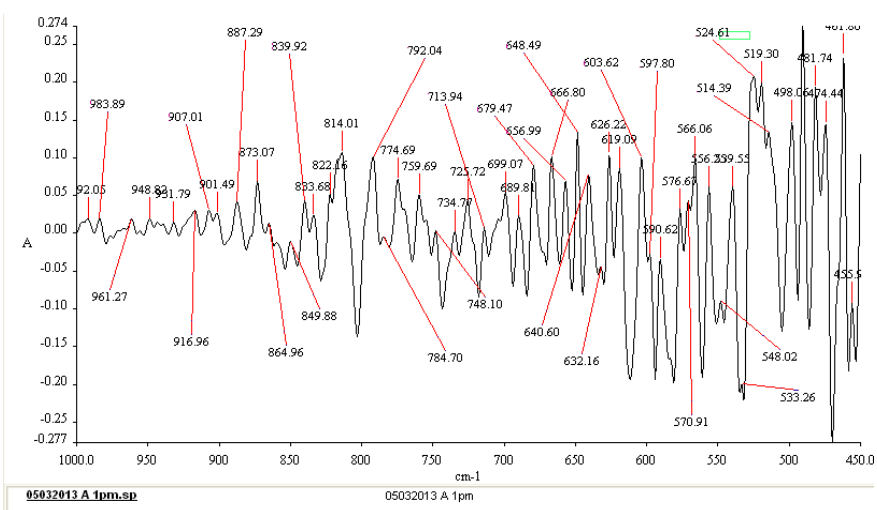


Figure 4.2.6(b) Chromatograph of A against F of an aggregated silver nanoparticle

5.0 DISCUSSIONS OF RESULTS

The findings of the characterization is discussed here in details, each and every sample is analyzed here. The freshly prepared and characterize sample of day 2 is used as reference to the other samples for comparison. Another chat gotten from literature is also used to evaluate the nature of bonds possible to be available within the sample is also been used.

5.1.1 Freshly Prepared and Characterized Sample of Day 2

From the graphs of figure 4.2.1a and 4.2.1b it can be seen that the sample has a vibration at wave number $4000\text{-}450\text{cm}^{-1}$ but the vibration is more prominent from $2500\text{-}450$ for both the absorbance and transmittance graph NTC (2001), Cioffi et al., 2005. The major goal of any absorption spectrum is to measure how well a sample absorbs light at each wavelength and the graph has shown that this sample absorb very well from wave number of $2500\text{-}450\text{cm}^{-1}$ and at a frequency of $4\mu\text{m}$ to $22.2\mu\text{m}$ The

absorption spectrum indicates the IR radiation absorbed by the sample whereas does that could not be absorbed made up the transmittance spectrum. Absorption is the resonant frequency, which is the frequency of the absorbed radiation matches the transition energy of the bond or group that vibrates as observed with Cioffi (2005). The more the number of peaks in the spectrum the more the material present in the sample and it can be seen clearly that the two spectrums have much numbers of peaks. The transmittance spectrum graph shows that the sample has a maximum value of 3545.97% transmittance and a minimum value of - 68.2% transmittance, whereas the absorbance graph has a maximum absorbance at 1.35 and a minimum of - 1.60 absorbance. Whereas for the type of bonds likely to be present are;

1. X-H (attached to heteroatom) because we had vibration at wave number between 4700-4600 cm^{-1} .
2. Triple bonds due to the vibrations between 2300-21000 cm^{-1} .
3. Doubles and singles (finger print) which is likely to be the most part of the sample because much vibration is present within the wave number of 1800 to less than 1000 cm^{-1} .

5.1.2 Freshly Prepared and Characterized Sample Using Different Method of Synthesis

From the graphs of figure 4.2.2a and figure 4.2.2b it can be seen that the sample has vibration at wave number 2505.3-355.7 cm^{-1} for both the absorbance and transmittance graph. Even though the sample was prepared using different method it has shown that it still has many peaks which indicated that there are many materials present but not up to the first one¹¹.

The transmittance spectrum graph shows that the sample has a maximum value of 4132.7% transmittance and a minimum value of -16.1% transmittance whereas the absorbance graph has a maximum absorbance at 1.35 and a minimum of -1.60 absorbance. Whereas for the type of bonds likely to be present are;

1. Triple bonds due to the vibrations between 2300-21000 cm^{-1}
2. Doubles and singles (finger print) which is likely to be the most part of the sample because much vibrations are present within those wave number of 1800 to less than 1000 cm^{-1} .

5.1.3 Freshly Prepared and Characterized Sample of Day 1

This sample was characterized immediately after synthesis it has almost similar qualities with the sample of day 2 as it can be seen on figure 4.2.3a and figure 4.2.3b, only that the nature of peaks and their positions on the spectrum differs. This sample has a maximum value of 5196.4% transmittance and a minimum value of -268.8% transmittance whereas for the absorbance spectrum it has a maximum value of 6.6 absorbance and -1.7 absorbance. The size of peaks present in both the transmittance and absorbance spectrum has indicates the present of large number of materials. About the nature of possible bonds present is also same with the sample in day 2 that is X-H, doubles and finger prints.

