SOLUTION COMBUSTION SYNTHESIS OF ALUMINIUM OXIDE NANO PARTICLES USING BIOFUELS AND IT'S CHARATCTERISATION

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CONTENTS

ABSTRACT

Nanoscience is an important research field which has seen a huge revolution and industrial interest over the last few years.This project entitled “Synthesis, Characterisation and Anti bacterial study of

Aluminium oxide nano particle deals with the synthesis of aluminium oxide nano particles via solution combustion method using three different fuels namely urea, glycine and citric acid and it’s characterisation”.

This project contains three chapters. The chapter 1 deals with the introduction of nanomaterials. Here we have already discussed about the different synthesis methods. And also discussed in detail about the properties of nanomaterials.

In chapter 2 a brief description on the experimental techniques used for the synthesis of aluminium oxide nanoparticles are discussed. The optical characterizations of the nanoparticles were done using UV- Vis spectroscopy. XRD method is used for the structural identification. SEM method is also used for surface analysi of pure aluminium oxide nanoparticles..

The chapter 3 deals with the result and discussion. Synthesis process is discussed in detail. XRD analysis, SEM analysis and UV-Vis method are discussed there. Crystalline structure of the sample is identified by using xrd method. From the result obtained from SEM analysis, sample U has a coral reef structure with large pores where as the other two samples have a flake like structure. UV Vis method reveals that the band gap energy of aluminium oxide is 3.31 eV. The antibacterial activities of the aluminium oxide were tested against E. Coli(gram negative bacteria) and S. Aureus(gram positive bacteria).The project work is concluded with the conclusion and future scope of the work .

CHAPTER 1

GENERAL INTRODUCTION

1.1 INTRODUCTION TO NANOPARTICLES

The prefix 'nano'in the word nanotechnology means a billionth of a meter. Nanotechnology is defined as the study and use of structure between1nm and 100nm in size. Nano science is the study and nanotechnology is the exploitation of the strange properties of materials smaller than 100 nanometers, used to create useful objects and they have new properties and behaviour. This happens because particles which are smaller than the characteristic lengths associated with particular phenomena often display new chemistry and physics leading to new behaviour which depends on size. The electronic structure, conductivity ,reactivity, melting temperature, and mechanical properties have all been observed to change when particles become smaller than a size.

Nanomaterials are characterized by large surface areas in comparison with their volumes, and have behaviours that are governed by the laws of quantum mechanics. Nanotechnology is often referred to as a general –purpose technology[1].This is because in its advanced form it will have significant impact on almost all industries and all areas of society. It is expected to offer better built ,longer lasting, cleaner, safer, and smatter products for the home, for communication ,for medicine, for transportation ,for agriculture and for industry in general. Nanotechnology has a multi-purpose role. It will be utilized in many commercial uses and also many military uses. A key advantage of nanotechnology is that it offers not just better products, but a vastly improved manufacturing process.

Nanomaterials are materials having size on the order of a billionth of a meter. The nanomaterials have lower weight but higher strength. Generally size varies from 1 nm to 100 nm. Atom, DNA diameter, protein , bacteria, sand etc. are examples of nanoparticles.

Nanomaterials are classified based on the chemical composition and shapes, diamensionality of their micro structural parameters. A famous scientist named, Richard W Siegel classified nanostructured particles ,according to their dimensions .The nanostructured materials are classified as follows [2].

Zero dimensional nanomaterials : In zero dimensional nanomaterials all the dimensions are measured within the nanoscale.

One dimensional nanomaterials : Here one dimension is outside the nanoscales. That class include nanorods, nanowires etc.

Two dimensional nanomaterials : In two dimensional nanomaterials,two dimensions are outside the nanoscale. it include grapheme , nanofilms, nanocoatings etc.

Three dimensional nanomaterials : These are the nanomaterials that are not confined to nanoscale in any dimension. it contains powders, bundle of nanowires, multi nanolayers etc.

1.2Characteristic features of nanomaterials

Main characteristic properties are the following

- **Large surface to volume ratio**

One distinct property of nanomaterials is that a significant percentage of the atom is related with the surfaces, grain boundary like factors. These materials have large surface to volume ratio,as a result it modifies the properties of bulk material.

- **Quantum confinement effect**

Suppose a crystal having same or smaller exciton Bohr radius of its constituent compound, then we can say it is in quantum confinement [3]. The word confinement indicates the confinement of motion of randomly moving electron to resist its motion in particularly defined energy levels and quantum reflects the atomic realm of particle. Quantom dots are example.

- **Reduced imperfections**

We know that the nanostructures favours a self purification process in that the intrinsic materials defects as well as impurities move near the surface on thermal annealing which increases the purification in materials .

1.3 PROPERTIES OF NANOMATERIAL

1.3.1. Electrical properties

The examples of change in electrical properties in nanomaterials are:

a. Conductivity of bulk material are available. But it does not depend upon dimensions like area of cross sections or diameter.

b. Conductivity of a multiwalled carbon nanotube is different than that of a single nanotube of same diamension.

There are mainly two types of nanotubes such as metallic or semiconductor. Metallic nanotubes can afford an electrical current density of the range more than 1000 times greater than metals such as copper Cu.

1.3.2. Optical properties

Optical properties of nano materials such as absorption , transmission, reflection and light emission are dynamic in nature. An optical response in a nanomaterial can be considered by using so many appropriate mechanisms..It depends upon the nanomaterial size , composition and arrangement. Optical property of nanomateial is very much useful in our day to day life. Laser, sensor, imaging, phosphor, display, solar cell and biomedicine etc. are the application field of optical properties of nanomaterials.

1.3.3 Mechanical Properties

Due to structural perfections of the nanomaterials an enhancement of mechanical properties of nanomaterials takes place [4] .There are so many improvements in the mechanical properties of nanomaterials as a result an interest in nanocomposite in various automotive and general industrial applications takes place. Bulk metallic and ceramic materials, influences of porosity , influences of grain size ,super elasticity, filled polymer composites, polymer based nanocomposites, particle filled polymers etc are some them. We know that nanoparticles have very small errors or defects, as a result strength of the bulk materials. By adding the nanofillers we can increase the strength of bulk materials. Nanomaterials are very harder in nature, water resistant so that is also corrosion resistant. And it is also very stronger in nature. Spark plugs is an example where we are applying these properties.

1.3.4. Magnetic Properties

There are so many magnetic nanomaterials that containing of magnetic elements such as iron, cobalt, chromium, manganese, galodinium, and their chemical compounds. Magnetic nanoparticles are super paramagnetic . Ferrite nanoparticles are the most widely using magnetic nanoparticles. When an external magnetic field is able to magnetise the nanomaterials, then after the removal of external

magnetic field the magnetisation in the nanoparticle becomes zero. This property is useful in controlled therapy and targeted drug injection.

1.3.5. Thermal Properties

Specific heat of nanomaterials is very much higher than that of coarse grained polycrystalline materials of the same composition of elements[5] . The growth of the grain reduces specific heat of the nanomaterials. If there are large number of grain boundaries, then it will increase the thermal expansion of nanomaterials. Enthalpy and entropy like scientific factors are increased for nanomaterials.

1.4.Synthesis of nanostructured particles

1.4.1 Combustion Method

Combustion synthesis or self propagating high temperature synthesis is an effective and cost effective method for the synthesis of various materials. Solution combustion synthesis is simple, rapid process [6] .It is also versatile in nature. Which allows effective synthesis of variety of nanomaterials. It is a self sustained process in homogeneous solution of different oxidizers. There are precursors, the SCS may occur as either volume or layer by layer propagating modes. We call it as self propagating because it does not need the input energy. Also a lot of energy can be saved by using this method. This is a highly exothermic reaction, which occurs with the evolution of heat and light when the fuel and oxidiser mixture are ignited. This method is also useful for the preparation of carbides, borides, nitrides,12 intermetallics, carbo-nitrides, composites, complex oxides etc.

