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Qualitative Synthensisation of Nanoparticles Using FTIR Spectroscopic Analysis

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Abstract

In this work, FTIR spectrometry was used to analyse the spectra configurations on the synthesised silver nanoparticles were synthesized using two different reducing agents (1) sodium borohydride ($NaBH_4$), at two temperature conditions of 27^0C and 4^0C (room temperature and ice cold temperature), and (2) trisodium citrate (3NaCi) at 100^0C (boiling temperature) by chemical reduction method. The objective of this work is to compare the results of Ag nanoparticles synthesised, using sodium borohydride as reducing agent, at room temperature and after precooling in an ice bath. Secondly, to comparing the results of the synthesized Ag nanoparticles using two different reducing agents and thirdly, to observe the effect of IR spectrophotometer on the nanoparticles in terms of wavelength (m) and absorbance (A) and (transmittance, T). The resulting nano-sized colloids have been characterized by FTIR spectroscopy. A clear and yellow colouration was obtained from all the synthesised samples, which indicates the presence of formed nanoparticles with strong absorption peaks obtained at wave length range of 1370 - 1380 cm^{-1} and 840 - 841 cm^{-1} proving the presence of silver nanoparticles based on the functional group. The elements found to be present at such peak were the nitro and N-O functional groups when compared with the reported literature. The results shows that Ag nanoparticle colloid was formed using sodium borohydride irrespective of temperature, but the synthesis performed at lower temperature yielded more stable colloids.

Keywords: *synthesis, FT-IR,* Nanoparticle, reducing agent and functional group

INTRODUCTION

Nanotechnology is one of the most important targets of science and technology. Nanotechnology is the formation of 100 nm sized particles and provides significant changed features in different materials. Nanoparticles are building blocks of nanotechnology and are referred to as a natural, incidental or manufactured material containing particles, in an unbound state or as an aggregate or as an agglomerate and where, for 50% or more of the particles in the number size distribution, one or more external dimensions is in the size range of 1 nm – 100 nm (European, 2011). Natural nanoparticles have been present in the environment for millions of years and they have been generated by natural processes including weathering, erosion, volcanic eruption, hydrolysis and biological activities. Recently, however, several sources have resulted in an increase in anthropogenic nanoparticles formation (Partim and Chang-Yu, 2005): among the different activities that contributed to the augmentation of the nanoparticles in the environment are: coal fired combustions, transportation's, welding processes followed by more recent processes where engineered nanoparticles are designed and produced deliberately.

Engineered nanoparticles are the backbone of modern nanotechnology where research and development are growing very fast and attract substantial funding both from public and private sectors (Joner et al., 2008). Particles in the nanometre-size (10^{-9} m) range have gained much attention due to their fascinating electrical, optical, magnetic and catalytic properties associated with their Nano scale dimension's (Christof, 2001). Those fascinating, unique and novel properties make nanomaterial's physicochemical difference and often superior to both the atomic and bulk material of the same element. For instance, Gold which is rarely insoluble in water at the macro scale, becomes more soluble in the Nano scale (Paolo Pengo, 2003), a material such as platinium, which is chemically inert at normal scales, can serve as a potent chemical catalyst at Nano scales (Luo et al., 2005, Tian et al., 2007). They have many applications varying from communications to catalyst, computing chips, Nano mechanical parts, photo sensors, novel platform for specific delivery of therapeutic agents, cosmetics and anti-aging drugs and they are attracted by a wide range of scientific researches in different fields of science (Ferrari, 2005, Templeton et al., 1999).

The problem statement is that, with the increase advancement in technology today nanoparticles have drawn special attention of scientists due to their unique properties. Nanotechnology has been given less attention in Nigeria so this research is determined to synthesis, characterise and show the importance of nanoparticles along with their uses. The achievement in this study is on the chemical synthesis method of nanoparticles to produce mono-dispersed spherical nanoparticles suspended in water was used in these studies which differ from the previous literatures.

RESEARCH BACKGROUND

Although, in general, nanoparticles are considered a discovery of modern science, they actually have a very long history. Nanoparticles were used by artisans as far back as the ninth century in Mesopotamia for generating a glittering effect on the surface of pots (Reiss Gunter; Huntten Andreas, 2010. Khan Firdos Alam, 2012). Even these days, potteries from the middle ages and renaissance often retain a distinct gold- or copper-coloured metallic glitter. This lustre is caused by a metallic film that was applied to the transparent surface of a glaze. The lustre can still be visible if the film has resisted atmospheric oxidation and other weathering (Reiss Gunter; Huntten Andreas, 2010. Khan Firdos Alam, 2012). The lustre 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 salt and oxides together with vinegar, ocher, 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 (Khan Firdos Alam, 2012).

In the heat the glaze would soften, causing the copper and silver ions to migrate into the outer layers of the glaze. There the atmosphere reduced the ions back to metals, which then came together forming the nanoparticles that gave the colour and optical effects (Khan Firdos Alam, 2012).

Luster technique showed that ancient craftsmen had a rather sophisticated empirical knowledge of materials. The technique originated in the Muslim world. As Muslims were not allowed to use gold in artistic representations, they sought a way to create a similar effect without using real gold. The solution they found was using luster (Khan Firdos Alam, 2012, Rawson Philip S. 1984).

Michael Faraday provided the first description, in scientific terms, of the optical properties of nanometer-scale metals in his classic 1857 paper. In a subsequent paper, the author (Turner) points out that: "It is well known that when thin leaves of gold or silver are mounted upon glass and heated to a temperature that is well below a red heat (~500 °C), a remarkable change of properties takes place, whereby the continuity of the metallic film is destroyed. The result is that white light is now freely transmitted, reflection is correspondingly diminished, while the electrical resistivity is enormously increased" (Faraday Michael, 1857. Beilby G.T., 1903. Turner T., 1908).

Therefore, nanotechnology is the science that deals with matter at the scale of 1 billionth of a meter (i.e., $10 - 9$ m = 1 nm), and is also the study of manipulating matter at the atomic and molecular scale. A nanoparticle is the most fundamental component in the fabrication of a nanostructure, and is far smaller than the world of everyday objects that are described by Newton's laws of motion, but bigger than an atom or a simple molecule that is governed by quantum mechanics.

Nanoparticles (NPs) originated from different sources and can be grouped into natural and anthropogenic (man-made) which, in turn, can be subdivided into accidental and engineered or manufactured nanoparticles or they can be classified further based on chemical composition, into organic (carbon containing) and inorganic (Nowack and Bucheli, 2007). Of particular importance, the optical property is one of the fundamental attractions and a characteristic of a nanoparticle. For example, a 20-nm gold nanoparticle has a characteristic wine red colour. A silver nanoparticle is yellowish gray. Platinum and palladium nanoparticles are black.

CLASSIFICATIONS AND TYPES OF NANOPARTICLES

A summary representation of the various classes and types of nanoparticles is shown in table 1.

NATURAL NANOPARTICLES

Natural nanoparticles (NPs) are found throughout the atmosphere, oceans, and soil systems, terrestrial water systems (groundwater and surface water), and on most living organisms both at the micro and macro levels which have been present in the environment like forever (Theng and yuan, 2008, Hochella et al., 2008). They have been formed mainly by the effect of naturally occurring physical, chemical and biological processes such as hydrolysis, erosion, weathering, volcanic eruption, sea spray, photochemical reactions, growth nuclei in super-saturated fluids, plants roots on rocks, minerals and microrganisms. Forest fires, as volcanic eruptions, for instance, can spread ash and smoke over thousands of miles and lead to an increase in the particulate matters, including NPs, exceeding ambient air quality standards (Sapkota et al., 2005).

