

**SYNTHESIS AND CHARACTERIZATION OF METAL
NANOPARTICLES USING SILICA AEROGELS FOR WATER
PURIFICATION**

Submitted in partial fulfillment of the requirements of the award of
Bachelor of science Degree in Microbiology

by

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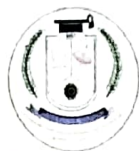
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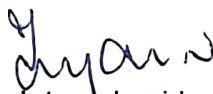
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BONAFIDE CERTIFICATE

This is to certify that this Project Report is the Bonafide work of **K HARIHARAN (41300012)**, **YOGITHA S (41300053)** who carried out the project report entitled "**SYNTHESIS AND CHARACTERIZATION OF METANANOPARTICLES USING SILICA AEROGELS FOR WATER PURIFICATION**" Under my supervision from November 2023 to April 2024.



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ABSTRACT

This research project focuses on the synthesis and characterization of silica aerogels embedded with metal nanoparticles for water purification applications. The study aims to develop advanced materials that can effectively remove contaminants from water, addressing the global challenge of water pollution. The synthesis process involves incorporating metal nanoparticles, such as silver, into the silica aerogel matrix using a hydrothermal method. Characterization techniques including scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) are employed to analyze the structural and chemical properties of the synthesized materials. The performance of the metal nanoparticle-based silica aerogels in water purification is evaluated through adsorption studies, microbial disinfection tests, and analysis of water quality parameters. The findings from this research contribute to the development of efficient and sustainable materials for improving water quality and ensuring access to clean drinking water globally.

KEY WORD: Water purification, Silver Nano particle, Silica aerogels, SEM, X-ray diffraction, FTIR .

TABLE OF CONTENT

CHAPTER NO	TITLE	PAGE NO
1	INTRODUCTION	1
	1.1 Introduction to Metal Nanoparticles	1
	1.2 Silver Nanoparticles	1
	1.3 Understanding silica aerogel: properties and applications	2
	1.4. Significance of water purification	2
	1.5. Research in Nanoparticle-Silica Composite Materials	2
2	LITERATURE SURVEY	4
3	AIM AND OBJECTIVES	11
	3.1 Aim	11
	3.2 Objectives	11
4	Materials and Methods	12
	4.1 MATERIALS REQUIRED	12
	4.2 COLLECTION OF BIOWASTE	12
	4.2.1 Preparation of corn husk ash.	13
	4.3 SYNTHESIS OF SILVER NANOPARTICLES	13
	4.3.1 <i>Preparation of Solutions</i>	13
	4.3.2 <i>Experimental Setup</i>	14
	4.3.3. <i>Synthesized AgNPs</i>	14
	4.3.4 <i>Characterization</i>	14
	4.4 SYNTHESIS OF SILICA AEROGEL USING SOL-GEL METHOD	15
	4.4.1 <i>Preparation of Sodium Silicate Solution</i>	15
	4.4.2 <i>Filtration</i>	15

4.4.3 <i>pH Adjustment</i>	15
4.4.4 <i>Gel Formation</i>	15
4.4.5 <i>Centrifugation</i>	16
4.4.6 <i>Drying</i>	16
4.4.7 <i>Crushing and Storage</i>	16
4.4.8 <i>Characterization</i>	16
4.5 SYNTHESIS OF SILVER/SILICA-NANO COMPOSITE USING HYDROTHERMAL METHOD	17
4.5.1. <i>Prepared Ammonium Chloride Solution</i>	17
4.5.2. <i>Added Surfactant Solution</i>	17
4.5.3. <i>Added Anhydrous Ethanol</i>	17
4.5.4. <i>Added Sodium Silicate Solution</i>	17
4.5.5. <i>Added Silver Nitrate and PVP Solution</i>	17
4.5.6. <i>Introduced Ascorbic Acid</i>	17
4.5.7. <i>Applied Ultrasonic Treatment</i>	18
4.5.8. <i>Autoclaved</i>	18
4.5.9. <i>Cooled and Freeze-Dried</i>	18
4.6 SOLATION OF BACTERIA FROM CONTAMINATED WATER	18
4.7 GRAM-STAINING	19
4.7.1 <i>Prepare a Bacterial Slide</i>	19
4.7.2 <i>Fixation</i>	19
4.7.3 <i>Staining Process</i>	19
4.7.4 <i>Decolorization</i>	19
4.7.5 <i>Counterstaining</i>	20
4.7.6 <i>Drying and Mounting</i>	20
4.7.7 <i>Microscopic Examination</i>	20
4.8 MEDIA COMPOSITION	21
4.9 Antimicrobial susceptibility test	22