1.4.2 Physical vapour deposition(PVD)

This method is fundamentally a vaporization coating technique, involving

transfer of material on an atomic level. It is an important method to electroplating. The material evaporated by heating, by ion bombardment, or by laser ablation, is deposited on substrate target. The depositional layer may have the nano thickness. Sputtering is an example of this method in which atoms in a solid-state are released and pass in to the gas phase by bombardment with energetic ions.we can use the sputtering principle to the energy of plasma on the surface of a target to pull the atoms of material one by one and deposit them on the substrate.

1.4.3 Chemical vapour deposition(CVD)

It is used to produce high purity ,high performance solid materials. It is used in semiconductor industry. CVD produce thin films. A reactant gas mixture in to contact with the surface, where it decomposes, depositing a dense pure layer of meta [7] .The deposit can be formed by a reaction between precursor gases in the vapour phase .The main disadvantage of this process is that it frequently requires high temperatures. The main disadvantage of this process is that it frequently requires high temperatures.

1.4.4 Hydrothermal process

Water is a very much useful solvent for many ionic compounds. Water can dissolve in the non-ionic compounds under high pressure and temperature. Water has been strongly exploited for the preparation of fine powders of metal oxides. Water behave like a pressure transmitting medium and solvent for the precursors. It effectively brings down the activation energy for the final phase, which can also increase the reaction between precursors, it occur only at high temperature. The pressure attained range is 10to 150 kilo bar. It also depends on the chosen temperature of water. Powders depending on the choosen hypothermal process.

1.4.5 Sol-gel method

Advantages of this method is it can used to control the microstructure of the final products, and we can control the physical, chemical, and mechanical properties of the final products. Starting point is a solution of precursors in an appropriate solvent. The precursor undergo polymerization reaction to form a colloidal suspension,, is called ‘sol’. Discrete finely dispersed particles kept in suspension by adding surfactant.sol –gel method can be the bases of materials include paints , detergents and cells. This method can produce so many materials at low temperature and materials having high purity.

1.4.6 Green synthesis

It is an important field in bio nanotechnology and provides economical well being and it is also an economic friendly one. This process processes using mild reaction conditions and non toxic precursors are emphasized in the nanotechnology. Chemical,physical and biological methods are developed for the synthesis of nanomaterials.But for chemical and physical methods are involved in the production of toxic byproducts[8]. These by products are dangerous to the nature.

Green synthesis is an important method used to reduce the destructive and bad effects related with the conventional methods of synthesis of nanoparticles. To synthesis the metal or metal oxide nanoparticles ,plant diversity is considered.

In this method there is no need for high pressure, energy,temperature and toxic chemicals.

1.5. Aluminium oxide nanoparticles

Aluminium oxide nanoparticles have so many applications in our research field or scientific field. Aluminium is Block P, Period 3 element, while oxygen is a Block P, Period 2 element[9].

We know the aluminium oxide is spherical shaped, white coloured powder. Aluminium oxide nanoparticles are highly flammable and an irritant ,so that it may cause eye problems or respiratory problems. Aluminium oxide has the formula Al_2O_3 .It is the most commonly occurring of several aluminium oxides, and specifically identified as aluminium (III) oxide.It occurs naturally in its crystalline polymorphic phase α-Al_2O_3 as the mineral corundum, varieties of which form the precious gemstones ruby and sapphire.

Aluminium oxides can be prepared from many techniques including sol-gel, sputtering, hydrothermal etc. All these methods were discussed above.

Aluminium oxide is insoluble in water. In its most commonly occurring crystalline form is called corundum or α-aluminium oxide, since it is very hard it can be used in cutting tools.

In aluminium oxide each Al^{3+} centre is octahedral. In terms of crystallography, corundum adopts a trigonal bravais lattice.

1.6 Chemical properties of aluminium oxide

Chemical symbol	Al_2O_3
CAS NO.	1344-28-1
GROUP	Aluminium 13 Oxygen 16
Electronic configuration	Aluminium[Ne] $3s^2 2p^3$ Oxyger [He]$2s^2 2p^4$

1.6 .1 Chemical Composition

ELEMENT	CONTENT(%)
Aluminium	52.92
Oxygen	47.04

1.6.2 Physical Properties

PROPERTIES	Metric	Imperial
Density	3.9 g/cm^3	0.140 lb/in^3
Molar mass	101.96 g/mol	

1.6.3 Thermal properties

Properties	Metric	Imperial
Melting point	2040^0C	3704^0F
Boiling point	2977^0C	$5391^{0f}F$

1.7 Applications

a. In YAG laser crystals

b. As cosmetic fillers

c. Polishing materials, cutting tools, high purity crucible

d. Catalyst, catalyst carrier, analytical reagents

e. Aerospace aircraft wing leading edges

f. In integrated circuit baseboards

1.8 APPLICATIONS OF NANOSTRUCTURED MATERIALS

1.8.1 . Next generation computer chips

Transistors, resistors, and capacitors are very small in size. It is an application of microelectronics. By reducing the size we can minimize the size of the system. By reducing the size there will be tremendous increase in speed. But we need advanced technological methods to use this microchips, because these chips will produce more heat due to its high speed operation.

1.8.2 . High power magnets

Coercivity and saturation magnetization values are the parameters using for measuring the strength of the magnet. These parameters will be increased with a decrease in the grain size and there will be an increase in the specific surface area of the grains. Automobile alternators, land based power generators, motor for ships, ultra sensitive analytical instruments etc.are the main applications of high power rare earth metals.

1.8.3 Fuel cells

Lithium is an important element in science. It can be used as lithium batteries. Lithium is a charge carrier in some of the batteries. Nanomaterials can be used as a coating to separate the electrodes from any liquids in the battery, when the battery is not in use[10]. Nanocrystalline materials can be prepared by using sol gel method. These can be used as candidates for separator plates in batteries. Foam like structure is the main reason. This structure can hold more energy than old techniques.

1.8.4 Agriculture

Nanotechnology plays an important role in agriculture industries such as crop production, packaging and food processing, water purification, crop improvement, food security etc.Genetically improved

animals andplants increase the crop production and it can provide more pest resistance. Nanotechnology has the potential of precise delivery of agrochemicals for overcoming the disease resistance, plant growth etc[10]. Site specific drug and gene delivery of molecules at cellular or molecular levels in plants as well as in animals. There are so many crops that are using nanotechnology. These effective methods help our farmers to attain more wages also. One is to be remembered that every new nano - technological application should be tested for toxic problem.Otherwise it will lead to the destruction of our land and crop.

1.8.5. High sensitivity sensors

Sensors that use sensitivity to the change in various parameters ,designed to measure. Electrical resistivity, chemical activity, magnetic permeability, thermal conductivity and capacitance. These parameters depend on the grain size designed in the sensors. I there is a change in its environment then there will be change in the nano crystalline materials. There are so many applications. Smoke detector is an example. Automobile engine performance sensor is an another example also.

1.9 Literature review

We know that aluminium oxide powder , a white coloured powder of formula Al_2O_3 has so much importance in our world. Ceramics, glass industry, military equipment field biomedical field such as in cancer treatment etc. Are the major applicational field of aluminium oxide nanoparticles. It is also used as a potential antibacterial agents against infectious organisms such as E. Coli and Staphylococcus aureus.[11]

Aluminium oxide nanoparticle can be synthesised using several methods. Here we are discussing about the formation of aluminium oxide by combustion method . Kingsley and Patil named author processed a chemical reaction by using the mixture of aluminium nitrate , urea and sp. The mixture is heated at a temperature range of 500±10 degree Celsius. It was carried out in less than 5 minutes

only. Water dissolving and heating are the processes involved here. This process got a result . Major findings is that new urea nitrate process for α alumina synthesis Also identified that the combustion method is controlled by the stoichiometry,mass of the combustion mixture and volume of the container.[11]

Chick et al. named author made a combustion process by using the mixture of aluminium nitrate, glycene and sp. Hot plate at a temperature of 180 degree Celsius is needed. Water is dissolved and heating started to get the result. Finally, he concluded that it is the new formula for the production aluminium synthesis using glycene nitrate mixture. It is also an effective, rapid , self sustaining process occurs at high temperature. [11]

