

CHARACTERISATION AND APPLICATIONS OF ALUMINA

Submitted in partial fulfilment of the requirements for the award of

Bachelor of Science in Chemistry

by

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DEPARTMENT OF CHEMISTRY

SCHOOL OF SCIENCE AND HUMANITIES

SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

(DEEMED TO BE UNIVERSITY)

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April – 2022



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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the bonafide work of **ABI. A (39030002)**, **PRITHIYA.P (39030021)**, **SHOBANA.G (39030034)** who carried out the project entitled **"CHARACTERISATION AND APPLICATIONS OF ALUMINA"** under our supervision from December 2021 to April 2022.

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DECLARATION

We **ABI. A (39030002), PRITHIYA.P (39030021), SHOBANA.G (39030034)** hereby declare that the Project Report entitled **CHARACTERISATION AND APPLICATIONS OF ALUMINA** done by us under the guidance of **Dr. K. CHENNAKESAVULU, M.Sc., Ph.D.**, Associate Professor, Department of Chemistry at **Sathyabama Institute of Science and Technology**, Jeppiaar Nagar, Rajiv Gandhi Salai, Chennai – 600119 is submitted in partial fulfillment of the requirements for the award of Bachelor of Science degree in Chemistry.

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ABSTRACT

Over last few years, there has been a growing attentiveness in researching and developing new antimicrobial agents from origin or source to shoot- out the growth or kill microorganisms. Habitually metal oxide nanoparticles are being endowed with robust antimicrobial properties. The bacterial growth resistance plays a major aspect in the medical domain. Presently, the metal oxide nanoparticles are hopefully utilized as an antimicrobial agent in various fields like pharmaceuticals, drug deliveries, wound dressing, bio fouling etc. Aluminium oxide nanoparticles have wide range of significance in medical field, industries as well as in personal health care products. In this inquest, 0.1nm commercially bought Al_2O_3 nanoparticles was used to study the growth inhibitory effect over two gram negative microorganisms namely *Pseudomonas aeruginosa* and *Klebsiella pneumonia*. Alumina nanoparticles were characterized using FTIR, XRD, TGA and BET. In this present study the bacterial resistance which is harmonious with the concentration of Al_2O_3 nanoparticles is being negotiated.

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LIST OF ABBREVIATION

FTIR	Fourier Transform Infrared Spectroscopy
TGA	Thermo Gravimetric Analysis
XRD	X-Ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 ALUMINA

Aluminum oxide is a compound composed of two aluminum atoms and three oxygen bonded together in a hexagonal close packed (hcp) crystal structure. The common name for the compound is alumina and it has found several applications in the industry due to its abundance and abrasiveness. These are nanosized round particles from an alumina mass. Alumina nanoparticles can be gotten by a few strategies including, pyrolysis, faltering, sol gel and the most normally favored method laser removal. Laser removal is helpful since it very well may be acted in the gas, fluid or even a vacuum climate.

This is invaluable since the assortment of nanoparticles in a vaporous climate is more diligently and lumbering than in a fluid. It is additionally simpler to set determinations for the sort of nanoparticles you want to get. Aluminum nanoparticles are normally found in two states, almost round molded individual particles or as situated filaments. The properties of mass alumina that permit it to be a particularly flexible component in the business are not the same as those of its nanoparticles.[1]

Mass aluminum's enormous measurement doesn't permit it to have a bigger surface of association with its environmental factors. In this way, the utilizations of its utilization can't proficiently be additionally extended to different regions like catalysis or adsorption. Spreading into the field of nanoparticles paints another image for the potential uses of the generally helpful and plentiful compound of aluminum oxide. The lessening in size has different benefits yet the significant factor is the adjustment in the science of the nanoparticles contrasted with their bigger partners that captivates the consolidation of these nanoparticles into numerous fields of science and designing.

The physiochemical properties of the nanoparticles are impacted vigorously by their size and measurements. The game plans of the particles in the nanoparticles are distinctive at whatever point the size is changed due to the designs that describe

the nanoparticles. It is realized that nanoparticles are not point protests and have three unmistakable layers to them. The first and furthest layer is the surface layer. This is included atoms or metal particles that will later on give work the outside of the nanoparticle. The subsequent layer is the shell layer which is altogether not the same as the other two layers. The third and last layer is the center of the nanoparticle which is regularly a portrayal of the fundamental compound recipe of the nanoparticle itself.



Fig:1.1 Alumina

Assurance of the constructions of nanoparticles is a progressing advancement since the strategies applied on a similar framework in mass can't portray the significant highlights introduced when the framework is at the nanoscale. The general physiochemical properties are electronic and optical properties, mechanical properties, attractive properties and warm properties. Electronic and optical properties are cooperative in light of the fact that the retention, emanation and electric conductance all rely upon the electrons and their energy levels.