	4.9.1 steps	22
	4.10 Characterization	23
	4.10.1 scanning electron microscope (SEM)	23
	4.10.2 UV SPECTROSCOPY	24
	4.10.3 FTIR	25
	4.10.4 XRD	27
	4.10.5 water analysis	28
	RESULT AND DISCUSSION	31
	5.1 Appearance of Corn Husks after Sterilization and Air Drying	31
	5.2. Progression of Corn Husk to Corn Husk Ash Preparation	32
	5.3. Visual Confirmation of Silver Nanoparticles Synthesized via Chemical Method	33
5	5.4. UV-Visible Spectroscopy Analysis of Silver Nanoparticles-]	34
	5.5. Scanning Electron Microscopy (SEM) Analysis of Silver Nanoparticles	36
	5.6. Visual Documentation of Thermochemical Conversion and Sol-Gel Products in Biogenic Silica Extraction from Cornhusk	37
	5.7. FOURIER-TRANSFORM INFRARED SPECTROSCOPY:	38
	5.8 XRD patterns of extracted silica xerogel-	40
	5.9 Visual images of the product during the extraction of biogenic- hydrothermal silver/silica-nanocomposite from corn husk	41
	5.10 Wastewater Analysis	42
	5.9 Antimicrobial susceptibility test	43
	5.10 Isolation of bacteria	44
	Summary and conclusion	46

	6.1 Summary and conclusion	46
6	REFERENCE	47

LIST OF ABRIVATIONS

ACRONYMS

AgNPs

FTIR

SEM

XRD

TMOS

TEOS

CCA

EXPANSION

Silver nano particle

Fourier Transform Infrared Spectroscopy

Scanning Electron Microscopy

X-ray diffraction

Tetramethyl Orthosilicate

Tetraethyl Orthosilicate

Corn Cob Ash

LIST OF FIGURES

FIGURE NO	DESCRIPTION	PAGE NO
4.7.7	Microscopic image of gram-negative bacteria	20
5.1	Depicts the appearance of corn husks following the process of sterilization and subsequent air drying at room temperature.	31
5.2	Process of preparing ash from corn husk	32
5.3	Silver nano particle	33
5.4	Sample B: AgNPs: Peak: $\lambda = 390$ nm and Range: 380-400 nm; Sample C: AgNPs+PEG: Peak: $\lambda = 390$ nm and Range: 380-400 nm	34
5.5	SEM image of silver nano particle	36
5.6	Visual images of the direct thermochemical conversion and sol–gel products during the extraction of biogenic silica from corn husk	37
5.7	FTIR graphical representation of sol-gel	38
5.8	XRD patterns of extracted silica xerogel	40
5.9	<i>The nanocomposite solution before and after autoclave for 5hrs</i>	41
5.11.1	Antibacterial activity of gel, nano particle and nano composite	43
5.11.2	Graph of antimicrobial activity. Data is represented as mean $\pm$ SD. $p=0.05$, $p\leq 0.0005$. $n=3$.	43
5.12	Isolation of bacteria from wastewater in different agars	44

LIST OF TABLES

TABLE NO	DISCRIPTION	PAGE NO
4.8.1	NUTRIENT AGAR-MEDIUM COMPOSITION	21
4.8.2	EOSIN METHYLENE BLUE AGAR COMPOSITION	21
4.8.3	LURIA-BERTANI-(LB) MEDIUM COMPOSITION	21
4.8.4	MAC-CONKEY MEDIUM COMPOSITION	22
5.10	PROPERTIES ANALYSIS OF WEAST WATER	42