Meng et al, named author made a reaction using the mixture of aluminium nitrate , urea ,ammonium nitrate,starch and SP .Temperature range was 150 ±10 degree Celsius. The reaction involved the dissolving of water to the mixture and addition of ammoniumnitrate or starch . Heating is carried out at different temperatures such as 800, 900 and 1200 degree Celsius. The result was quite interesting that pure α-alumina phase with small crystallite size was obtained from urea and ammonium nitrate as starting fuels. [11]

Sharma et al, named author combusted using the mixture of aluminium nitrate, glycene with 40% of fuel addition primarily. Later the process repeated with 80% and 120% of fuel addition. It is carried outin muffle furnace at a temperature range of 350<T<450 degree Celsius. After the dissolving of water to the mixture it is stirred well for 2 hours. And combustion takes place. From this process, result is obtained as follows. This process has greater potential for the synthesis of alumina with amorphous structure. Amount of gas and flame increases with the increase in fuel/oxidizer ratio.[11]

But in this project report, we are discussing about the production of aluminium oxide powder by combustion method.Aluminium nitrate+glycene, aluminium nitrate+ urea ,

aluminium nitrate+ citric acid mixture areused separately. Aluminium nitrate is taken 10gram and urea is taken for 10 gram also.It is allowed to dissolve in 10 ml of distilled water, and stirred well for half an hour. This solution is allowed to combust until we get the required ash. After getting the ash, it is allowed to calcinate in a furnace at 850 degree Celsius for 5 hours. After that we got the pure aluminium oxide. It is then allowed for the characterization techniques like XRD, UV- Vis method and SEM.

CHAPTER 2

CHARACTERISTIC TECHNIQUES

2. INTRODUCTION

Characterization of nanoparticle is important to establish the understanding and control of nanoparticle synthesis and applications.Different method have been used to characterize the size,crystal structure,elemental composition and other properties of nanoparticles. There are so many methods are available in each of these areas and an orderly application of so many tools lead to a complete understanding of the system. The properties of individual nanoparticles can be characterised by various methods. Each method has its own advantages and limitations.

2.1 X RAY DIFFRACTION METHODS(XRD)

It is an important method used for the characterization of the nanoparticle. XRD provides or gives the information regarding the crystalline structure, nature of the phase, crystalline grain size, and lattice parameters etc.

This method is also suitable for the qualitative as well as quantitative analysis of solid phases. So many materials can be identified by using this method. XRD method is useful to examine whether it is amorphous or crystalline in nature. Crystalline phases can be identified by comparing the d values obtained from X ray diffraction method data with the fundamental data in Joint Committee on powder Diffraction Standards (JCPDS)[12].

2.1.1 . Theory of XRD

In 1912, a scientist named Max Lause first observed the diffraction of x ray from a crystal. XRD method is based on constructive interference of monochromatic X-rays and a crystalline sample.

From a cathode ray tube X rays are generated. And is filtered to produce monochromatic radiation, and is collimated to concentrate or focus. That is directed to the sample under study. The interaction between incident rays with the sample produces constructive interference and a diffracted ray if the conditions are satisfied by Bragg's Law.

When a crystal is bombarded with X-rays of fixed wavelength at incident angles, highly intense reflected X-RAYS are generated when the wavelength of the scattered X- rays interfere constructively. There is a condition for constructive interference, that is the travel path must be equal to integer multiples of the wavelength.[13]

By Bragg's Law ,

$$n\lambda=2d\sin\theta,\ n=1,2,3...$$

Where d is the spacing between two planes, θbe the angle that X ray beam makes with respect to the plane, and λ be the wavelength of the incident rays(X ray). The intensity of the diffracted rays depends on the diffraction angle 2θ . And it is also depends on the orientation of the specimen. By using X ray diffraction method we can easily understand the structure of the material we have to identify. Each sample produces X rays of particular wavelength. Because each material differs in structure.So that we can identify the sample easily.

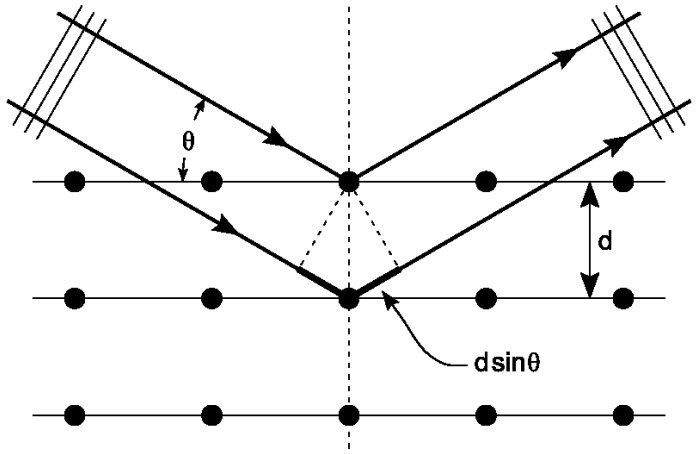


Figure of bragg's law (Fig 1)

Each crystallographic material has three indices,represented by h,k,l.The distance d between two parallel crystallographic planes with indices hkl for a simple cubic lattice constant a has the form given below.

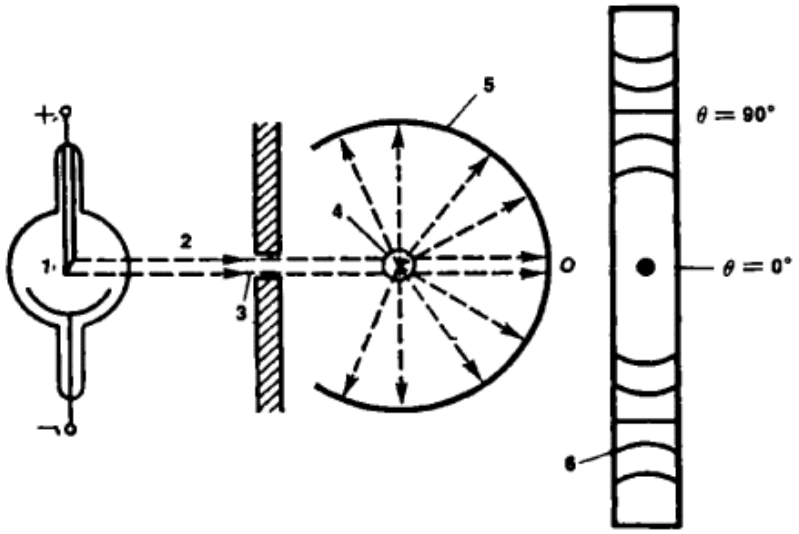
$d_{(h,k,l)} = a/(h^2+k^{2+}l^2)^{1/2}$

We know that XRD technique is a well known method for the characterization of homogeneous and inhomogeneous strains. From XRD pattern , we can easly measure the peak positions. The change in interplanar spacing d can be calculated from the shift created there. Due to the inhomogeneous strain there is the possibility of the broadening of the peaks .The broadening increases with sinθ. There is another reason for this broadening, finite size of the crystals is that reason. In this case broadening is independent of sinθ. We can use Debye-Scherrer's formula for the calculation of the size 'D' of the crystallite from the width of the peak condition is that there should not inhomogeneous strain. The Debye- Scheerer formula is the following,

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

Where K is the Scherrere's constant, λ is the wavelength of X-rays, β is the full width half maximum (FWHM) of the diffraction peak and θ is the diffraction angle.

Debye-Scherrer method employs a monochromatic X- ray beam incident on a powder sample that is contained in a good mannerly walled glass tube. That tube can be easily rotated to make the diffracted pattern smooth. For each conical angle 2θ, there is also the emerging of X-rays, with θ satisfies the Bragg condition is independent on that film that is using in arcs as shown below. Bragg angle has the value $\theta = S / 4R$. Where S is the distance between two corresponding reflections on the film and R be the radius the film cylinder. So that for a single exposure of the powder sample to the X-ray radiation will lead to the Bragg angle at the same time.