Nanoparticles show spectra of absorbance and emanation that isn't found in the mass. The mechanical properties like hardness, versatile modulus, attachment, grinding and slender power of the nanoparticles are broadly concentrated to encourage the novel utilization of these particles in the business. Upgraded hardness is used in the assembling of wear safe items, low rubbing is utilized for planning greases that last more and the attachment property is used in molecule expulsion measures. Ultimately, the warm properties of nanoparticles are attached to the sort of

metals that they are included. It is general information that metals like copper and the oxides of aluminum have higher warm conductivities contrasted with the greater part of different solids or liquids of non-metallic mixtures. Expansion of nanoparticles of these metals and metal oxides permits the liquid that was beforehand a terrible warm conduit to have an improved warm conductivity.[3]

Aluminum powder is a fine granular powder produced using Aluminum. In structure of powders, Aluminum is utilized for a few applications like production of slurry, touchy and detonators, thermit measure utilized for fabricate of ferro compounds and for specific welding applications like rails, pyrotechnic to fabricate wafers, shines and other pyrotechnic items; make of aluminum glue, paints and a few powder parts utilized in cars. The main property of aluminum powder to go through a enthusiastic exothermic response when it gets oxidized discovers application in pyrotechnic measure.[1] In foundry, aluminum powder is utilized as a deoxidant and exothermic tapping mixtures to expand the yield of projecting. The aluminum powder is a consumable item. By and by there are four significant coordinated producers of aluminum powder. They are Metal Powder Company, Thirumangalam, INDAL, Mumbai, Khosla Metal Powder Organization, Pune and Arasan Aluminum Ventures, Sivakasi. What's more, there are various limited scope ventures situated in Karnataka, M.P., Maharashtra, Gujarat and Delhi with an introduced limit of 1 ton each day.[4]

In a new report of DSIR, the absolute creation of aluminum powder in the nation has been assessed at in excess of 10,000 MT each year. DGTD has assessed the development of interest between 8 to 10% per annum. Taking everything into account, it tends to be said that creation of aluminum powders of different evaluations and items, for example, aluminum glue is grounded in the country. The aluminum powder industry is of an exceptional size. There is a developing business sector for fare of aluminum powder and glue. Great openings exist in the field of setting up new units in limited scope areas with demonstrated innovation and proper quality direction.

White Fused Alumina is created by combination of high immaculateness Bayer prepared alumina in electric curve heaters at a temperature of around 2,050 degree Celsius. JCT's white melded alumina has the highlights of very much disseminated

molecule size creation, low attractive substance content, high mass thickness and durability, great sturdiness, and high tidiness, and so on The fortified abrasives made by white intertwined alumina are suggested for the crushing of high carbon steel, fast steel and chilled steel. It can likewise be utilized as impacting, lapping and cleaning media and overlaying flooring materials.[6]

Aluminum powders of various molecule sizes (from nano-[as = 0.01–0.5 μm] to micron-sized (as = 1–500 μm) and molecule shapes (circular and chips) are generally utilized in energetics: from rocket drive to self-proliferated high-temperature amalgamation and water-parting hydrogen-creating pieces. In this part the highlights of aluminum powder properties will be talked about alongside their response energy and oxidation conduct in various oxidation media: from frail oxidizers (water and air) to solid oxidizers, for example, rocket fuels.

Alumina, regularly known as aluminum oxide (Al_2O_3), is a latent, scentless, white undefined material frequently utilized in modern pottery. Because of its extraordinary properties, alumina has added to countless life-broadening and society-upgrading applications. It is broadly utilized in the clinical field and current fighting.

Aluminum oxide is a thermally unsteady and insoluble compound that happens normally in different minerals like corundum, a glasslike variation of the oxide, and bauxite, which is considered as its main aluminum metal. The greater part of the aluminum oxide created is utilized to shape aluminum metal. Oxygen regularly catalyzes consumption in response with the metal aluminum. Nonetheless, when fortified with oxygen to frame aluminum oxide, a defensive covering structures and forestalls further oxidation. This adds strength and makes the material less helpless against crumbling.[2]

1.2 BACTERIAL STUDY

1.2.1 Antibacterial Property

There is some concern that antibiotic residues in applied manure may promote the emergence of antimicrobial organisms in the environment. Soil-marketed antibiotics have been found to maintain their antimicrobial properties because both antibiotics inhibited the growth of all bacterial species.

The main characteristics of the abiotic are climate, including temperature, humidity, soil texture, soil composition, salt and pH. Climatic conditions also affect the body of soil organisms as they vary around the world and, in the same areas, during the seasons.