In many environments, biogenetic nanoparticles are formed directly by microorganisms to fulfil metabolic requirements (Suzuki et al., 2002, Schüler and

Frankel, 1999) or as an indirect result of microbial activity (Glasauer et al., 2002, Hansel et al., 2004, Banfield et al., 2000). Dominant phases of the natural NPs include: Iron oxides/ hydroxides, aluminium oxides/hydroxides, clay minerals (hydrated aluminosilicates of K, Mg, and Fe etc.) and silica. Biological activities also assemble following the bottom up process, a wide range of carbon-containing NPs including humic substances, bacteria, viruses, cells and their organelles. Though air pollution is mostly associated with human activities such as transportation, industry, and charcoal burning, natural events such as dust storms, volcanic eruptions and forest fires can produce vast quantities of nanoparticulate matter in the atmosphere that affect air quality worldwide (buzea et al., 2007). So the exposure of humans and other living organisms to nanoscale particles is not as recent as the new field of nanotechnology.

Table 1 Classification of nanoparticles

Type	**Chemical composition**	**Nano-formation**	**Formation**	**Examples**
Natural	Carbon-containing	Biogenic	Organic colloids	Humic, fulvic acids
			Organisms	Viruses
		Geogenic	Soot	Fullerenes
		Atmospheric	Aerosols	Organic acids
		Pyrogenic	Soot	CNT
				Fullerenes
				Nanoglobules, onion-shaped nanosheres
	Inorganic	Biogenic	Oxides	Magnetite
			Metals	Ag, Au
		Geogenic	Oxides	Fe-oxides
			Clays	Allophane
		Atmospheric	Aerosols	Sea salt
Anthropogenic (manufactured, engineered)	Carbon-containing	By-product	Combustion by-products	CNT
				Nanoglobules, onion-shaped nanosheres
		Engineered	Soot	Carbon Black
				Fullerenes
				Functionalized CNT, fullerenes
			Polymeric NP	Polyethyleneglycol (PEG) NP
	Inorganic	By-product	Combustion by-products	Platinum group metals
		Engineered	Oxides	TiO_2, SiO_2
			Metals	Ag, iron
			Salts	Metals-phosphates
			Aluminosilicates	Zeolites, clays, ceramics.

Source: (*Nowack and Bucheli, 2007*)

ACCIDENTAL NANOPARTICLES

The activities of human beings have released different forms of nanoparticulate matter for millennia as by-products of some activities which were essential for their survival such as agriculture, construction, food cooking, mining and mineral processing. However, accidental NPs input to the environment has risen sharply and dramatically, since the beginning of the industrial revolution in the mid-18th century and after, due to manufacturing emissions, nuclear waste generation and the combustion of fossil fuels (Wiesner et al., 2009). Diesel and automobile exhausts are the primary source of atmospheric nano-and micro-particles in urban areas. Since their productions are spontaneous and uncontrolled accidental nanoparticles are more likely to be poly dispersed/heterogeneous and have irregular shapes. They contain sulphide, sulphate, nitrate, ammonium, organic carbon, elemental carbon and trace metals (Sioutas et al., 2005)

ENGINEERED NANOPARTICLES

The modern nanotechnology exploits the novel nanoscale properties of nanoparticles which has led to the production of a vast amount of engineered NPs. Engineered NPs are deliberately manufactured by human activities to serve special purposes and they are different from both incidental NPs that are produced as a side product of human activity, for example, from industrial processes or from transport and from natural NPs. Engineered NPs can be divided into a number of classes and not as a single homogeneous group. Based on their core materials, these manufactured nano-materials can be classified into organic and inorganic. Organic NPs can be further defined as fullerenes (C60 and C70 and derivatives) and carbon nanotubes (multi-walled or single walled CNTs), while inorganic NPs can be subdivided into metal oxides (of iron, zinc, titanium, cerium etc.), metals (mainly silver and gold) and quantum dots (such as cadmiumselenides) (Ju-Nam and Lead, 2008).

Fullerenes and Carbon Nanotubes

- **Fullerenes**, also called Bukminsterfullerene, are spherical cages composed of 60, 70 or 80 carbon atoms, which are bound to three other atoms in sp^2 hybridation and each atom has the remaining electron of the four outer shell electrons free. Unlike diamond (which is another allotrope of carbon), fullerenes can conduct electricity due to free electrons on its surface and in this respect has similar properties to graphite. Although fullerenes can be found spontaneously in the nature as by-products of combustion reactions, it was first synthesized in 1985 (Kroto et al., 1985). Since then, many processes for the production of fullerenes of different sizes have been developed including arcing of graphite, combustion of hydrocarbons, thermal and non-thermal plasma pyrolysis of coals and hydrocarbons and thermal decomposition of hydrocarbons (Huczko and Byszewski, 1998).

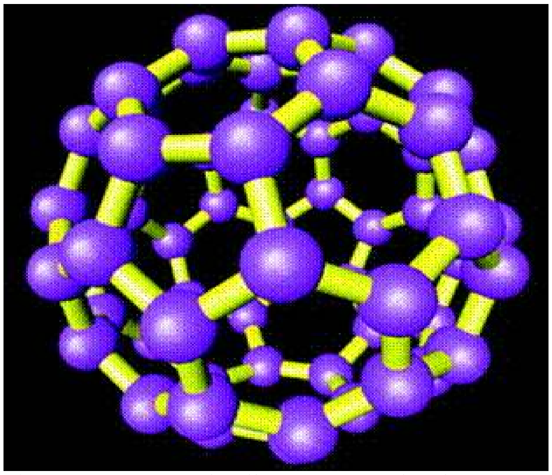


Figure 2 Structure of Fullerene C60 molecules. Purple balls represent the places of carbon atoms (Buseck, 2002).

- Fullerenes have many applications, including lubrication, superconductors, semiconductors, photoconductors, optical limiters and atom encapsulation. Since fullerenes are empty structures with dimensions similar to several biologically active molecules, they can be filled with different substances and find medical applications. These include anti HIV-protease activity, photodynamic DNA cleavage, free radical scavenger, antimicrobial action and use of fullerenes as diagnostic agents (Mehta and Thakral, 2006, 2001).
- **Carbon nanotubes** are similar to fullerenes in structure and compositions but they have elongated shapes with 1-2 nm in diameter. Using an arc-discharge evaporation method similar to that used for fullerene synthesis, they were first produced in 1991 (Iijima, 1991). Normally, carbon nanotubes are made of one sheet of graphite folded to form cylindrical single walled carbon nanotubes (SWCNT) (Iijima and Ichihashi, 1993) although multi walled carbon nanotubes (MWCN) (Iijima, 1991) can be formed by folding more than one sheet of graphite.

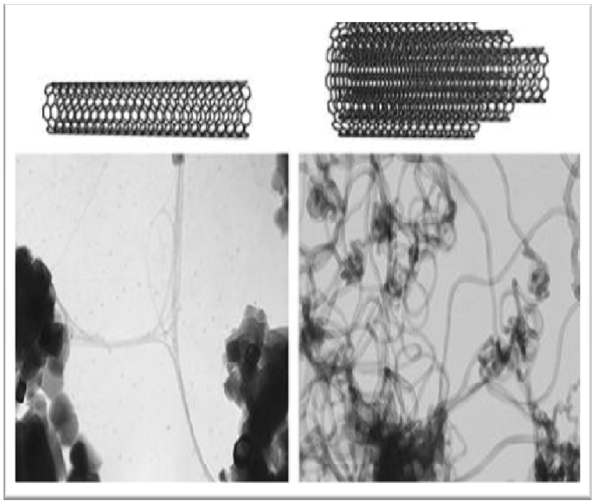


Figure 3: Representation of SWCNT and MWCN at the top and their TEM images at the bottom (Donaldson et al., 2006). Reprinted with permission from copyright 2006 Oxford University Press.