Debye- Scherrer method figure (Fig 2)

2.2. UV – VISIBLE SPECTROSCOPY

UV – Visible spectroscopy or UV – Vis method measures the extinction (scatter+absorption) of light passing through a sample under study. We know that nanoparticles are very much sensitive to the change in size, shape, concentration, refractive index near the nanoparticle surface , that makes UV method an easy important tool for identification , characterization and studying the material under study. UV Spectroscopy is qualitative in nature at first, with the absorption of near UV(180 – 390 nm) or visible radiation (390 – 780) by using the chemical materials [14].

In UV SPECTROSCOPY , there are so many superimposition of vibrational and rotational transitions the UV visible spectrum of analytes in solution shows little fine structure . This is a drawback of this method. So this method is not widely using for the identification, but this method is very much useful in qualitative analysis .

Scientists and researchers are using this method for both organic and inorganic species . By using Beer – Lambert law we can measure the elemental concentrations quantitatively in a solution as per the following

$A = \log_{10} I_0 / I = \varepsilon bc$

Where A is the measured absorbance, I_0 is the intensity of light at a particular wavelength, I is the transmitted intensity, L is the path length through the sample, c is the concentration of the absorbing species, and ε is the constant called as the molar absorptivity or extinction coefficient for each species and wavelength. Normally, ε at the wavelength of maximum absorption is allowed in quantitative analysis, because the errors are arising from instrumental wavelength's uncertainty are minimized at the peak of the absorbance curve drawn already.

2.2.1 . The UV – Visible Photospectrometer

UV – Vis spectroscopy can be used for finding out the concentration of the absorbing species. It is only for a fixed path length. It is a simple process. Also it is versatile process and provide accuracy. It is also cost effective in nature. Instrument used for the UV-Vis spectroscopy is known as UV-Vis Spectrophotometer. Light source for the emission of UV and visible light, monochromator using for wavelength selector, sample stage, and detector are the main components of an UV Spectrometer. Tungsten filaments are the main light source that is commonly using since this covers the entire UV region. Tungsten filaments normally emit red radiations of wavelength 375 nm. Also monochromators play an important role in UV-Vis spectroscopy. It is made up of prisms and slits [15] . The radiation emitted from the primary source is dispersed with the help of rotating prisms. The different wavelengths are there for the light source. So that the light emitted from the light source having different wavelengths are then selected by the slits such the rotation of the prism results in a series of continuously increasing wavelength to pass through the slits for recording purpose. If the beam selected by the slit is monochromatic then it is divided into two such beams with the help of another prism. One beam passes through the sample solution and second beam passes through the reference solution . Reference solution and sample solution are contained in cells. There is a need of this strong cells .The cells are made up of either using silica material or quartz material. There is a fact that we cannot use glass there because glass absorb light in the UV region. Also there are two photocells used for the purpose of detector in UV Spectroscopy. There are two

photocells. One of that photocell receives the beam from the reference in the cell. Intensity of beam of sample cell is less than that of the reference cell. As a result generation of pulsating or alternating currents in the photocells takes place. Produced alternating current is transferred to the amplifier. Amplifier is using to amplify the signals.[16]

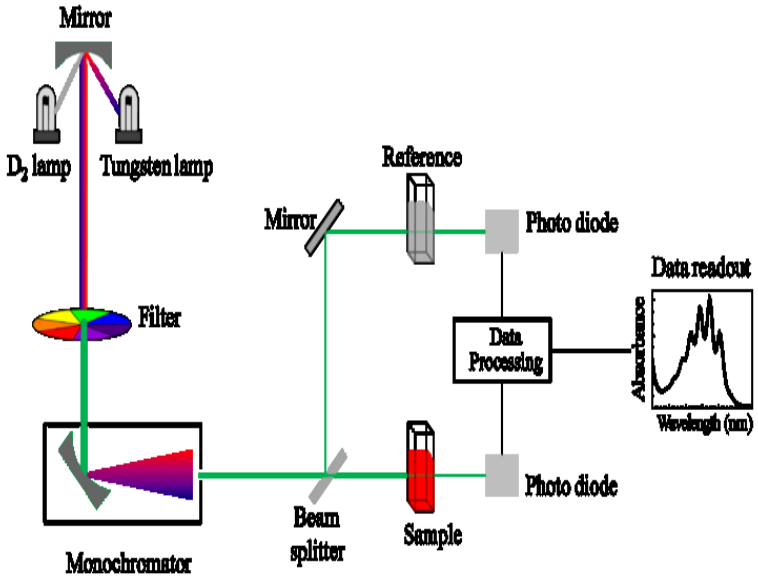


Figure of UV-Vis spectrometer (Fig 3)

2.2.2 APPLICATION OF UV-Vis SPECTROSCOPY[17]

Important applications of UV-Vis spectrometer are the following

a. Detection of impurities

b. Structural explanation of organic compounds.

2.3 SEM

Scanning electron microscopy(SEM) or SEM analysis is a powerful analytical technique to perform analysis on a wide range of materials at very high magnifications to produce high resolution images.

2.3.1 SEM IMAGING

SEM relies on the of electrons having high energy , emitted from the surface of the required sample after exposed to a highly focused beam of electrons emitted from an electron gun. This beam of electrons is allowed to focus on a small spot on the sample surface, by using the objective lens . Accelerating voltage used, size of aperture used and the distance between the sample and electron gun are the variables here. These variables can be optimised to achieve the best quality images.

There are mainly two modes of electron detection which allows for different types of imaging and analysis. Secondary electrons emitted close to the surface of the sample, providing information about the surface topography. Backscattered electrons can be detected to give contrast based on different chemical compositions across an image.[18]

2.4. Fourier transform infrared spectroscopy

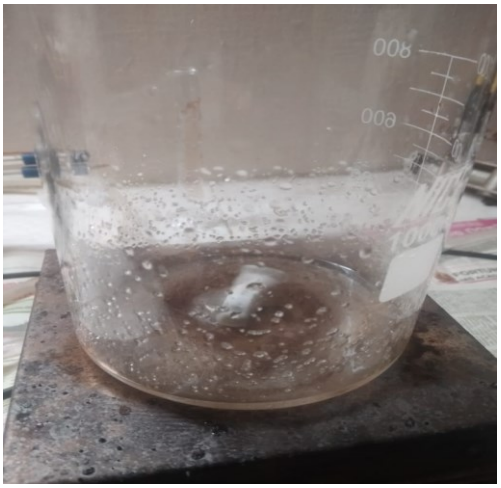
FTIR stands for Fourier Transform Infrared Spectroscopy . When we pass an IR radiation through a sample under study , some radiation is absorbed by the sample and some radiation will be transmitted or passed through that sample. We will obtain a resulting signal at the detector , that will be the fingerprint of the sample under study . There are so many chemical structures or molecules , so that they will produce or generate different spectral fingerprints.

CHAPTER 3

RESULT AND DISCUSSION

3.1 Synthesis of aluminium oxide particle.

Al_2O_3 nanoparticles have the potential applications in various fields . And this nanoparticle can be synthesised using different types of methods. Out of that we preferred the solution combustion method. 10 gram of aluminium nitrate hydrate was dissolved in minimum amount of distilled water to which 10 gram of fuel is added and stirred well complete dissolution.. The mixture is stirred magnetically upto 30 minutes to obtain a uniform solution. There after, the solution is heated inside a combustion chamber for 10 to 15 minutes. The water evaporates and the solution becomes thicker. Once the solution reaches its ignition temperature, combustion starts locally at one point and spreads to other parts of the beaker leaving nanopowder in the beaker. After the combustion process the resultant powder is heated in a muffle furnace at 840^0C for 5 hours. The experiment was repeated with urea, citric acid and glycine as fuels and the respective samples were labelled as Sample U, Sample C and Sample G. These white powdered samples were analysed for its optical and structural characteristics.