1.2.2 Pseudomonas

Pseudomonas is a common genus of bacteria, which can create infections in the body under certain circumstances. There are many different types of Pseudomonas bacteria. Only a few types can cause an infection. Pseudomonas bacteria tend to live and breed in water, soil, and damp areas. Pseudomonas is a genus of Gram negative, Gamma proteo bacteria, belonging to the family Pseudomonadaceae and containing 191 validly described species

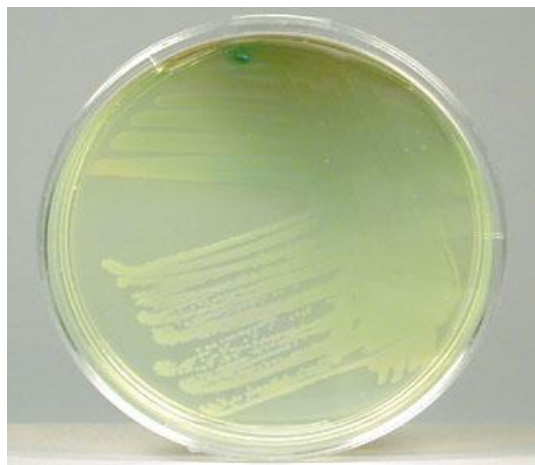


Fig.1.2. Cultured Pseudomonas

Functions of pseudomonas like other members of the Pseudomonas genus, it is known for its metabolic versatility and its ability to colonize a wide range of ecological niche such as rhizosphere, water environments and animal hosts, including humans where it can cause severe infections.[8]

1.2.3 Klebsiella

Klebsiella is a type of bacteria commonly found in nature. In humans, the bacteria are often present in parts of the digestive tract where they do not generally cause problems. Klebsiella is a genus of negative, oxidase negative, rod shaped bacteria with a prominent polysaccharide based capsule. Klebsiella species are found everywhere in nature. This is thought to be due to distinct sub lineages developing specific niche adaptations, with associated biochemical adaptations which make them better suited to a particular environment. They can be found in water, soil, plants, insects and other animals including humans.



Fig.1.3. Cultured Klebsiella

Klebsiella organisms can lead to a wide range of disease states, notably pneumonia, urinary tract infections, sepsis, meningitis, diarrhea, peritonitis and soft tissue infections. Klebsiella species have also been implicated in the pathogenesis of ankylosing spondylitis and other spondyloarthropathies.[9]

After the incubation we should check the bacterial growth in the cultured media. The cloudy appearance in the tubes indicating the presence of bacterial growth.

CHAPTER 2

LITERATURE SURVEY

1.TITLE: Antimicrobial activity of aluminium oxide nanoparticles for potential clinical applications.

AUTHOR: Amitava Mukherjee, Mohammed Sadiq I., Prathna T.C., N. Chandrasekaran.

JOURNAL AND YEAR: 20 May 2014

INFERENCE: The last two decades have seen a drastic increase in bacterial resistance to antibiotics and is a major concern for medical professionals world-wide. Metal oxide nanomaterials are currently the most promising tools applied as antimicrobial agents for diagnosis of diseases, drug delivery systems, sun screens and ceramics in biomedical and pharmaceutical arena. The antimicrobial activity of metal oxide nanoparticles is due to the large surface area which ensures a broad range of reactions with bio-organics present on the cell surface. Alumina nanoparticles have immense commercial applications and are being studied for their antimicrobial behaviour. At near-neutral pH, an electrostatic interaction plays a possible role in toxicity of alumina nanoparticles, due to interaction between its positive surface charge and negatively charged bacterial cells leading to NP adhesion onto bacterial surfaces and decrease in cell viability. This review attempts to elaborate the antimicrobial behaviour of alumina nanoparticles and its potential clinical applications.

2.TITLE: Antibacterial potential of Al₂O₃ nanoparticles against multidrug resistance strains of Staphylococcus aureus isolated from skin exudates.

AUTHOR: Mohammad Azam Ansari • Haris M. Khan • Aijaz A. Khan • Ruchita Pal • Swaranjit Singh Cameotra

JOURNAL AND YEAR: 21 September 2013

INFERENCE: To date very little studies are available in the literature on the interaction of Al₂O₃ nanoparticles with multidrug-resistant strains of Staphylococcus aureus. Considering the paucity of earlier reports the objective of present study was

to investigate the antibacterial activity of Al₂O₃ NPs (150 nm) against methicillin-resistant *S. aureus* and methicillin-resistant coagulase negative staphylococci by various methods. Al₂O₃ NPs were characterized by scanning electron microscopy, high resolution transmission electron microscopy, and X-ray diffraction.

3.TITLE: Synthesis, Characterization And Antibacterial Activity Of Aluminium Oxide Nanoparticles

AUTHOR: Manyasree D., Kiranmayi P., Ravi Kumar R. V. S. S. N.