At present, the three main methods employed for CNT synthesis are arc-discharge, laser ablation, and chemical vapour deposition (CVD) (Trojanowicz, 2006). Carbon nanotubes are light, chemically stable, have high strength, high aspect ratio (long length compared to a small diameter) and remarkable optical properties (Hou et al., 2002, Tersoff and Ruoff, 1994) and, because of these useful properties, they become ideal material for many applications. Nanotubes have attracted a considerable amount of interest in the past few decades due to their potential application in large numbers of academic and industrial areas for diverse application possibilities (Cao and Rogers, 2009) ranging from nanoscale circuits to voltage devices (Philip Wong, 2005), to light-emitting devices (Freutag et al., 2006), thermal heat sinks (Kordás et al., 2007), electrical interconnects (Close et al., 2008), chemical/biological sensors (Kim et al., 2007b), ultra-strong fibres (Baughman et al., 2002), high-power electrochemical capacitors, (Niu et al., 1997) gas storage components (Gadd et al., 1997, Rakhi et al., 2008), magnetic data storage devise and drug delivery systems (Bianco and Prato, 2003).

RESEARCH METHODOLOGY AND INSTRUMENTATION

Instrumentation - IR Spectroscopy

Infrared spectroscopy (IR spectroscopy) is the spectroscopy that deals with the infrared region of the electromagnetic spectrum, which is light with a longer wavelength and lower frequency than visible light. It covers a range of techniques, mostly based on absorption spectroscopy. As with all spectroscopic techniques, it can be used to identify and study chemicals. For a given sample which may be solid, liquid, or gaseous, the method or technique of infrared spectroscopy uses an instrument called infrared spectrometer to produce an infrared spectrum. A basic IR spectrum is a graph of infrared light absorbance (or transmittance) on the vertical axis vs. frequency or wavelength on horizontal axis. A common laboratory instrument uses this technique in a Fourier transform infrared (FTIR) spectrometer. The synthesised colloidal silver was studied using an FTIR spectrometer

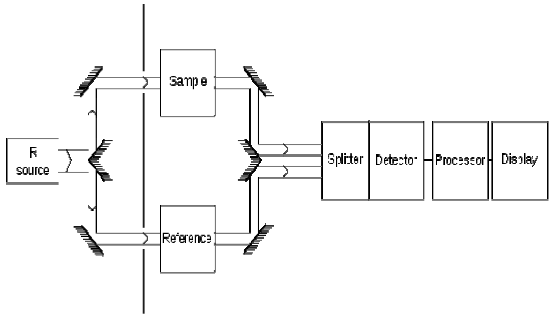


Figure 4 schematic of a two beam absorption spectrometer.

METHODOLOGY

The Chemical Synthesis of Silver Nanoparticles

Many chemical reduction methods have been used to synthesize silver nanoparticles from silver salt. Two main synthesis methods were adopted in this work to synthesize silver nanoparticles, namely the sodium borohydride and trisodium citrate reduction of silver nitrate. These were chosen primarily because of their possibility to produce particles of different size regimes (Bonsak, 2010), but also because of their synthesis simplicity requiring simple laboratory setups and including few reactants. The experiment was carried out under different temperature conditions to study the behaviour of these nanoparticles.

Chemicals

- Silver nitrate ($AgNO_3$, M = 169.87 g/mol)
- Trisodium citrate ($Na_3C_6O_7.2H_2O$, M = 294.10 g/mol)
- Sodium borohydride ($NaBH_4$, M = 37.83 g/mol)

Reduction of Silver Nitrate using Sodium Borohydride

The silver nitrate reduction can be written as:

$$AgNO_3 + NaBH_4 \rightarrow Ag + \tfrac{1}{2} H_2 + \tfrac{1}{2} B_2H_6 + NaNO_3 \qquad (1)$$

The expected particles size are in the region 7-15nm in diameter (Bonsak, 2010), great care has been taken during the synthesis to obtain colloidal silver, especially when dealing with a strong reducing agent such as sodium borohydride. Proper cleaning of laboratory equipment in advance is crucial, and the silver nitrate solution must by no means be exposed to the aqueous sodium borohydride except during controlled addition.

20.5 mg of $AgNO_3$ was dissolved in a 200 ml distilled water to prepare a 0.6 mM aqueous silver nitrate solution. Also 27.24 mg of $NaBH_4$ was dissolved in 600 ml distilled water to form a 1.2 mM aqueous solution of sodium borohydride. The compounds dissolved quickly both at room temperature and in ice cold water. The concentration of the sodium borohydride solution was twice that of the silver nitrate to ensure colloid stability, as suggested by Solomon et. al.

The reaction was done in a 250 ml reaction vessel. 7 mL aqueous silver nitrate contained in a pipette was added drop wise, using an electronic pipette aid, to 25 mL aqueous solution of sodium borohydride. The solution was stirred vigorously on a magnetic stir plate throughout the whole reaction and stopped immediately dropping was complete (magnetic stirring instrument speed = 600 rpm). The same procedure was repeated for different ratios of silver nitrate solution : sodium borohydride solution of 10:25, 5:25, 2.5:25. Two experiments were performed,

- First was done at room temperature, without precooling the reactants
- Second was done after precooling reactants in an ice-bath for about 15 minutes.

Reduction of Silver Nitrate using Trisodium Citrate

90 mg of silver nitrate was dissolved in 500 mL of distilled water to give 1.06 mM aqueous concentration of silver nitrate and 1 g of trisodium citrate (3NaCi) in 100 ml distilled water to form a 38 mM concentration of aqueous 3Naci. In the first run a conical flask containing 50 mL silver nitrate solution was placed directly on top of the magnetic stirrer which serve both as a heater and a stirrer, the sample was heated up to boiling temperature. 25 mL of trisodium citrate solution was then added drop-wise to the solution using a handheld electronic pipette aid as a dropper. Vigorous stirring and boiling was maintain throughout the whole process and post

addition stirring and boiling was done for 20 minutes before the sample was collected. The same was repeated for solution ratios of silver nitrate : trisodium citrate of 50:35 and 50:45. The reaction mechanism is as follows:

$$4Ag^{+} + C_6H_5O_7Na_3 + 2H_2O \rightarrow 4Ag^{0} + C_6H_5O_7H_3 + 3Na^{+} + H^{+} + O_2^{+} \quad (2)$$

Figure 5 some selected pictures from the laboratory experiment

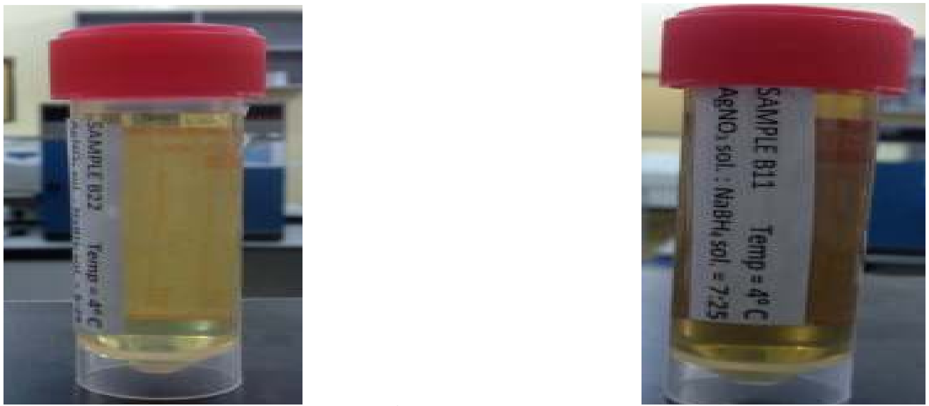
EXPERIMENTAL RESULTS

Figure 6 freshly prepared silver nanoparticles at room temperature using sodium borohydride as reducing agent and using different concentrations ratio

Figure 7 Image of freshly prepared Silver nanoparticles using sodium borohydride as reducing agent.