Magnetic stirring

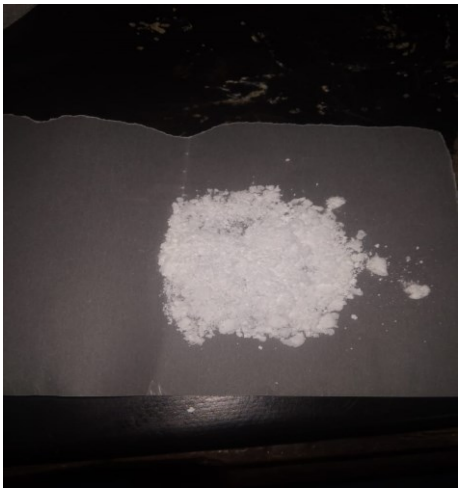
(Fig 4)

Ignition phase in the combustion process

(Fig 5)

Ash obtained after combustion

(Fig 6)



Al_2O_3 nanopowder formed after calcination

(Fig 7)

Fig : Different stages of Combustion process

Results and Discussion

3.2 XRD ANALYSIS

X-ray diffraction pattern is a basic method to explain the crystal phase and crystalline size of the synthesized nanoparticles. The monochromatic CuKα radiations (λ = 0.15418 nm) is utilized as a source of energy 40 kV/35 mA. The graph is recorded in the 2θ range of 20^0 to 80^0 with scanning speed of 5^0/min. The sharp peaks in the XRD pattern of Sample U shown in figure ---1significantly indicates the formation of crystalline phase. The interplanar spacing, 2θ values and relative intensity values of aluminium oxide nanoparticles corresponding to the observed diffraction peaks were compared with the standard values of aluminium oxide nanoparticles reported by JCPDS International Centre for Diffraction Data. The obtained pattern was found to match well with JCPDS-ICDD pattern number #71-1683havingrhombohedral structure. Ten reflections observed at 2θ angles around 25° (012), 35° (104), 38° (110), 43° (113), 52° (024), 57° (116),61° (018), 66° (214), 68° (300)and 76^0(119) confirms the formation of the α-Al_2O_3phase.The additional peaks at around 45^0indicates the presence of γ-Al_2O_3. The absence of characteristic peaks corresponding to the impurities further confirms the purity of the formed nanoparticle.[19]

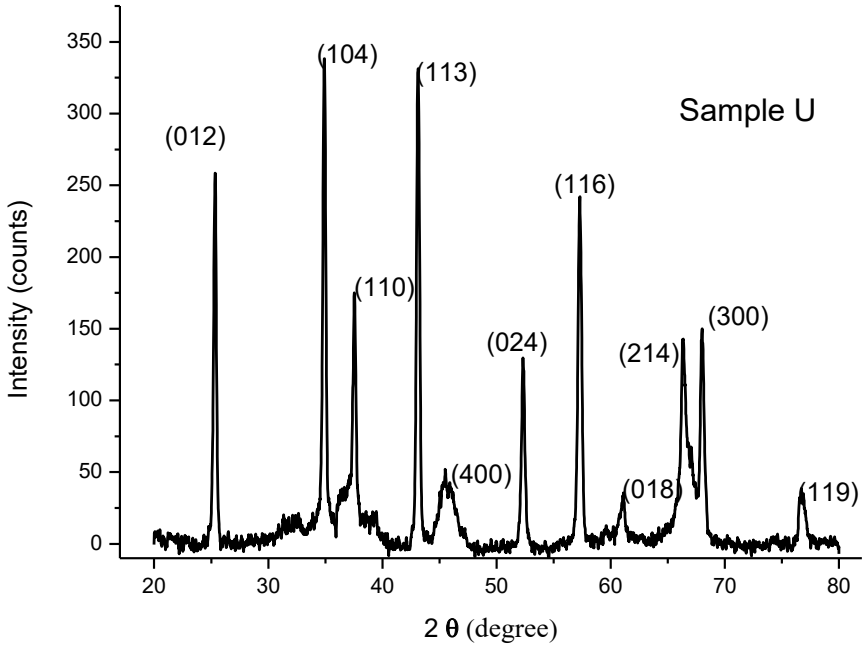
The average crystalline sizes of α-Al_2O_3phase calcinated at 850°C/5h is calculated from the full width at half maximum (FWHM) of the peaks using below Debye-Scherrer formula (1):

$D=0.9\lambda/\beta\cos\theta$ ---------- 1

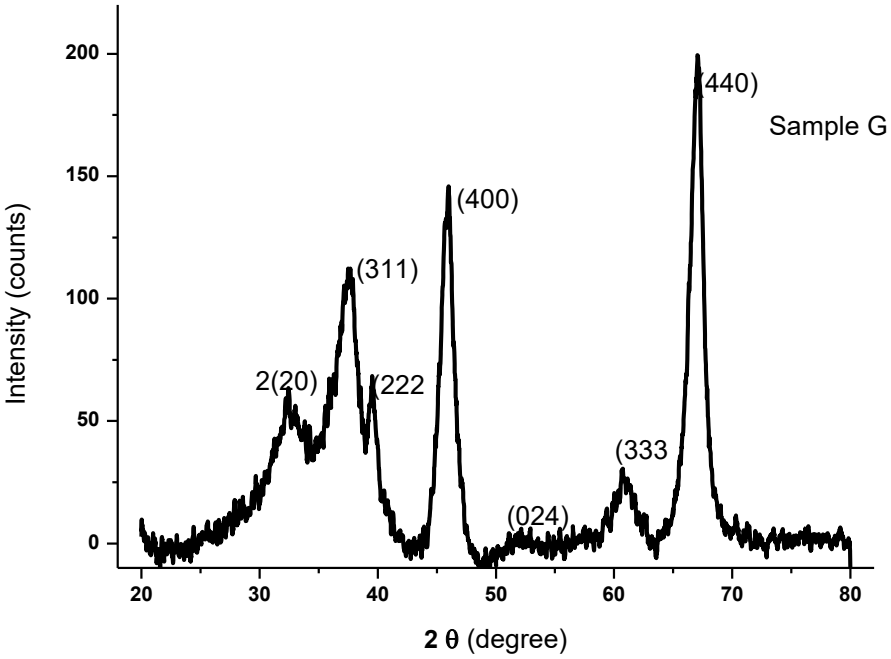
where, 'D' is the crystalline size, 'λ' is the wavelength of CuKα radiation, 'β' is full width half maximum (FWHM) of the diffraction peak and 'θ' is Bragg's angle of X ray diffraction peak

Figure --.2 to Figure --.3 shows the XRD of Samples G and C respectively. These diffuse diffraction peaks in XRD pattern show index regarding γ-Al2O3, which match with JCPDS Card No. 29–0063 [20] and indicating the presence of some amorphous phase in this sample during transformation with no characteristic peaks corresponding to the impurities. The broader diffraction peaks obtained indicated the smaller crystallite size. The observations reported by Hyuk-Joon et al. [21], noted that completion of the most stable phase of α- alumina occurs at high temperature around 1200^0C. The crystallite size, microstrain and dislocation density for the three samples : Sample C, Sample U and Sample G are tabulated in tables 1,2 and 3 respectively.