JOURNAL AND YEAR: International Journal of Pharmacy and Pharmaceutical Sciences, 2017

INFERENCE: In the present study, synthesized alumina (Al₂O₃) nanoparticles were characterized and their antibacterial activity against gram positive and gram negative organisms were studied. In conclusion, aluminium oxide is a good antibacterial agent against both gram positive and gram negative organisms.

4.TITLE: Effects of aluminium oxide nanoparticles on bacterial growth

AUTHOR: Nina Doskocz, Katarzyna Affek, and Monika Załęska-Radziwi

JOURNAL AND YEAR: 2017

INFERENCE: Production and wide application of nanomaterials have led to nanotechnology development but their release to environment and the induction of toxic reactions, affects the natural microbial communities. Therefore, studies on the impact of nanoparticles on microorganisms and environment are required and needed. The aim of this study was to assess the impact of aluminium oxide nanoparticles on the growth of *Pseudomonas putida*. To compare the harmfulness of different forms of aluminium oxide, the ecotoxicity of its macro-forms was also evaluated in the study. Research showed that the exposure to nanoparticles can negatively influence microorganisms.

5.TITLE: Silver-Doped Anodic Alumina with Antimicrobial Properties—Synthesis and Characterization

AUTHOR: Denitsa Kiradzhiyska , Nikolina Milcheva , Tsvetelina Batsalova , Balik Dzhambazov , Nikolay Zahariev and Rositsa Mancheva

JOURNAL AND YEAR: Metals 2022

INFERENCE: The incessant need for the elimination of pathogenic viruses and multi-drug resistant bacteria has been a critical issue during recent decades, and requires the creation of new antimicrobial materials. Our study describes the production of silver-modified anodic alumina substrates by two methods, and estimation of their bactericidal activity. Aluminum oxide coatings were obtained via an anodization process of low-purity aluminum in an acidic bath for different time periods. The realization of silver infiltration into the pores of the alumina layers was carried out employing two different routes- electrochemical deposition, and in situ thermal reduction. The obtained films were characterized using scanning electron microscopy (SEM).

CHAPTER 3

AIM AND SCOPE

The alumina sample was characterized for the following,

- To know the functional groups present in the sample by FTIR
- To identify the presence of minerals by XRD studies
- To evaluate the thermal stability by TGA
- To study the anti- bacterial properties using gram negative bacteria for the alumina.

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS

4.1.1 Preparation of Al₂O₃ nanoparticles dispersion:

The obtained Al₂O₃ nanoparticles were made up with five different concentrations as 10, 50, 100, 200 and 300 µg/L by using ultrasonic vibrator for about 15 minutes. The alumina nanoparticle suspensions were dissolved using DMSO as it dissolves both polar and non-polar compounds. Different concentrations were prepared to determine their minimum inhibitory concentrations by using agar well diffusion method.

4.1.2 Anticipation of Agar solution:

Muller Hinton Agar (MHA) solid media was dissolved completely in distilled water. This prepared solution was kept in steam autoclave for more than 45 mins at temperature between 121- 134°C for sterilization. Along with this empty petri dishes were also kept for sterilization. After the heating process, the sterilized media was kept to cool-down for around 10- 15 minutes.

4.1.3 Bacterial culture:

Inoculation of two different gram negative bacteria *Pseudomonas aeruginosa* and *Klebsiella pneumonia* were used to appraise the antibacterial activity of Al₂O₃ nanoparticles. These bacteria were inoculated in two different vial glass bottles containing agar solution. This was kept in vibrator for nearly 10 minutes and then in the incubator over-night.

4.1.4 Determination of Agar well diffusion method:

This bacterial culture was then spread over six petri dishes with each bacterium in the plates each. Once it got solidified, the wells were made using 6mm diameter tips into the agar plates containing inoculums. Subsequently, different volumes of the as-prepared alumina nanoparticle solution were introduced into the

well. Then the plates were incubated at 35 °C over- night. DMSO at 10% concentration was employed as a controlled medium. Finally, the antibacterial activity was disclosed by measuring the inhibition zone including the well diameter appeared after the incubation period.