5.1.4 Sample Characterized after 3 Weeks of Synthesis

Clear differences can be seen between the freshly prepared and characterized with those that are characterized days after the synthesis whose graph are shown in figure 4.2.4a and figure 4.2.4b. Vibrations are only present within the wave number of 1000-450 cm^{-1} for both the absorbance and transmittance spectrum, which is equivalent to the frequency of 10 μm to 22.2 μm . the number and size of the peaks are relatively low when compared with the fresh once. The maximum transmittance value of this spectrum is 152.2% transmittance while the minimum value is 24.5% transmittance which means no negative transmittance value whereas in the case of the absorbance spectrum the maximum value is 0.61 of absorbance and the minimum is -0.18 of absorbance^{10, 11}. The size and number of the peaks has shown that the number of materials in the sample is low which indicated that the particle has start aggregating with each other forming bigger particles and therefore their number decreases. The fact that all the vibrations are within the wave number of 1000-450 cm^{-1} indicates that the only bond likely to be present is the single bond or the finger prints.

5.1.5 Sample Characterized after 5 Weeks of Synthesis

Although both the spectrums begins from 1000 to 450cm⁻¹ but prominent peaks are only notice at around 700 to 450cm⁻¹ as it can be seen in figure 4.2.5a and figure 4.2.5b. The nature, size and numbers of the peaks has decline again far lesser than those characterized after 3weeks of synthesis. The transmittance and absorbance value has also drop to 340 to 58% of transmittance and 0.23 to -0.53 of absorbance which was in thousands for the transmittance and units for the absorbance Capatina (2005), Klabunde (2001).

. The amount of materials (particle) present in this sample is far low due to low number of peaks present and this is a clear indication that the particles have bonded to each other forming larger particles that are no more in nanoscale, in short the sample has almost aggregate only that the colour has not indicate that the sample has aggregate completely.

Here too the only possible bonds that could be found are the single bonds (finger) prints anything apart from that could not be found within the sample Klabunde (2001), Mathew (2012).

5.1.6 Sample Characterized after it has Aggregates

This sample is almost similar with the previous one in terms of their peaks size, number and nature, the only difference is the absorbance and transmittance value which has decline to 176-85.9% of transmittance and 0.274 of absorbance as it can be seen in figure 4.2.6a and figure 4.2.6b. The numbers of peaks are not much which indicates that the number of particles present is low and with the colour of the sample changing to violet is a conclusion that the sample has aggregated. In terms of possible bonds that could be found here is still the single bond or finger print^{10, 11, 12}.

5.2 Summary of Results

From the discussion above one can clearly deduce that the fresh samples have much number of peaks which indicates that the particles present is also much, the size of the peaks as well because the maximum transmittance values of fresh samples were in thousands and the minimum values were in negative values, however the aggregated or almost aggregated samples are in hundred for their maximum values and positive tens for their minimum values. In the case of the absorbance the fresh one's has maximum values in tens and minimum values in negatives tens whereas the aggregated or almost aggregated one's has their for the maximum in positive decimal while the minimum in negative decimals.

In the case of possible bonds that are likely to be found in the sample, for the fresh samples about 2 or 3 difference bonds are likely to be gotten while for the aggregated or almost aggregated samples only 1 bond is likely to be found Capatina (2005), Klabunde (2001), Mathew (2012)

6.0 CONCLUSIONS

In conclusion on this research work, I would like to say that synthesizing nanoparticle is possible in a small scale and it doesn't cause much. Having the nanoparticles at hand will create much room for more research to be carried out on them. Using the FT-IR to characterize the nanoparticles has shown some of the properties of the nanoparticles in terms of wave number, maximum and minimum absorbance and transmittance, nature of bonds likely to be found and the amount of vibrational peaks in the spectrum. As stated before that aggregation is one of the major problem of these nanoparticles, it can be seen clearly from the result and discussion that aggregation has effect on absorbance, transmittance, number of peaks and even the nature of bonds likely to be found within the sample. From physical observation, the physical transformation that the sample undergoes from pale yellow down to grey which indicates aggregation has been seen on the synthesized sample and that has confirm to me that nanoparticles do aggregates and a solution should be found for that.

7.0 Recommendation

The recommendations are as follows:

1. Since it has been observed that bacteria's are now proving to be resistant to antibacterial drugs and that silver nanoparticle can substitute that, other researchers should carry a comparative study

between the anti bacterial drugs and the silver nanoparticle and see which one is most effective in fighting bacteria's taking price and health effect that the silver nanoparticles can cause into considerations.

2. Nanotechnology is one of the fast and growing aspect of technology now where many larger materials are been converted into smaller chips which are even more secured than the larger ones e.g. micro chips. I would like to recommend that engineers should work tirelessly in this field so that we can have nano-chips which am sure will be secured that the micro one's.
3. The department laboratory is seriously in need of help because many chemicals and equipment are not available. The availabilities of the necessary equipment and chemicals will encourage the student to work harder in their research work as they don't have to go round the school, metropolis or even outside the state looking for materials for their research work.
4. Any researcher conducting research on silver nanoparticle should be very conscious of safety because silver nitrate (AgNO_3) is a harmful chemical which can even burn one's skin on contact.
5. More research work should be carried out on the reduction method of synthesizing nanoparticles in order to bring a lasting solution to quick aggregation or even to eradicate the aggregation problem entirely.

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