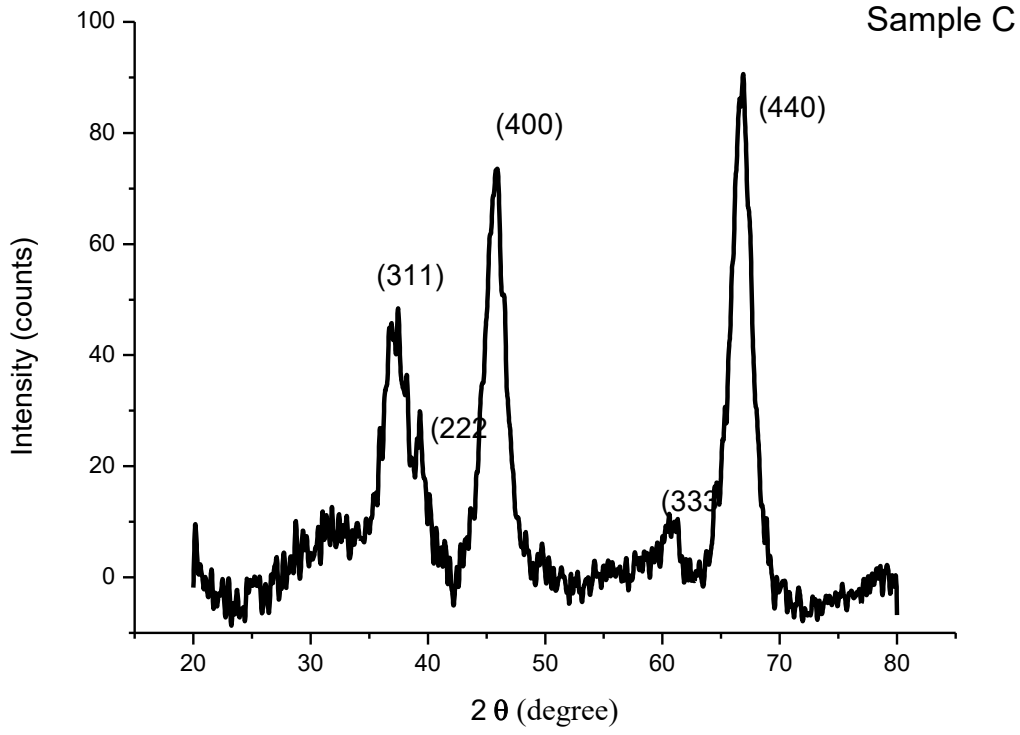
GRAPH I



GRAPH II



GRAPH III



2θ	FWHM	**Grain size , D**	**Dislocation density**	**microstrain**
degree	**degree**	**Nm**		
30.1	5.5	1.562196427	0.409759206	0.089251172
37.48	8.49	1.03202218	0.938905617	0.10919252
45.6	2.29	3.930415297	0.06473261	0.023770052
60.9	4.3	2.238352669	0.199591927	0.031915703
61	3.6	2.67496122	0.139754249	0.02666683
66.65	2.17	4.576136697	0.04775315	0.014400581
	Average	2.669014082	0.300082793	0.049199476

Sample C TABLE I

2θ	FWHM	**Grain size , D**	**Dislocation density**	**Microstrain**
degree	**degree**	**Nm**		
25.312	0.264	32.21208037	0.000963746	0.005129825
34.887	0.256	33.97395554	0.000866379	0.003554925
37.57	0.35	25.04058934	0.001594817	0.004489865
43.076	0.278	32.08715793	0.000971264	0.00307341
52.32	0.303	30.50922488	0.00107433	0.002691577
57.252	0.313	30.20073099	0.00109639	0.002502206
66.31	0.6	16.5182093	0.003665001	0.00400758
67.943	0.29	34.50027324	0.000840146	0.001877993
76.642	0.55	19.22903677	0.002704487	0.00303642
	Average	28.25236204	0.001530729	0.003373756

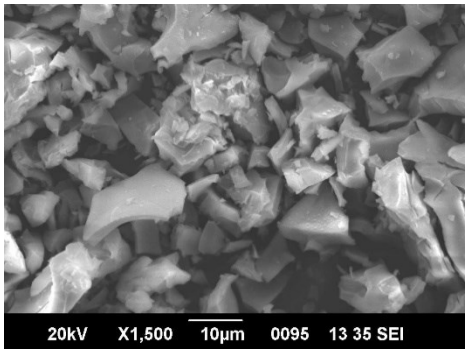
Sample U TABLE II

2θ	FWHM	**Grain size , D**	**Dislocation density**	**Microstrain**
degree	**degree**	**Nm**		
28.3	3.3	2.593029059	0.14872543	0.057113697
32.65	3.93	2.199987163	0.206613981	0.058546266
37.28	3.85	2.274466659	0.193304004	0.049801568
45.76	1.53	5.886239027	0.028861853	0.015819435
61.12	3.06	3.148957349	0.100847803	0.022612623
67.1	1.3	7.65846136	0.017049708	0.008553713
	Average	3.960190103	0.115900463	0.035407883

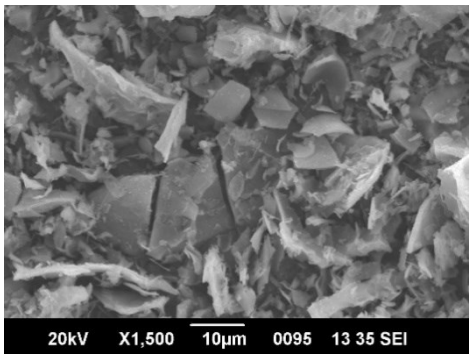
Sample G TABLE III

3.3. SEM ANALYSIS

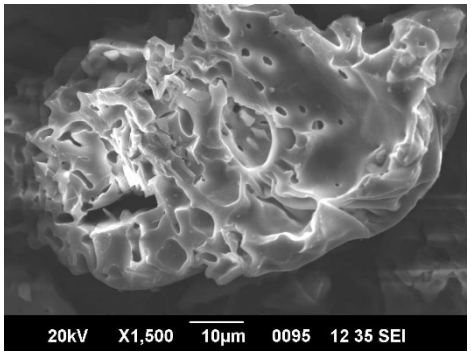
Morphological characterization of powder samples were observed by scanning electron microscopy analysis (SEM) at 5000k and 20,000k magnification. The SEM images are taken after calcinations at 850°C/5h . The images of alumina powder obtained with citric acid , glycine and urea as fuels are displayed in the figure -.1,2.and 3 respectively. Sample U has a coral reef structure with large pores whereas the other two samples have a flake like structure. The flakes in Sample G are thinner and smaller when compared to sample C though they both are in nanosize regime. SEM image of samples confirms the formation of nanosized particles.



Sample C (Fig 8)



Sample G (Fig 9)



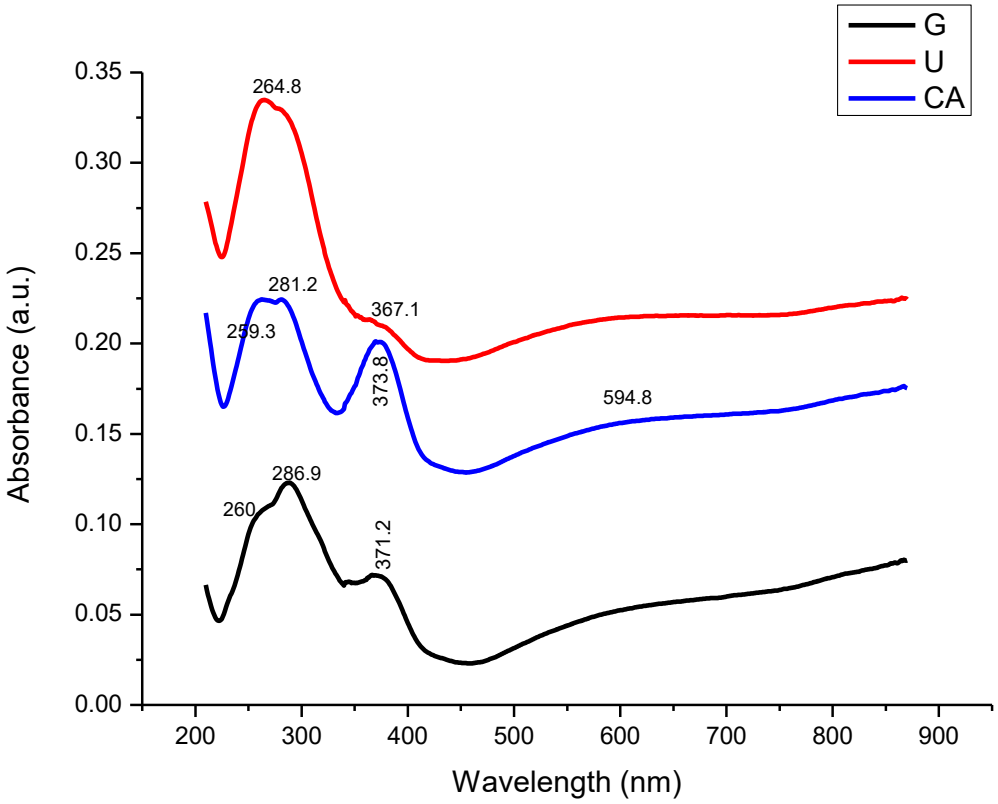
Sample U (Fig 10)