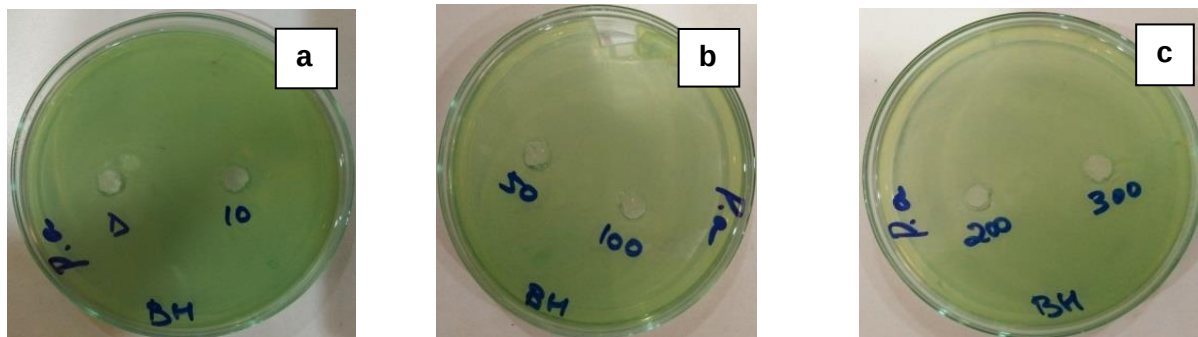


Fig:4.1. Alumina solution in *Pseudomonas*

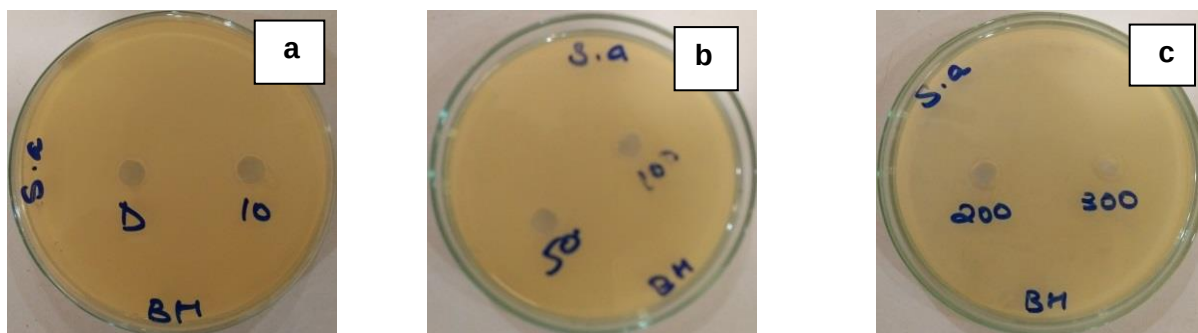


Fig:4.2. Alumina solution in *Klebsiella*

4.2 CHARACTERIZATION TECHNIQUES

The chemical, physical and biological characteristics of alumina are essential to identify the specific properties presence.

4.2.1. Fourier Transform Infrared Spectroscopy - (FTIR)

FTIR is a technique used to obtain an infrared spectrum of absorption or emission of a solid, liquid or gas. An FTIR spectrometer simultaneously collects

high-resolution spectral data over a wide spectral range. This confers a significant advantage over a dispersive spectrometer, which measures intensity over a narrow range of wavelengths at a time. Infrared spectroscopy exploits the fact that molecules absorb frequencies that are characteristic of their structure. These absorptions occur at resonant frequencies, i.e., the frequency of the absorbed radiation matches the vibrational frequency. The energies are affected by the shape of the molecular potential energy surfaces, the masses of the atoms, and the associated vibronic coupling. In particular, in the Born–Oppenheimer and harmonic approximations, i.e., when the molecular Hamiltonian corresponding to the electronic ground state can be approximated by a harmonic oscillator in the neighbourhood of the equilibrium molecular geometry, the resonant frequencies are associated with the normal modes corresponding to the molecular electronic ground state potential energy surface.

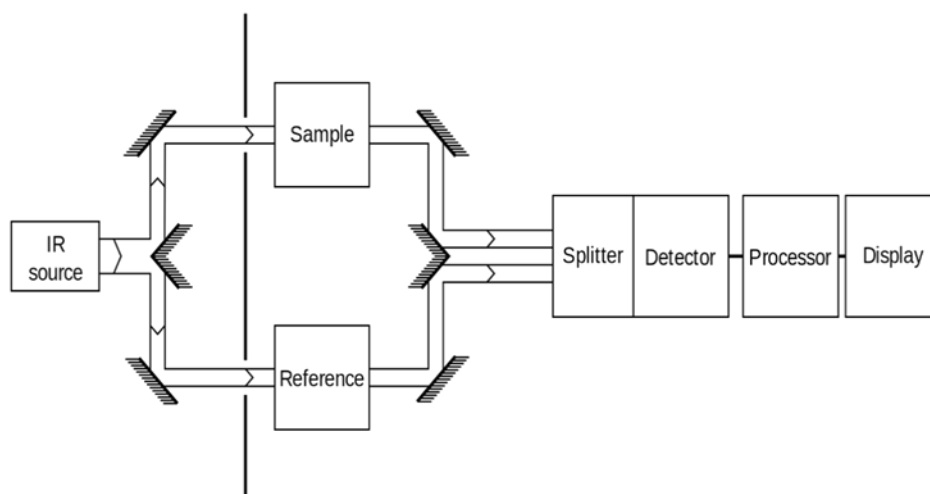


Fig.4.3. FTIR absorption spectrometer

In short, the IR spectrum is divided into three wavenumber regions: far-IR spectrum ($<400\text{ cm}^{-1}$), mid-IR spectrum ($400\text{--}4000\text{ cm}^{-1}$), and near-IR spectrum ($4000\text{--}13000\text{ cm}^{-1}$). The mid-IR spectrum is the most widely used in the sample analysis, but far- and near-IR spectrum also contribute in providing information about the samples analysed. This study focused on the analysis of FTIR in the mid-IR spectrum. The mid-IR spectrum is divided into four regions: (i) the single bond region