CHAPTER 1

INTRODUCTION

1.1 Introduction to metal nanoparticles

Nanotechnology has revolutionized various fields by enabling the synthesis and manipulation of nanoparticles, typically ranging from 1 to 100 nanometers (nm) in size. These nanoparticles possess unique properties, such as a high surface-to-volume ratio and tunable reactivity, making them highly attractive for applications in diverse domains including molecular biology, medicine, and material science (Jamkhande *et al.* 2019 .,). The term 'nanoparticle' originates from the Greek word 'nano,' meaning 'dwarf or small,' indicating their diminutive size of 10^{-9} meters or 1nm.

Metallic nanoparticles, such as gold, silver, and platinum, have garnered significant interest due to their remarkable properties and potential applications. They exhibit enhanced reactivity and functionality compared to their bulk counterparts, rendering them valuable in catalysis, composite materials, biomedical devices, and sensor technologies. For instance, the catalytic activity of gold nanoparticles in CO oxidation, discovered by Haruta in 1987, revolutionized the field of nanocatalysis, redirecting focus towards smaller nanoparticles with enhanced catalytic performance.

1.2 Silver nanoparticles

Among metallic nanoparticles, silver nanoparticles (AgNPs) stand out for their unique optical, electronic, and antibacterial properties. These characteristics have propelled their utilization in various applications such as biosensing, photonics, electronics, antimicrobial treatments, and more. The broad-spectrum antimicrobial activity of AgNPs has led to their incorporation into textiles, medical devices, and water purification systems, highlighting their potential in combating microbial contamination (Astruc & Didier *et al.*, 2020).

The synthesis of AgNPs employs diverse methods including chemical, physical, and biological routes, each offering distinct advantages in terms of control over size, shape, and surface properties. Biological synthesis methods, utilizing non-toxic molecules from living organisms, have emerged as eco-friendly alternatives to traditional chemical methods, paving the way for safer biomedical applications of AgNPs (Wei, Liuya, *et al.*, 2018).

1.3. Understanding silica aerogel: properties and applications

Aerogels, another class of remarkable materials, are dried gels with exceptionally high porosity and surface area. Among aerogels, silica aerogels stand out for their low density, high porosity, excellent thermal insulation, and versatile applications in various fields (Pierre & Alain C *et al.*, 2002). The synthesis of silica aerogels has undergone significant advancements in recent decades, facilitated by innovations in sol-gel chemistry and drying techniques. (A. Soleimani Dorcheh *et al.*, 2008)

1.4. Significance of water purification

Water purification is a critical global challenge, necessitating the development of efficient and sustainable technologies (Weber's (2022)). Nanotechnology has played a pivotal role in revolutionizing water purification methods by leveraging the unique properties of nanomaterials for enhanced adsorption, catalysis, and disinfection. Silver nanoparticles, in particular, have demonstrated efficacy in water treatment applications, offering a promising solution to microbial contamination. (Ahmed & Toqeer *et al.*, 2014).

1.5. Research in Nanoparticle-Silica Composite Materials

Integration of silver nanoparticles into silica matrices has led to versatile nanocomposites with applications in theranostics, fluorescence enhancement, electron field emission, and surface-enhanced spectroscopies (ref). These composite materials exhibit synergistic properties derived from both silver nanoparticles and silica, showcasing their potential across various technological domains. (Qu *et al.*, 2019)

In conclusion, the synthesis and characterization of metal nanoparticle-based silica aerogels represent a significant advancement in the field of water purification technology, offering a sustainable and efficient solution for addressing water quality challenges. Through systematic investigation and optimization, these nanocomposite materials hold great (Das *et al.* 2014) promise for mitigating the adverse effects of water pollution and ensuring access to clean and safe drinking water worldwide.