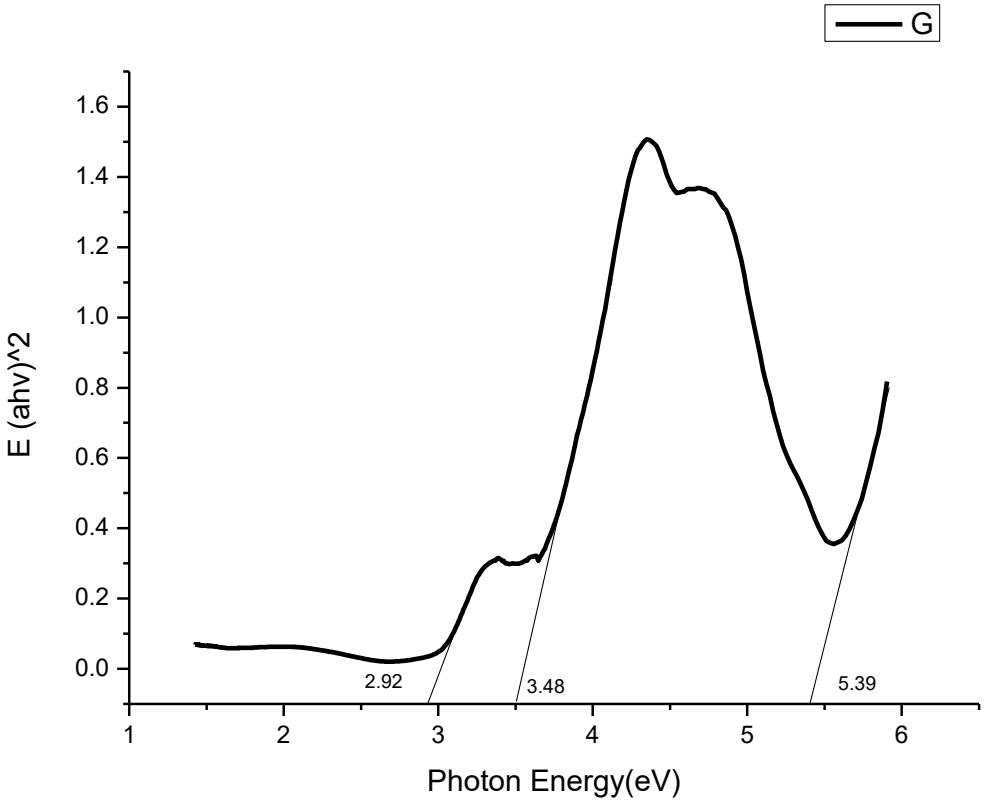
3.4. UV-Vis Method Analysis

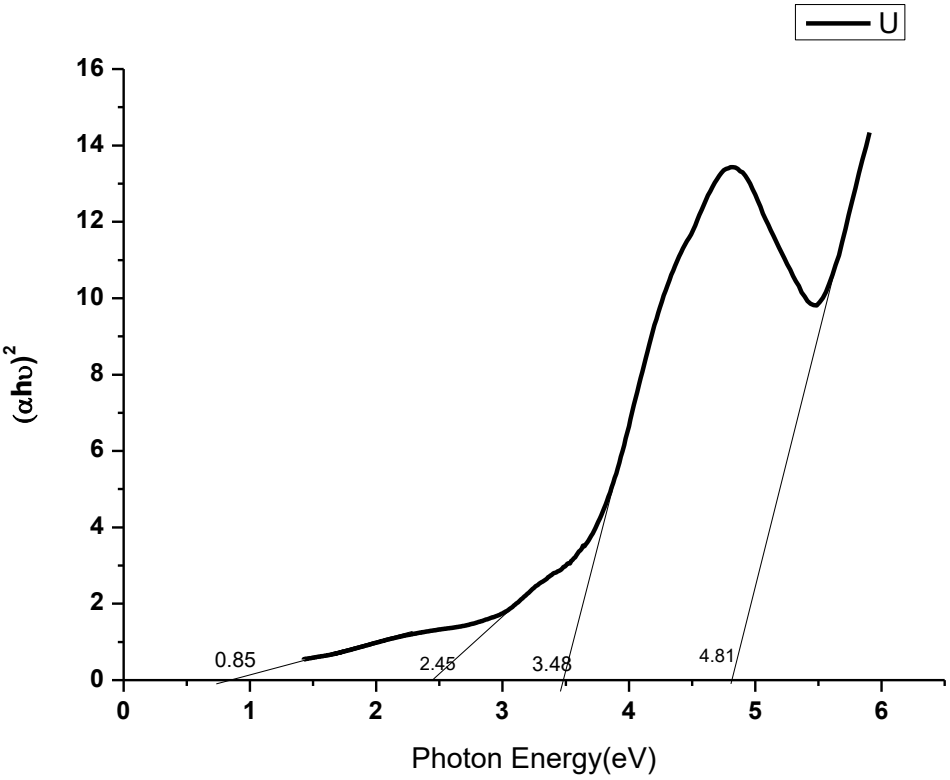
The UV/Vis absorption spectrum was obtained using JASCO V-650 UV visible spectrophotometer. The absorbance spectrum of all the three samples were studied and compared. The samples showed absorbance in both UV and visible region. Figure ---- shows the absorption spectrum of the three samples : Sample U, Sample C and Sample G. The UV absorption ability of Al_2O_3 is related with band gap energy. The spectrum shows multiple absorption peaks which indicates the presence of sub gaps. The photo excitation of electron from valence band gives band gap energy (Eg). The band gap energy (Eg) was estimated by the Wood and Tauc method, according to the following equation,

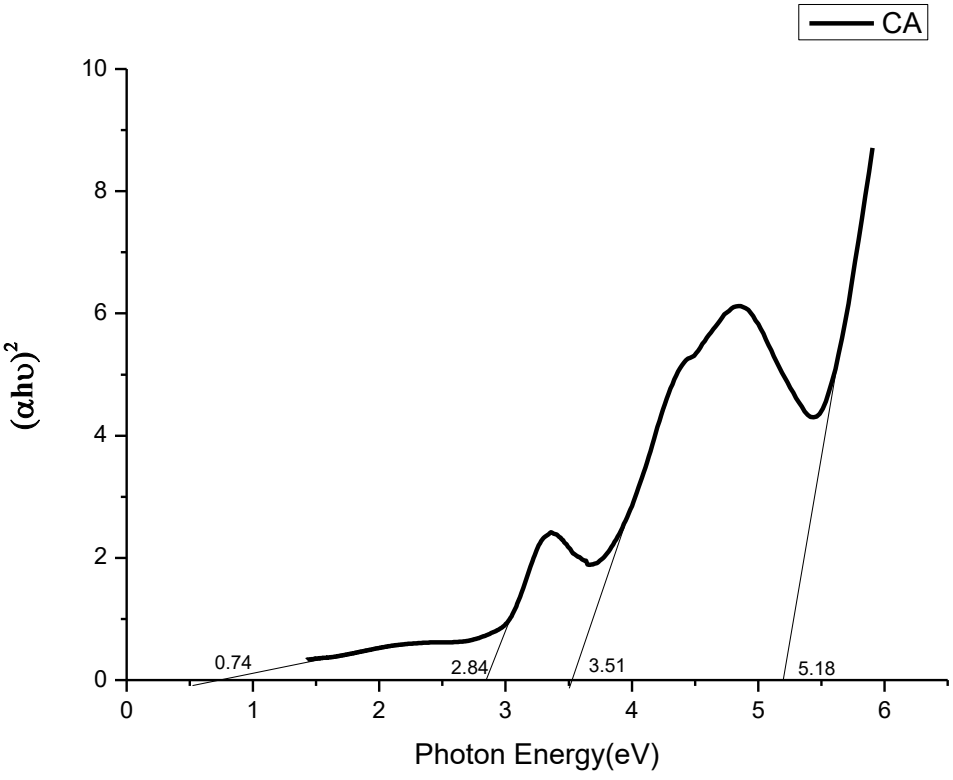
$(h\nu\alpha) = B(h\nu – Eg)^n$, ---------- Equation II

where, 'α' is the absorption coefficient, 'h' is Planck's constant, 'ν' is the frequency, 'Eg' is absorption band gap energy and 'n' is a constant associated to the different type of electronic transitions (n=1/2, 2, 3/2 or 3 for direct allowed, indirect allowed, direct forbidden, indirect forbidden transitions respectively).The $(\alpha h\nu)^2$ Vs hν for the samples were also plotted. Figure----- shows Tauc plots drawn for the samples G,U and C respectively. These results are in good agreement with those previously reported by Varghese et al. and Farahmandjou [23]