(2500-4000 cm^{-1}), (ii) the triple bond region (2000-2500 cm^{-1}), (iii) the double bond region (1500-2000 cm^{-1}), and (iv) The fingerprint region (600-1500 cm^{-1}).

Identifying the fingerprint region (600-1500 cm^{-1}) This area is typically specific and unique. See detailed information in Table 1. But several identifications can be found: (1) Between 1000 and 880 cm^{-1} for multiple band absorption, there are absorption bands at 1650, 3010, and 3040 cm^{-1} . (2) For C-H (out-of-plane bending), it should be combined with absorption bands at 1650, 3010, and 3040 cm^{-1} which show characteristics of compound unsaturation. (3) Regarding vinyl-related compound, about 900 and 990 cm^{-1} for identifying vinyl terminals ($-\text{CH}=\text{CH}_2$), between 965 and 960 cm^{-1} for trans unsaturated vinyl ($\text{CH}=\text{CH}$), and about 890 cm^{-1} for double olefinic bonds in single vinyl ($\text{C}=\text{CH}_2$). (4) Regarding aromatic compound, a single and strong absorption band is around 750 cm^{-1} for ortho and 830 cm^{-1} . [10]

4.2.2. Thermo Gravimetric Analysis – (TGA)

Thermo Gravimetric Analysis or Thermal Gravimetric Analysis (TGA) is a method of thermal analysis in which the mass of a sample is measured over time as the temperature changes.



Fig 4.4. Thermo Gravimetric Analyzer

This measurement provides information about physical phenomena, such as phase transitions, absorption, adsorption and desorption; as well as chemical phenomena including chemisorption's, thermal decomposition, and solid-gas reactions (e.g., oxidation or reduction).

The upper temperature used for TGA is normally 1000 °C. Quantitative analysis is done from the thermal curve by measuring the loss in mass m . [12]

4.2.3. X-Ray Diffraction Analysis – (XRD)

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. It is used to study the structure, composition, and physical properties of materials. XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that leave the material (X-ray diffractometer).

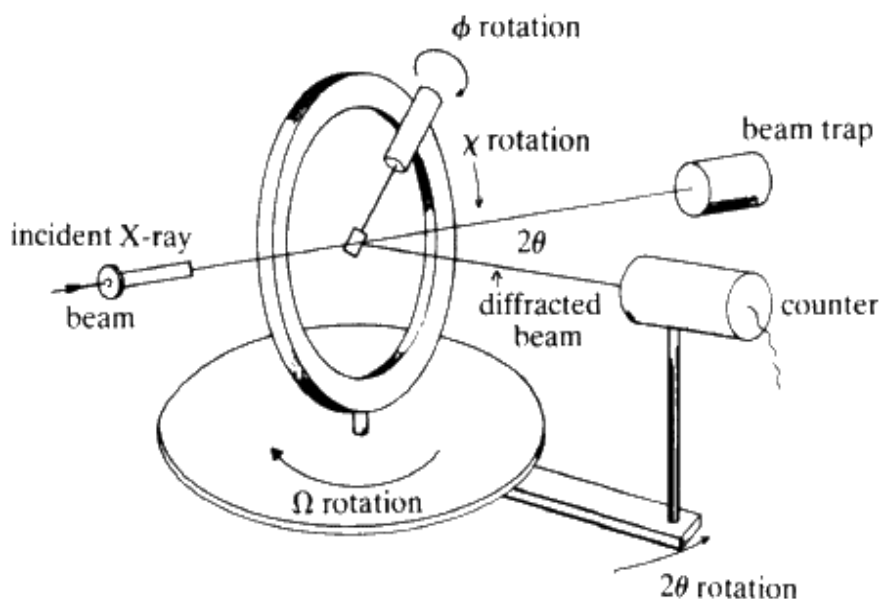


Fig 4.5. X-ray Diffractometer

CHAPTER 5

RESULTS AND DISCUSSION

As a outcome of this study, various characterization techniques like Fourier Transform Infrared Spectroscopy(FTIR), X-Ray Diffraction(XRD), Thermal Gravimetric Analysis(TGA) are outlined in this study.