CHAPTER 2

LITERATURE SURVEY

Li et al., (2020) Nanoparticles have emerged as a significant area of research due to their unique physical and chemical properties, making them highly versatile for various applications. These nanoscale materials, typically ranging from 1 to 100 nanometers in size, exhibit distinct characteristics such as high surface area-to-volume ratio, quantum effects, and enhanced reactivity compared to bulk materials. One prominent type of nanoparticle is silica nanoparticles, which are composed of silicon dioxide (SiO₂) and have garnered attention for their exceptional properties, including biocompatibility, optical transparency, and thermal stability. Silica nanoparticles find wide-ranging applications in fields such as nanomedicine, catalysis, electronics, and environmental remediation.

(Zhao, S &, Liu. 2020)-Silica aerogels are highly porous materials known for their lightweight, low thermal conductivity, and remarkable surface area. These unique properties make them promising candidates for various applications such as insulation, catalysis, sensors, and biomedical devices. The synthesis of silica aerogels involves the sol-gel process, where a silica precursor undergoes hydrolysis and condensation to form a gel, followed by solvent removal to produce the aerogel structure. The structural and textural properties of silica aerogels, including pore size distribution, surface area, and porosity, are crucial factors influencing their performance in different applications. Characterization techniques such as scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD) are commonly used to analyze the morphology, chemical

composition, and crystal structure of silica aerogel. In recent years, research efforts have focused on enhancing the properties and functionalities of silica aerogels through various modifications, such as doping with metal oxides, incorporating nanoparticles, and surface functionalization.

(Khan et al., 2020) Water purification is a critical process with significant implications for public health, environmental sustainability, and socio-economic development. Access to clean and safe drinking water is essential for human well-being and plays a pivotal role in preventing waterborne diseases and improving overall quality of life

(Chen et al., 2021) The development and implementation of efficient water purification technologies are paramount, particularly in regions facing water scarcity, pollution challenges, and the impacts of climate change

(Kumar et al., 2019) Purifying water involves the removal of contaminants, such as pathogens, chemicals, heavy metals, and pollutants, to meet regulatory standards and ensure water safety for consumption and various domestic, industrial, and agricultural uses

(Lok et al., 2019) The advancements in nanotechnology have spurred significant interest in the utilization of nanoparticles for water treatment applications. Various studies have highlighted the potential of nanoparticles such as silver nanoparticles (AgNPs), titanium dioxide nanoparticles (TiO₂NPs), and iron oxide nanoparticles (Fe₃O₄NPs) in addressing water quality challenges. For instance, **AgNPs** have been extensively studied for their antimicrobial properties, making them effective agents for disinfection and microbial control in water treatment systems.

Punia, Pinki, et al., (2021) Nanotechnology deals with very small size (1–100 nm) particles of different types of materials. Various studies are being carried out for contaminated treatment using nanoparticles. Further nanoadsorbents,

nanomembranes, bioactive nanoparticles, nano enzymes, nanometals, magnetic nanoparticles, photocatalysts, etc. are recently developed nanomaterials for the treatment of wastewater through nanoparticles enhanced filtration methods. Nanomaterials such as nanoferrites, which are classified into two categories i.e. hard and soft ferrites depending on their magnetic coercivity, are extensively used in

contaminated water treatment. Nanoferrites are further categorized into four types based on their crystal structure i.e. Spinel, Garnet, Hexaferrite, and Orthoferrite. The unit cell of a Spinel structured ferrite, Garnet structure of yttrium iron garnet is shown in and Hexaferrite structure of barium is shown in. A special class of nanoferrites are developed having a core-shell structure where the specifically functionalized shell provides a specific purpose and the magnetic core facilitates magnetic separation.