Sample B33: reaction temperature of 4^{0}C
Solution ratio of $AgNO_3$: $NaBH_4$ of 10:25

Sample B44: reaction temperature of 4^{0}C
Solution ratio of $AgNO_3$: $NaBH_4$ of 2.5:25



Sample B22: reaction temperature of 4^{0}C
Solution ratio of $AgNO_3$: $NaBH_4$ of 5:25

Sample B11: reaction temperature of 4^{0}C
Solution ratio of $AgNO_3$: $NaBH_4$ of 7:25

Figure 8 Freshly prepared nanoparticles using sodium borohydride as reducing agent at ice cold temperature (4^{0}C)

Sample C1 ($AgNO_3$ sol : 3NaCi sol = 50:25), Sample C2 ($AgNO_3$ sol : 3NaCi sol = 50:35), Sample C3 ($AgNO_3$ sol : 3NaCi sol = 50:45)
Figure 9 Silver nanoparticles prepared using trisodium citrate as reducing agent

FTIR ANALYSYS

Fourier Transform Infrared Spectroscopy (FTIR) is carried out with prepared samples since it is a useful technique to study structure and structural transformation.

FTIR spectrum taken over the range 450 cm^{-1} - 4000 cm^{-1} of silver nano-colloids is as shown below:

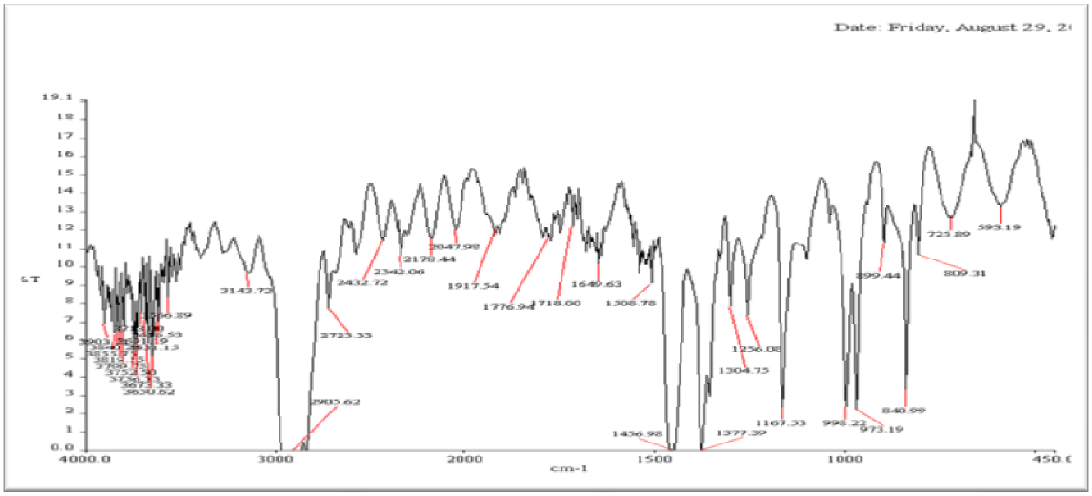


Figure 10 Sample A11 IR Spectrum

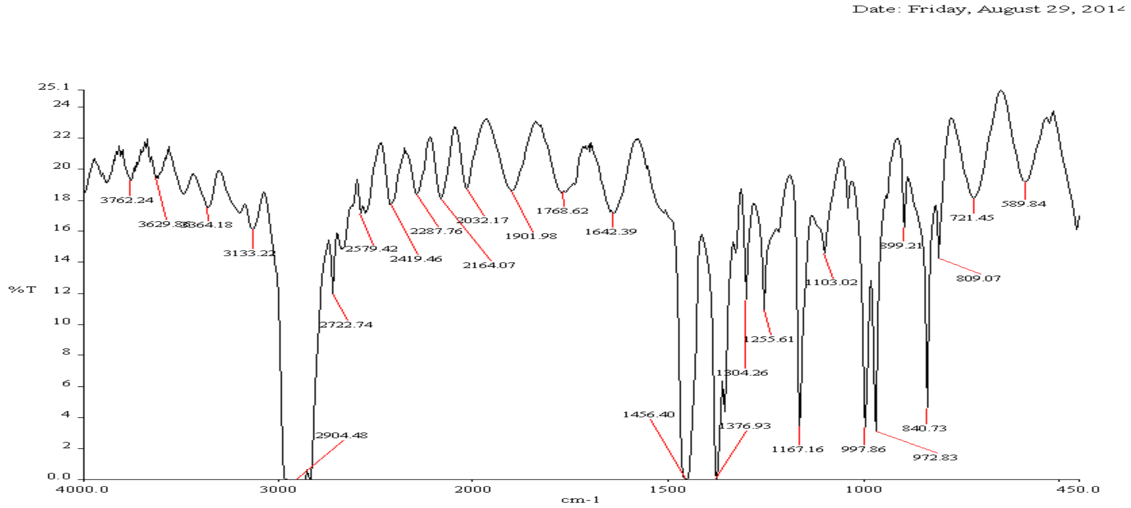


Figure 11 Sample A22 IR Spectrum

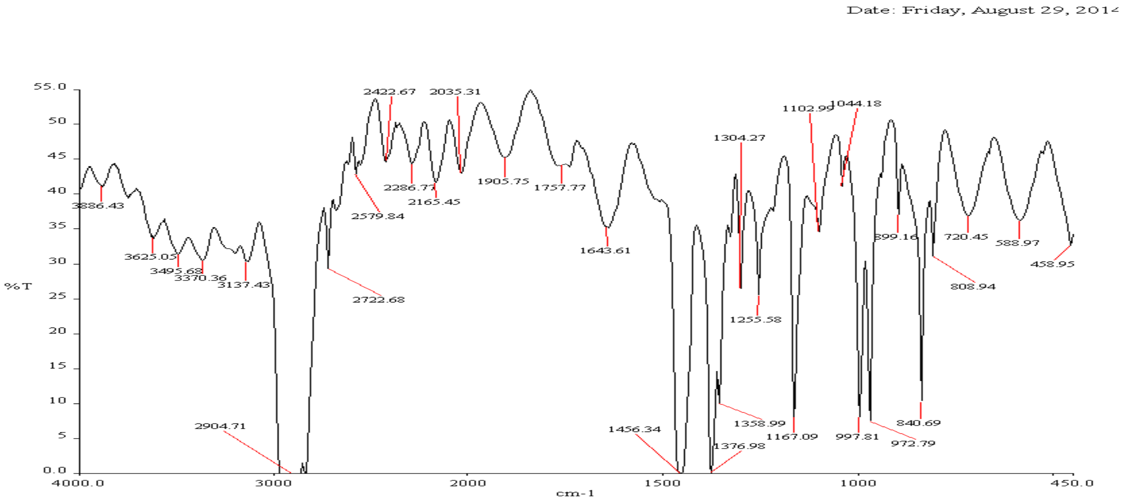


Figure 12 Sample A33 IR Spectrum

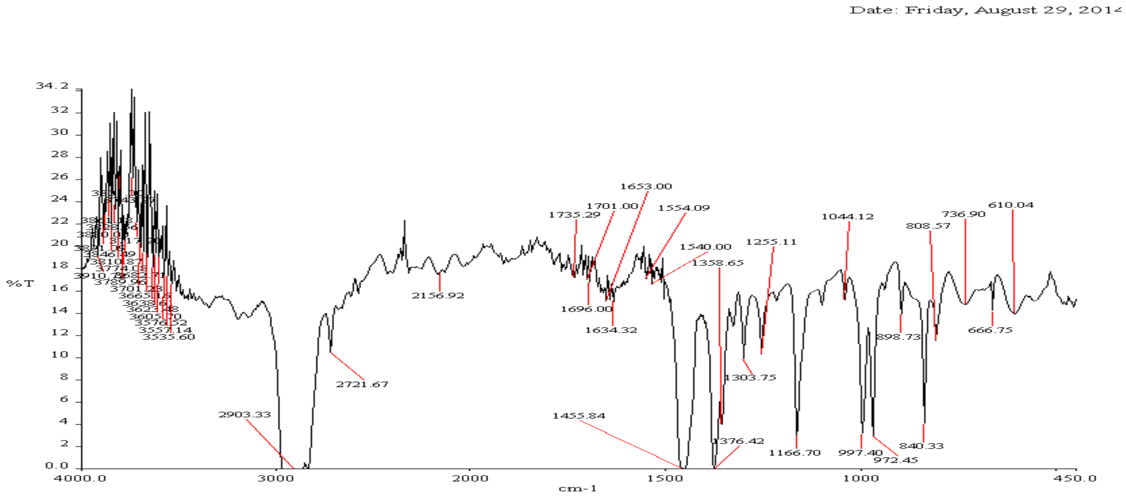


Figure 13: Sample A44 IR Spectrum

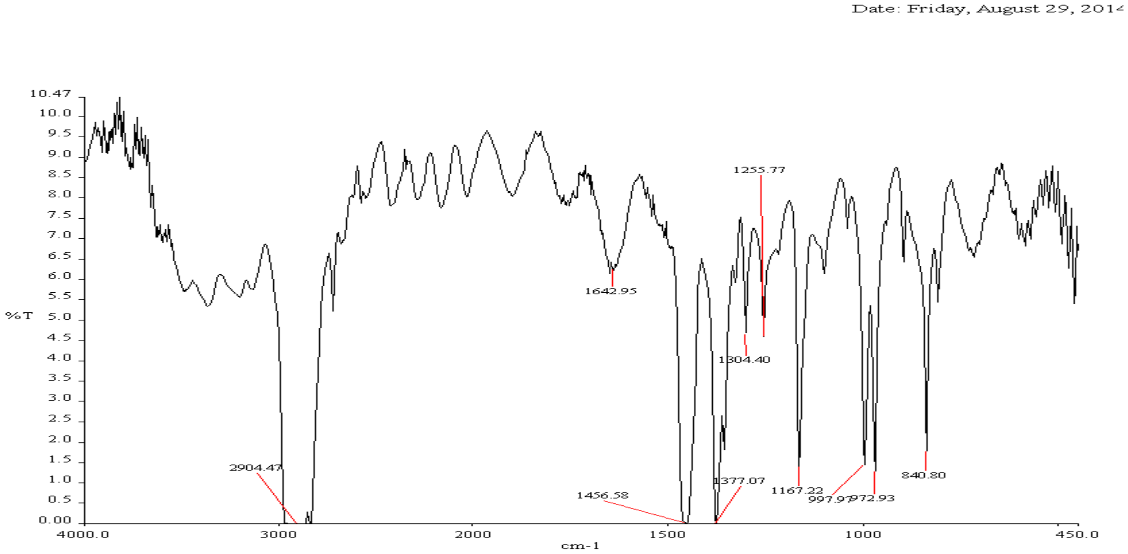