G
U
CA
Absorbance (a.u.)
Wavelength (nm)
264.8
281.2
259.3
367.1
373.8
594.8
286.9
260
371.2


G
E (ahv)^2
Photon Energy(eV)
2.92
3.48
5.39


U
16
14
12
10
8
6
4
2
0
(αhυ)²
0.85
2.45
3.48
4.81
0
1
2
3
4
5
6
Photon Energy(eV)


CA
10
8
6
4
2
0
(αhυ)²
0.74
2.84
3.51
5.18
0
1
2
3
4
5
6
Photon Energy(eV)

3.5 Antibacterial Activity

The antibacterial activities of all the three samples were studied in detail. The antimicrobial activity of the nanoparticles is generally known to be a function of the surface area which is in contact with the microorganisms. Reactions take place at the surface of a chemical or material. Hence, the smaller size and the higher surface to volume ratio i.e., larger surface area, enhanced interaction with the microbes is seen. Figure 5.4 shows the comparison of antibacterial activity of Sample U, Sample C ad Sample G against the gram positive bacteria*Staphylococcus aureus* and gram negative bacteria *Escherichia coli*. Table 5.1 shows this comparison. Antimicrobial effects of Al_2O_3 nanoparticles can be attributed to several mechanisms: [22]

1. Induction of oxidative stress due to ROS (reactive oxygen species) generation,
2. Membrane disorganization due to accumulation of nanoparticles in the bacterial membrane and also their cellular internalization,
3. Release of metal ions that may be responsible for antimicrobial activity by binding to the membrane of microorganisms.

The antibacterial mechanism can also be attributed to the destruction of the outer membrane of bacteria by the generated superoxide anion radicals ($\bullet O^{2-}$) as the reactive species. The reactive species such as •OH and $\bullet O^{2-}$ are generated at the catalyst's surface, hence the high surface area is very beneficial for degradation of bacteria. The activity was found to be highest in case of gram negative bacteria. The variation in the sensitivity or resistance to both gram positive and gram negative bacteria populations could be due to the differences in the cell structure, physiology, metabolism or degree of contact of organisms with nanoparticles.

Sample	Staphylococcus aureus	*Escherichia coli*
Sample U	6	8
Sample C	5	7
Sample G	6	8

Table 5.1 Zone of inhibition of Sample U, Sample C and Sample G

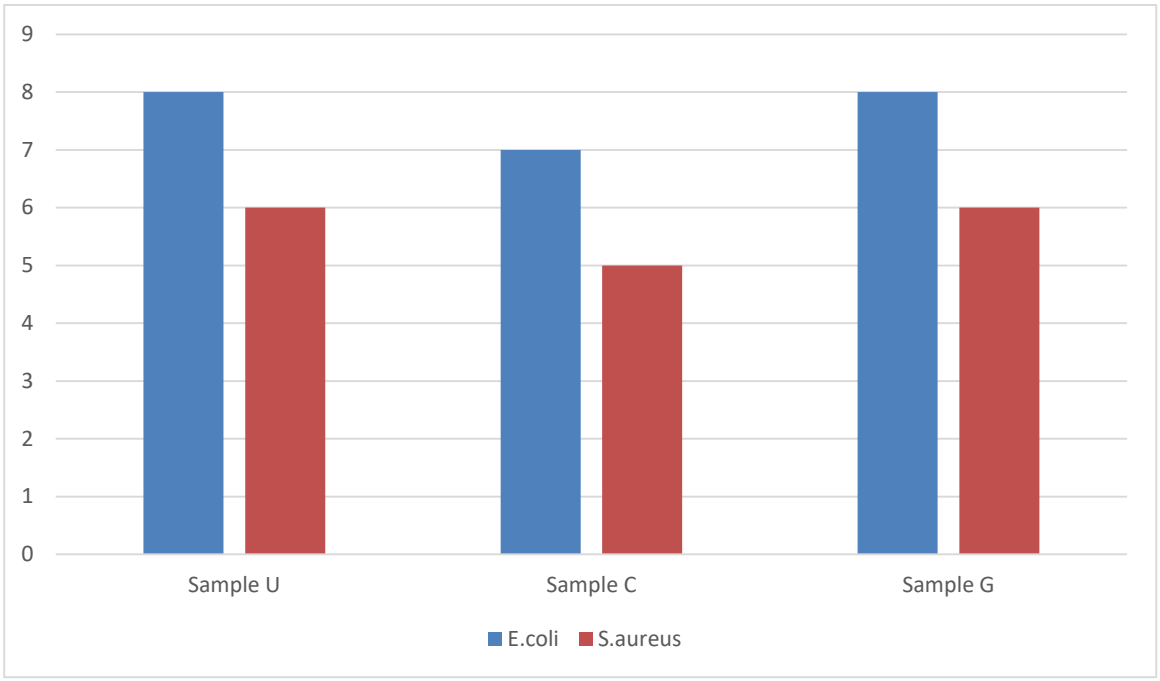


Figure 5.4 Comparison of antibacterial activity of Samples U,C and G nanoparticles on *Escherichia coli* and Staphylococcus aureus

3.6. SUMMARY AND CONCLUSION

The Aluminium oxide (Al_2O_3) nanoparticles were prepared by Solution combustion method using three different fuels urea, glycine and citric acid. The structural, optical, morphological and antibacterial properties of the samples were investigated. Structural characterizations of all the synthesized samples were carried out using XRD. Comparison of XRD‘s of metal oxides with JCPDS confirmed that the formed metal oxide is aluminium oxide. Phase purity and crystallinity of these samples were confirmed from XRD. Crystallite sizes were calculated using Scherrer equation.

X-Ray diffraction patterns confirm the formation of different phases of Al_2O_3 nanoparticles. The formation of aluminium oxide nanoparticles was validated from SEM analysis.

All the samples prepared in this work showed strong UV –visible absorption. Hence these materials could be suitable for anti UV applications such as anti UV glass, anti UV sunscreen, etc. Also, these materials could be a good candidate as photocatalyst for photocatalysis in presence of UV as well as visile light, as antibacterial agents, etc. From band gap calculation it was seen that all of them showed sub gaps. So they are expected to show good photocatalytic activity as they have good absorbance in both UV and visible region with sub bands .Further, these nanoparticles showed good antibacterial activity against gram positive and gram negative bacteria.

3.7. FUTURE SCOPE

In the present work, aluminium oxide is prepared by solution combustion method. Also there are many methods for the preparation of nanoparticle of aluminium oxide. It has so many importance because of wide range of applications such as ceramic materials, biomedical applications like cancer therapy etc. In this project method we used oxidiser to fuel ratio as 1:1.[24] The work can be extended to analyse the dependence of oxidiser to fuel ratio in the formation of the nanoparticles by varying the proportion. Further, the factors affecting the formation of different phases of Al_2O_3 can also be investigated by varying the synthesis parameters like concentration of fuel, concentration of oxidiser, pH of the solution, chemical constituents of the reactants, calcination temperature and time and so on. Antibacterial activity of the samples against one gram positive and one gram negative bacteria was only studied. The analysis can be extended to other bacteria so that the potential of aluminium oxide as an antibacterial agent can be confirmed. The presence of sub bands increase the photocatalytic activity of the photocatalyst. Thus the samples can be further studied for its photocatalytic activities.[25]

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