MalekiHajar.,et.al.,(2019) Nanostructured solid silica networks are formed through the hydrolysis and condensation process of silica precursor molecules, resulting in the creation of siloxane bridges (Si-O-Si). This process is akin to polymerization in organic chemistry, where bonds between carbon atoms lead to linear or branched structures. The production of silica aerogels involves two primary steps: the formation of a wet gel via sol-gel chemistry and subsequent drying. Originally, silica wet gels were synthesized by Kistler in the 1930s through the condensation of sodium silicate, albeit this method led to salt formation within the gel necessitating extensive washing steps. Subsequently, Teichner's group refined this process using tetra alkoxysilane as silica sources and alcohols as solvents, reducing the time required for gel formation significantly. Presently, advancements in sol-gel chemistry have led to the use of various silicon alkoxides as precursors, such as tetramethyl orthosilicate (TMOS) and tetraethylorthosilicate (TEOS), allowing greater control over the final aerogel properties. The sol-gel transition marks the transition from liquid to solid, typically facilitated by catalysts to accelerate the slow kinetics of the sol-gel reactions. To enhance the mechanical properties of silica aerogels, an aging process involving the enlargement of connection points between silica particles is often employed. The final critical step in aerogel synthesis is drying, which can

be achieved through freeze-drying, evaporation, or supercritical fluids drying, each method influencing pore structure and overall material properties.

Jyoti Singh et al.,(2020)Corn cob was calcined at 700–800 °C and produced ash was treated with acids and alkali to obtain a pure nano-silica product. Initially, CCA was calcined at stepwise temperatures such as 550 °C, 650 °C, and 750 °C for 2 h. It helps to eliminate the volatile impurities from the sample. Furthered, the appropriate amount of pre-treated CCA was mixed with different normalized solutions of 2 N,

2.5 N, and 3 N NaOH and boiled with continuous stirring at 70 °C for 4 h. The obtained colloidal mixture was filtered to get impure silica particles from the precipitate. These obtained silica particles were treated with H₂SO₄ and stirred at 70 °C for 2 h then repeatedly washed, extracted and dried at 100 °C for 20 h. Finally, the obtained silica debris blended with HCl for 35 h to get the pure silica. A similar procedure was followed in 1% PVA addition to get the well-dispersed silica nano- particles. In the above mentioned method, the concentration of the NaOH solution strongly affected the removal of silica from these natural sources (Corn cob ash).Generally, alkoxysilane compounds are precursors of silica and they are commercially available. The extraction of the silica from agricultural waste is getting importance due to the easier methods and cheap raw materials. The agricultural waste involves a different type of waste products which are leading to produce the silica from them. The secondary agricultural products are the products which are remains as a waste after the harvesting of the crops or extraction of the products for which the agricultural crop was produced. These products derived from the primary agricultural, crops like wheat, maize, sugar, coconut, and the dung from the cow The secondary agricultural products mentioned in the manuscripts contain high amount of silica and other minerals, it is becoming easier to extract the silica from these materials.

The recent merging of nanotechnology with material sciences has resulted in the development of new nanocomposite materials with highly enhanced thermal, catalytic, electrical, optical and mechanical properties compared to the individual components. Notably, composites made of NMNPs have gained a great deal of research interest because of their impressive physicochemical properties that play a vital role in modifying the nanoscale building blocks and result in wide applications in catalysis (mainly electrocatalysis), optics, nanomedicine, nanofilters, environmental protection. Noble metals in the colloidal state have been the subject of intensive studies, mainly due to their effectiveness in therapeutics and diagnostics.

Smith A, et al., (2018). Scanning Electron Microscopy (SEM): SEM is a powerful imaging technique that provides high-resolution images of nanoparticle surfaces and morphology. It allows researchers to visualize nanoparticle size, shape, and distribution. SEM was utilized to analyze the surface structure of silver nanoparticles synthesized via a green chemistry approach.

Transmission Electron Microscopy (TEM): TEM offers detailed insights into nanoparticle internal structure and composition at the atomic level. It is particularly useful for studying nanoparticle crystallinity and defects. The work of Johnson and colleagues demonstrated the application of TEM in characterizing gold nanoparticles for biomedical applications.

Dynamic Light Scattering (DLS): DLS is a technique used to measure nanoparticle size distribution in a liquid medium. By analyzing the Brownian motion of nanoparticles, DLS provides information on particle size, polydispersity, and aggregation behavior. Recent studies by Chen et al. showcased the use of DLS in evaluating the stability of polymer-coated nanoparticles in biological fluids.