Figure 14 Sample B11 IR Spectrum

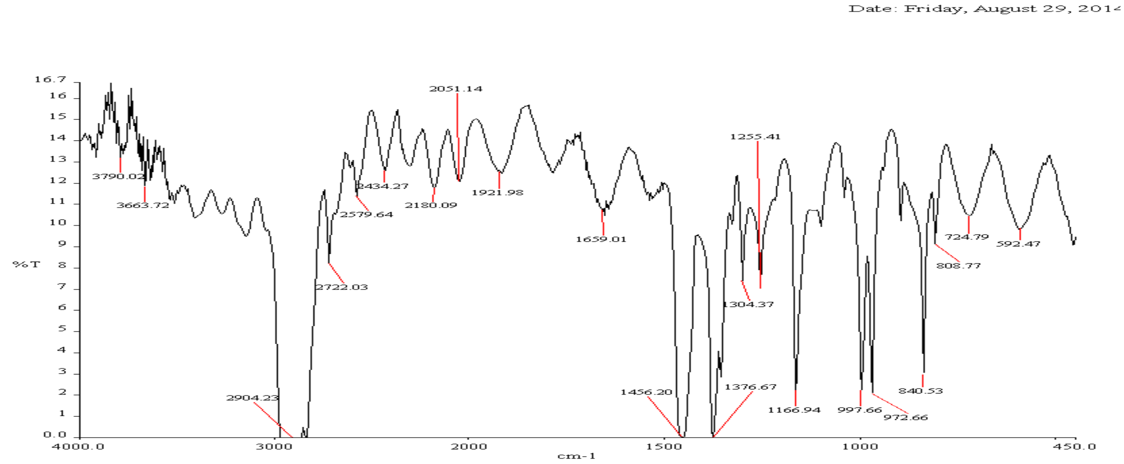


Figure 15 Sample B22 IR Spectrum

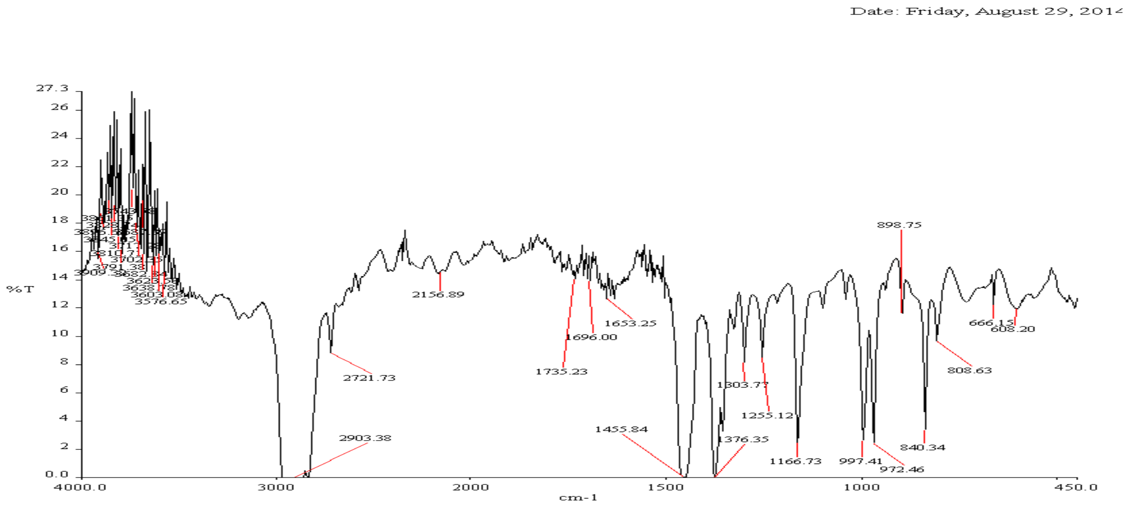


Figure 16 Sample B33 IR Spectrum

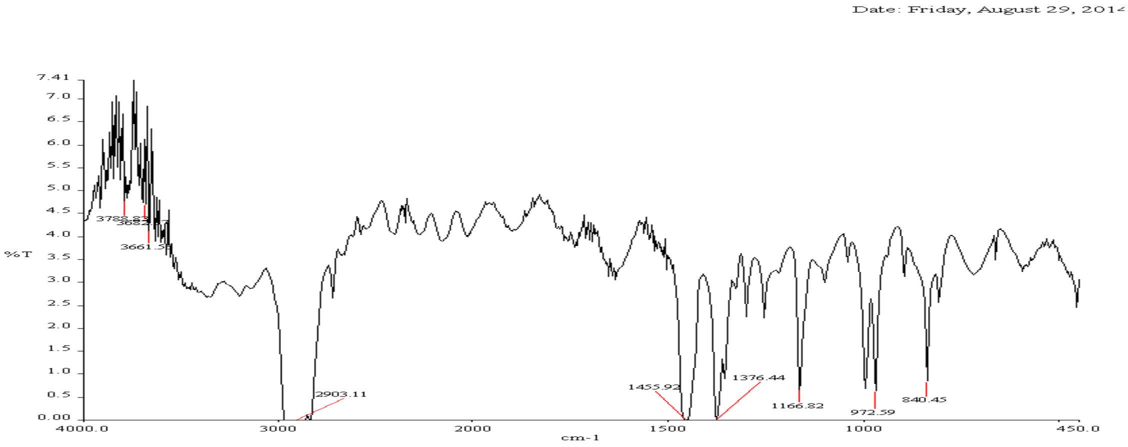


Figure 17 Sample B44 IR Spectrum

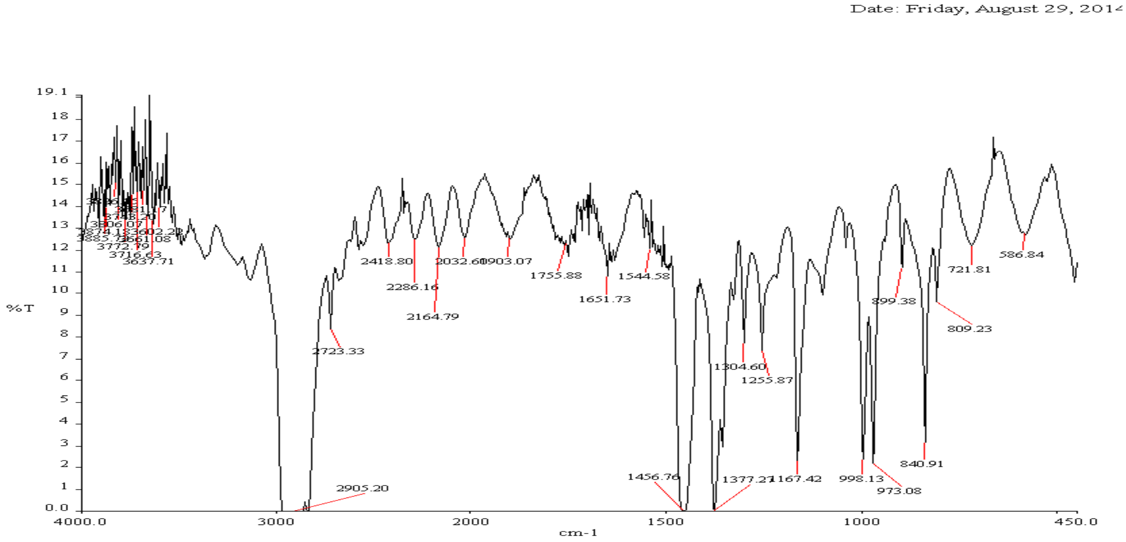


Figure 18 Sample C1 IR Spectrum

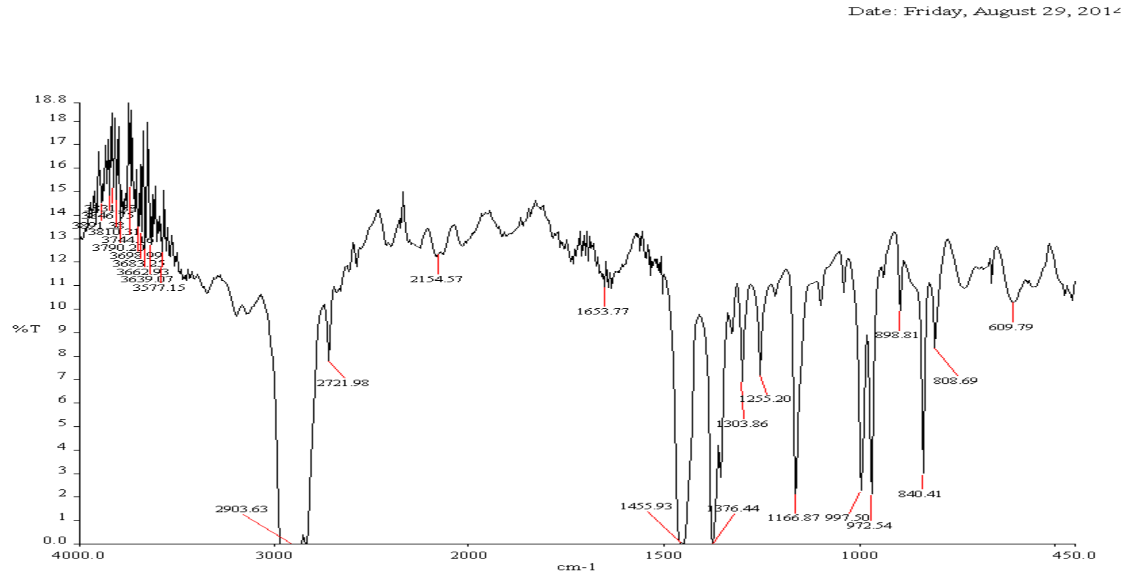


Figure 19 Sample C2 IR Spectrum

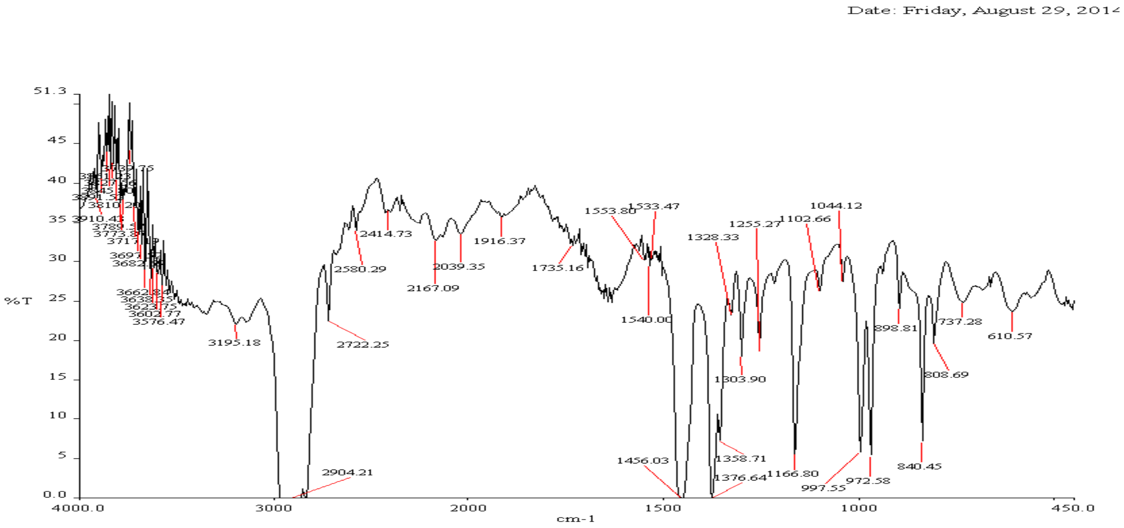


Figure 20 Sample C3 IR Spectrum

Infrared spectrum was taken for all the pure aqueous solutions of the reactants, this was done for comparison purpose, and the results are as follow:

Date: Monday, September 29, 2014

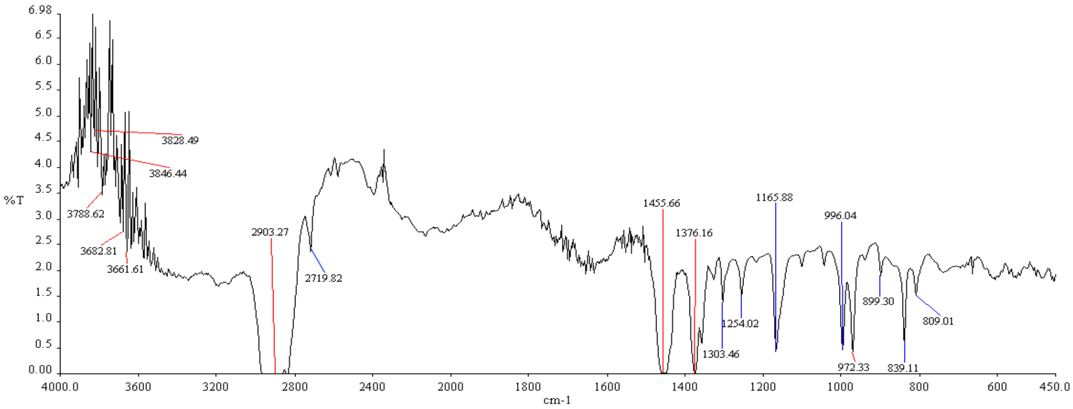


Figure 4.16: spectrum for $AgNO_3$ solution

Date: Monday, September 29, 2014

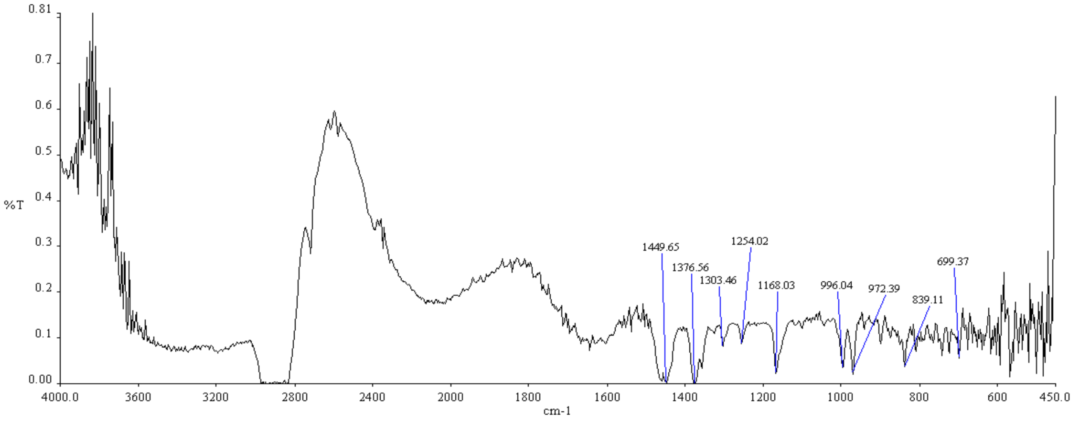


Figure 4.17: spectrum for $NaBH_4$ solution

Date: Monday, September 29, 2014

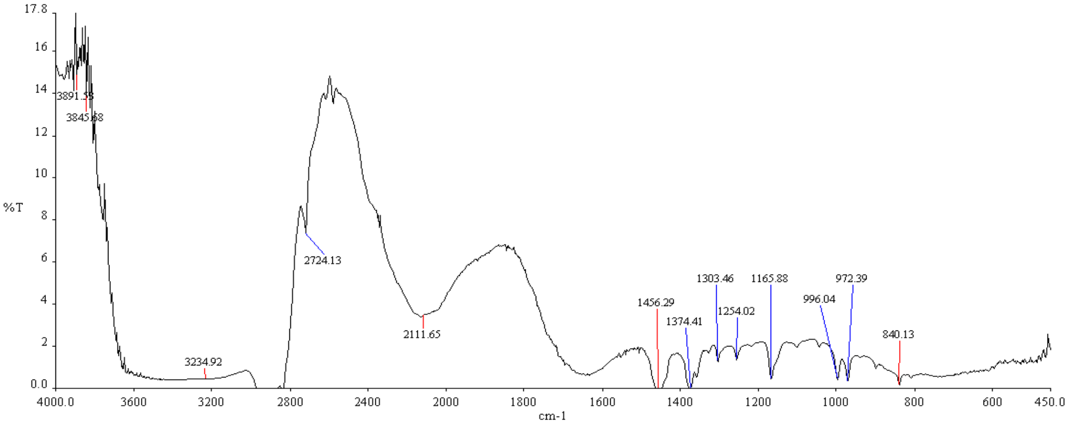


Figure 4.18: Spectrum for 3NaCi solution

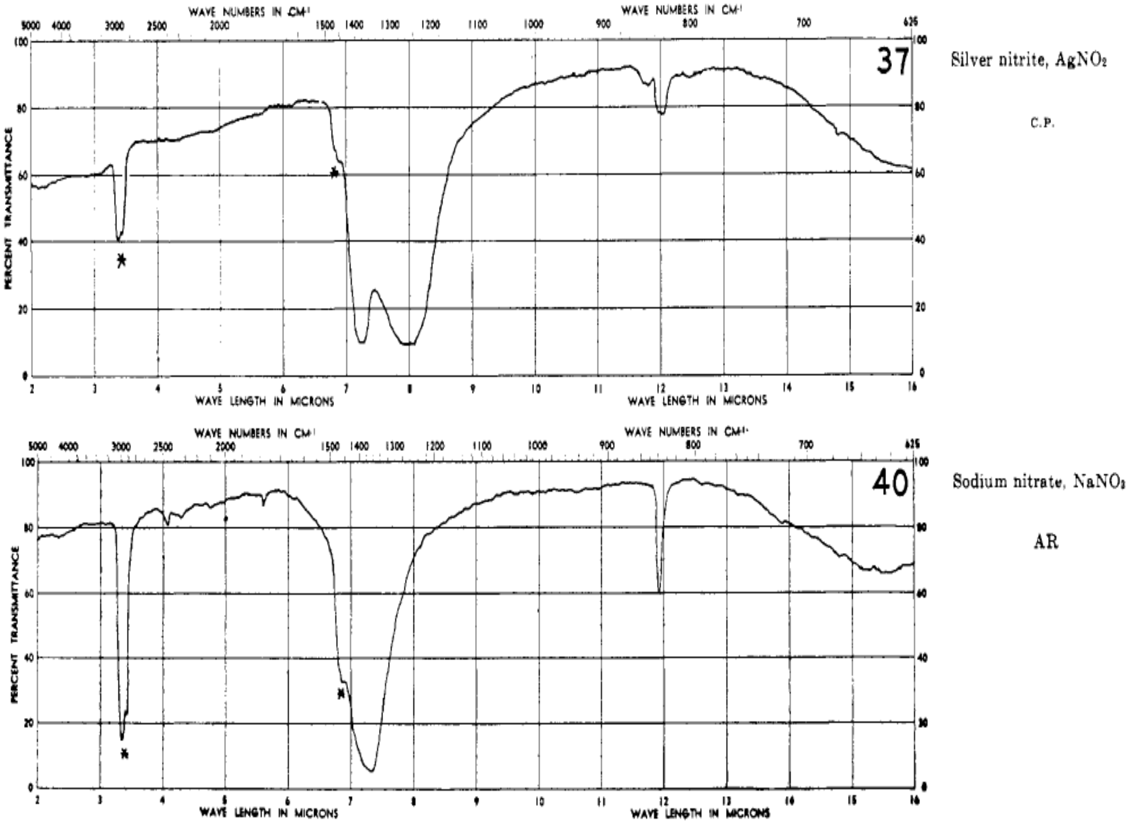


Fig 4.: IR spectrum for $AgNO_3$ and $NaNO_3$
Source: Infrared Spectra and Characteristic Frequencies of Inorganic Ions, *Their Use in Qualitative Analysis*. FOIL A. MILLER AND CHARLES H. WILKINS June 7*,* 1952, *Department* of *Research in Chemical Physics, Mellon Institute, Pittsburgh 13, Pa.*