5.1. Fourier Transform Infrared Spectroscopy (FTIR):

IR spectrum of the sample was recorded using a Thermo Mattson Satellite FT-IR spectrophotometer. The IR spectrum of the product complex shows peaks.

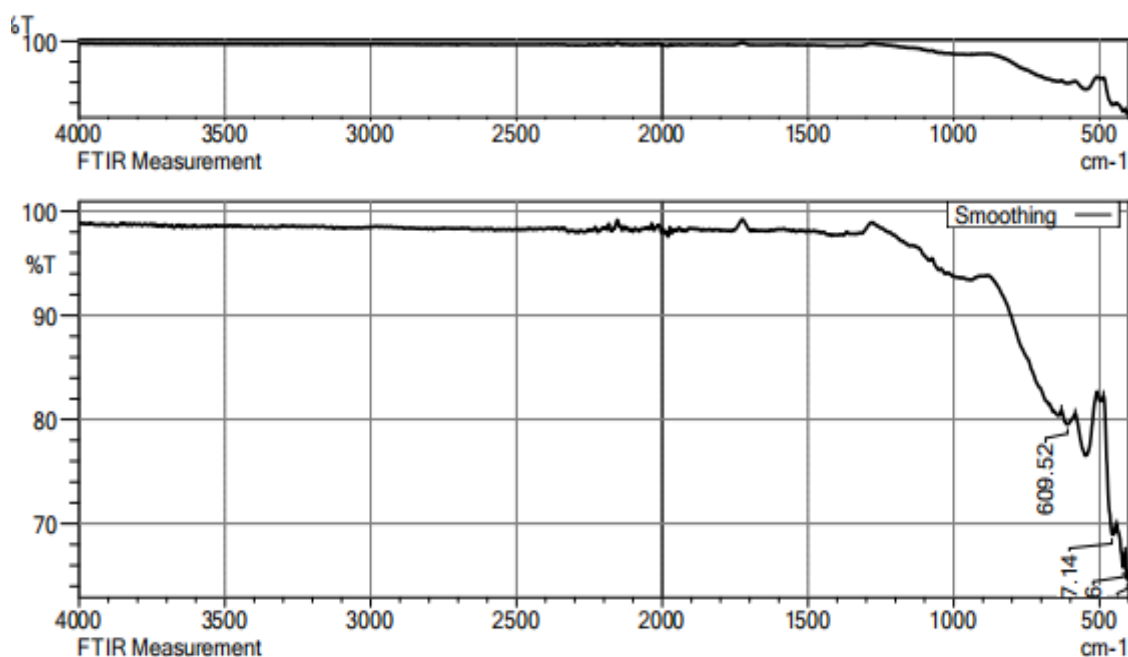


Fig. 5.1. FTIR peak of Alumina

	Peak	Intensity	Corr. Intensity	Base (H)	Base (L)	Area	Corr. Area	Comment
1	405.06	64.65	1.57	411.81	400.24	397.960	8.700	
2	418.56	65.77	2.08	441.71	411.81	964.159	25.021	
3	457.14	68.99	2.21	486.07	452.32	879.887	63.539	
4	609.52	79.53	1.21	630.73	583.48	944.754	34.032	

Table: 5.1.Spectral data of Alumina

The characterized nano and buk sample of alumina was subjected for Fourier Transform Infrared Spectroscopy (FTIR) spectrum ranging from 4000 to 500 cm^{-1} to find the functional groups presents in it. The absorption spectrum of nano alumina possesses a very broad band in wavenumber range from 500-850 cm^{-1} .