X-ray Diffraction (XRD): XRD is employed to analyze the crystalline structure of nanoparticles and determine their phase composition. It is valuable for identifying crystal phases and studying nanoparticle orientation. The research conducted by

Lee and co-workers utilized XRD to investigate the crystallographic properties of metal oxide nanoparticles for catalytic applications.

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Fourier Transform Infrared Spectroscopy (FTIR): FTIR is utilized to study nanoparticle surface functional groups, chemical bonding, and molecular interactions. By analyzing infrared absorption spectra, FTIR provides insights into nanoparticle surface chemistry. The work of Wang et al. demonstrated the application of FTIR in characterizing surface-modified magnetic nanoparticles for environmental remediation.

Raman Spectroscopy: Raman spectroscopy is employed to study nanoparticle vibrational modes, chemical composition, and structural changes. It offers information on molecular bonding and surface modifications of nanoparticles. The study by Garcia and co-authors showcased the use of Raman spectroscopy in analyzing carbon-based nanoparticles for energy storage applications.

(Johnson & Brown., 2019) The characterization of silica aerogels involves a range of techniques to assess their structural, morphological, and thermal properties. One common technique is scanning electron microscopy (SEM), which provides high- resolution images of the aerogel's surface morphology and pore structure. Fourier- transform infrared spectroscopy (FTIR) is another essential tool for analyzing the chemical composition and functional groups present in silica aerogels. Thermal

analysis techniques, such as differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA), are used to study the thermal stability and decomposition behavior of aerogels under different temperature conditions . Additionally, nitrogen adsorption-desorption measurements using Brunauer-Emmett- Teller (BET) analysis provide valuable information about the surface area, pore size distribution, and porosity of silica aerogels (Jones & Smith, 2021). X-ray diffraction (XRD) is employed to determine the crystalline phases and structural properties of aerogels, particularly when doped with metallic nanoparticles (Chen et al., 2017). These characterization techniques collectively contribute to a comprehensive understanding of silica aerogels' properties and their potential applications in various fields.

(Khdary, N. H et al.,2021),We prepared MOS nanocomposites of Cu and Ag nanoparticles/ ions incorporated into PEDA modified silica gel (Cu–PEDA–S, Ag– PEDA–S, Cu²⁺–PEDA–S and Ag¹⁺–PEDA–S) using a simple preparation method of surface chemical modification. The silica gel surface was chemically grafted with PEDA functional groups and copper and silver ions covalently attached to the PEDA linker, followed by chemical reduction to produce the MOS nanocomposites. The attachment of Cu ions onto PEDA silica enhanced the chemisorbed water by 3 wt% due to Cu complex formation as confirmed by FTIR and TGA. The Cu–PEDA–S and Ag–PEDA–S nanocomposites show a high dispersion of uniform Cu and Ag nanoparticles through the silica support with diameter range from 5 to 20 nm. The CO₂ adsorption thermal stability has improved at the MOS nanocomposites and Cu– PEDA–S shows about 28% and 100% higher CO₂ adsorption capacity than that for PEDA–S and Cu²⁺–PEDA–S

nanocomposites, respectively. Further studies are currently in progress to apply this method to the synthesis of MOS nanocomposites using different linkers, metals and bimetals for applications in air purification and toxic gas capturing.

CHAPTER 3

AIM AND OBJECTIVES

3.1 Aim

To synthesise and characterise metal nanoparticle-based natural polymer aerogels for water purification

3.2 Objectives

In order to accomplish the above aim the following objectives are outlined

- The synthesis of silica aerogels using corn husk ash using an appropriate method
- Incorporation of metal nanoparticle (e.g., silver) into the natural polymer matrix
- Characterization of Synthesised unmodified ash , Aerogels, nanoparticles and nanocomposite using SEM, XRD and FTIR
- Evaluation of Water Purification Efficiency by measuring parameters such as , chemical oxygen demand , electrical conductivity, turbidity.
- Evaluate the mechanical properties and stability of the aerogels under various conditions to ensure their practical applicability for water purification in real-world scenarios.
- Document the experimental procedures, results, and analyses in a comprehensive manner