DISCUSSION OF RESULTS

Results from Sodium Borohydride Reduction

Samples from sodium borohydride reduction were labelled sample A11, A22, A33, A44 and B11, B22, B33, B44. The discussions of the results obtained are as follows. Based on the spectrum obtained and as shown above it can be observed that common peaks exist as summarized in the table below:

Table 5.1: Summary of the common absorption peaks in the different samples synthesised at room temperature of 27^{0}C

Sample	Wave length cm^{-1}	%Transmittance or Absorbance
A11	1456.98	11.5
	1377.39	10.0
	1167.39	14.0
	840.99	14.0
	2905.62	12.0
A22	1456.40	17.0
	1376.93	16.0
	1167.16	20.0
	840.73	18.0
	2904.48	19.0
A33	1456.34	37.5
	1376.78	35.0
	1167.09	45.0
	840.69	45.0
	2904.71	37.5
A44	1455.84	18.0
	1376.42	14.0
	840.33	15.0
	1166.70	18
	2903.33	15.0

Table 5.2: Summary of the common absorption peaks in the different samples synthesised at temperature of 4^0C

Sample	Wave length cm^{-1}	%Transmittance or Absorbance
B11	1456.58	7.0
	1377.07	6.5
	1167.22	8.0
	840.80	7.0
	2904.47	7.0
B22	1456.20	12.0
	1376.67	10.0
	1166.94	14.0
	840.53	12.0
	2904.23	11.5
B33	1455.84	14.0
	1376.35	13.0
	1166.73	14.0
	840.34	12.0
	2903.38	13.0
B44	1455.92	3.5
	1376.44	3.5
	1166.82	4.0
	840.45	4.0
	2903.11	3.5

From the reaction mechanism of silver nanoparticles synthesised using sodium borohydride, $AgNO_3$ and $NaNO_3$ are compounds with positive polyatomic ions which exhibit characteristic infrared spectra (Foil A. Miller et. al., June 7, 1952).

The spectra range in quality from surprisingly good ones, with sharp intense bands to very poor ones. From the spectrum obtained for $AgNO_3$ aqueous solution it can be seen that significant peaks exist at 839.11 cm^{-1} and 1376.16 cm^{-1} which are similar to that obtained from literature (Foil A. Miller et. al.) along with that obtained from literature for $NaNO_3$ with peaks at 836 cm^{-1} being medium and sharp and 1358 being very strong (Foil A. Miller et. al., June 7 , 1952), from this, it can be can be said that the silver ions (Ag^+) of the silver nanoparticles attach themselves to the compound therefore giving similar peaks in all eight samples of characteristic frequencies at 840.33 – 840.80 cm^{-1} and 1376.35 – 1377.07 cm^{-1}.

Furthermore, from the result obtained it can be seen that sample A33 shows the highest % transmittance or absorbance of 45% at 840.69 cm^{-1} and 35% at 1376.78 cm^{-1} proving that the ratio silver nitrate to sodium borohydride and the experimental condition taken gives the best result with stable nanoparticles.

Also from the table of characteristic IR absorptions in organic compounds it can be seen that the possible functional group and bond present at 1345 – 1385 cm^{-1} is the nitro functional group and N – O single bond which is a strong, two bands stretch and very much evident from the spectra obtained in this report.

The strong broad band found 2500 – 3000 cm^{-1} is attributed to the O – H single bond functional group and is located within the range 2903.20 – 2904.75 in the spectra obtained for the samples in this report and this is attributed to the presence of water in the sample solutions.

Results from Trisodium Citrate (3NaCi) Reduction

The samples gotten from 3NaCi reduction were labelled sample C1, C2 and C3

The discussions of the results obtained are as follows. Based on the spectrum obtained and as shown above it can be observed that common peaks exist as summarized in the table below:

Table 5.3: Summary of the common absorption peaks in the different samples synthesised

SAMPLE	WAVELENGTH CM^{-1}	%TRANSMITTANCE OR ABSORBANCE
C1	2904.92	8.0
	1456.61	8.0
	1167.39	7.5
	997.95	10.5
	840.81	9.0
C2	2905.20	12.5
	1456.76	12
	1377.27	11
	1167.42	13
	998.13	14.0
	840.91	14.0
C3	2904.21	25.0
	1456.03	30.0
	1376.64	25.0
	1166.80	30.0
	997.55	30.0
	840.45	**25.0**

As established earlier for $AgNO_3$ and $NaNO_3$ to possess positive polyatomic ions with characteristic infrared spectra. The spectra gotten from 3NaCi solution and samples C1, C2, C3 show similar peak absorbance within the range 1370 – 840 cm^{-1} and as discussed earlier ions of silver nanoparticles attach to Na^+ within the $NaNO_3$ compound

Furthermore the broad strong peak at 2904.21 – 2904.92 cm^{-1} is attributed to O-H functional group from the water present in the solution sample, and also sample C3 shows the highest %transmittance or characteristic absorbance around 25% at 1376.44 cm^{-1} and 840.45 cm^{-1}.

Finally it was observed that for synthesis of silver nanoparticles, using $NaBH_4$ as reducing agent, both at room temperature and at ice cold temperature i.e. Samples A11-44 and B11-B44, colloids were formed within 3-5 minutes and stirring had to be stopped as soon as dropping stops, a yellow colouration indicated the presence of formed nanoparticles. In the case of synthesis using 3NaCi as reducing agent post addition stirring was done so as to enhance colloid formation, also a yellow colouration was observed indicting the presence of formed silver nanoparticles.

CONCLUSION

From the experiment performed and the results obtained it can be concluded that synthesis of silver nanoparticles with sodium borohydride can be performed at room temperature of 27^0C, after being compared with that performed by precooling silver nitrate and sodium borohydride in ice cold water bath, giving the best result at ratio quantity in mL of $AgNO_3$ solution: $NaBH_4$ solution of 10:25 respectively giving the highest and strongest absorbance when characterised with FTIR spectrophotometer. This proves the presence of silver nanoparticles at absorption peaks within range of wavelength 1370-1380 cm^{-1} and 840-841 cm^{-1}.

Also the same goes for the silver nanoparticles synthesised using trisodium citrate as reducing agent at boiling temperature of 100^0C, here similar results with that of the sodium borohydride reduction were obtained, therefore, proving the presence of silver nanoparticles at absorption peaks within wavelength range of 1370-1380 cm^{-1} and 840-841 cm^{-1} attributed to the N-O, nitro, functional group in organic compounds. With sample C3, having $AgNO_3$ solution : 3NaCi solution in mL of 50:45 respectively, having the highest and strongest peak absorbance.

It has also been discover, during the experiment, that silver nanoparticle colloid was formed using sodium borohydride irrespective of temperature but the synthesis performed at lower temperature yielded more stable colloids. Also to prevent aggregation when using sodium borohydride as reducing agent stirring was stopped immediately after dropping is complete and the time taken for stirring and dropping is no more than 2-3 minutes. When using a weaker reducing agent like trisodium citrate post-addition stirring for at least 30minutes is most ideal for colloid formation and nanoparticles stability.

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