5.2. X – Ray Diffraction:

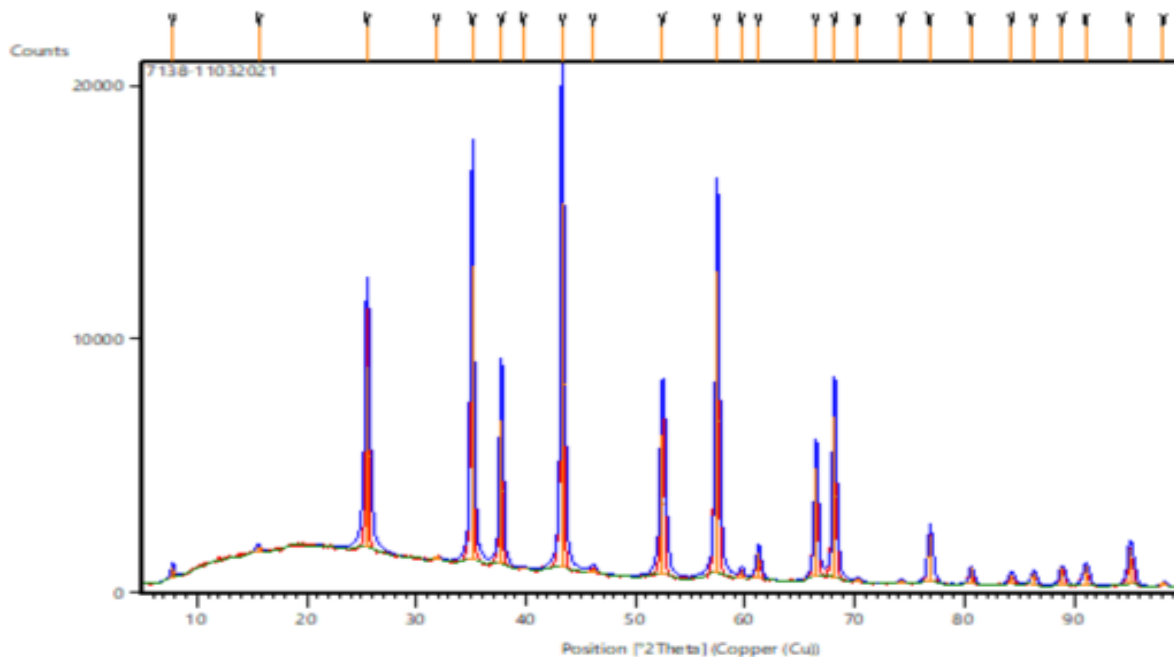


Fig.5.2 XRD of Alumina

There are sharp peaks in nano alumina reflecting the alpha phase of alumina and it is confirmed that this is the phase transition temperature.

5.3. Thermo Gravimetric Analysis (TGA)

Thermo Gravimetric Analysis of clay sample was done in Nitrogen (N_2) atmosphere. It is observed that the sample was thermally highly stable. It reveals that the 90% of the material is stable even above 900 $^{\circ}\text{C}$.

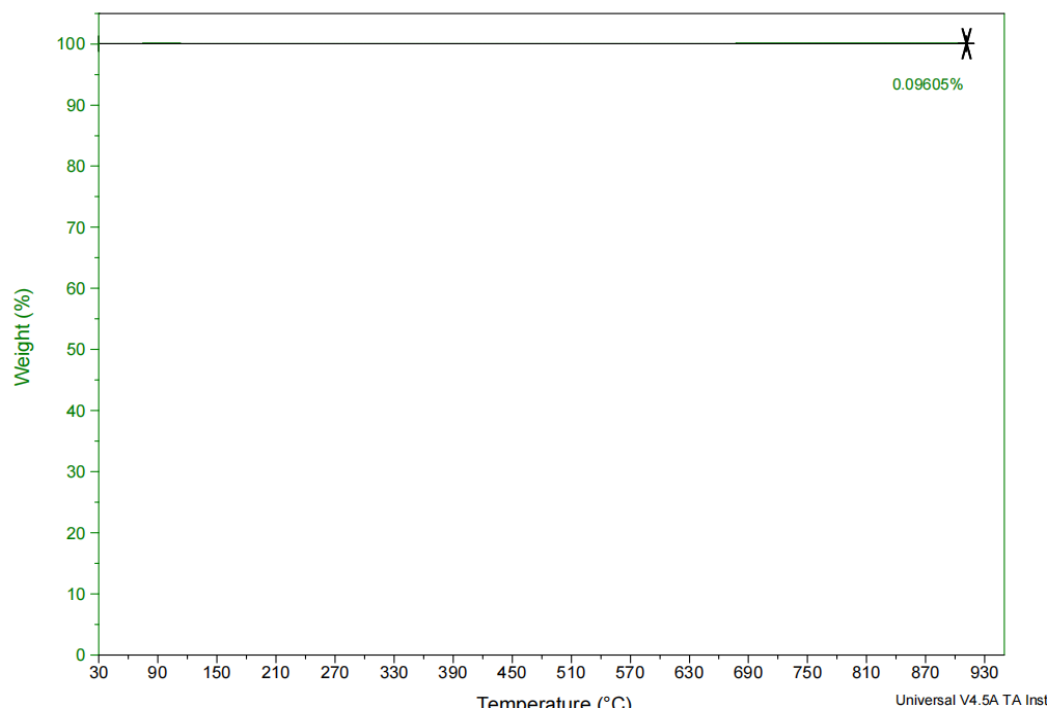


Fig.5.3 TGA of Alumina

CHAPTER 6

CONCLUSION

The tests of antimicrobial properties against two gram negative bacteria *Pseudomonas aeruginosa* and *Klebsiella pneumonia* indicate that surface antibacterial layer of alumina possesses excellent antimicrobial properties and the maximum value of the antibacterial rate can reach 99.9% for both the gram negative bacteria.

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