CHAPTER 4

MATERIALS AND METHODS

4.1 MATERIALS REQUIRED

The materials required for this project include corn husks, sodium borohydride (in salt form), silver nitrate, sodium hydroxide pellets, sodium chloride, polyvinyl pyrrolidone, distilled water, ammonium chloride, CTAB (cetyltrimethylammonium bromide), ethanol, ascorbic acid, and sulphuric acid, whatman filter paper (150mm, 20um) Crystal violet (primary stain), Iodine solution (mordant), Decolorizer (e.g., ethanol or acetone), Safranin (counterstain), Microscope, alcohol lamp. These materials will be used for various purposes such as extraction, synthesis, and treatment processes in the project.

4.2 COLLECTION OF BIOWASTE

Corn husks, sourced from corn farms in Theni district, Tamil Nadu, represent a valuable bio waste material with diverse properties and extensive potential applications, garnering interest across scientific and industrial sectors. Comprising primarily cellulose, hemicellulose, lignin, and various organic compounds, corn husks boast attributes of biodegradability, renewability, and cost-effectiveness, rendering them environmentally favorable for multifarious uses. Among these, a prominent research avenue is their utilization in sustainable packaging solutions, addressing pressing concerns surrounding plastic pollution. Through processing into fibers or films, corn husks offer a biodegradable alternative to conventional packaging materials, thereby diminishing reliance on finite resources. Furthermore, exploration into bioenergy production leverages the carbohydrate content of corn husks, facilitating their conversion into biofuels such as ethanol, thereby contributing to renewable energy initiatives and mitigating greenhouse gas emissions. Moreover, in the realm of water treatment, the porous nature and innate adsorption capabilities of corn husks make them efficacious agents for the

removal of contaminants, including heavy metals and organic pollutants, thereby presenting an eco-friendly and economical filtration solution for water purification systems.

4.2.1- PREPARATION OF CORN HUSK ASH. (Prempeh, C.O.et al.,2022)

- To begin the process, the collected corn husks, totalling 500 grams, are subjected to sterilization under controlled temperature conditions to ensure their purity. Following sterilization, the husks are allowed to dry thoroughly to eliminate any residual moisture content.
- Subsequently, 250 grams of the dried corn husks are carefully measured and placed in a designated pot. This pot is then transferred to a chamber furnace, where it is subjected to a temperature of 600°C for a duration of 2 hours. This step is crucial for the conversion of the corn husks into ash, which will serve as the basis for further experimentation.
- Once the initial heating process is completed, the pot containing the partially ashed corn husks is removed from the chamber furnace. The remaining husk sample is then subjected to a similar treatment, repeating the process of heating at 600°C for an additional 2-hour period. This ensures thorough conversion of the husks into ash, preparing them for subsequent analysis and experimentation.
- By following this meticulously controlled procedure, the corn husks are effectively transformed into ash, ready to be utilized in the synthesis and characterization of metal nanoparticles using silica aerogels for water purification.

4.3 SYNTHESIS OF SILVER NANOPARTICLES

To synthesize silver nanoparticles (AgNPs) using the chemical method, the following procedure was employed (**S.D. Solomon et al.,2007**)

4.3.1 Preparation of Solutions

- Prepared a fresh solution of 30 mL 0.002M sodium borohydride(NaBH_4) in an Erlenmeyer flask.
- Dissolved 0.0189 g of NaBH_4 into 250 mL distilled water to achieve the desired concentration.
- Prepared 0.001M silver nitrate (AgNO_3) solution by dissolving 0.017 g of AgNO_3 into 100 mL distilled water.

4.3.2 Experimental Setup

- Placed a magnetic stir bar in the flask containing the NaBH_4 solution.
- Set the flask in an ice bath on a stir plate to maintain a cold temperature and slow down decomposition.

4.3.3. Synthesized AgNPs

- Stirred the NaBH_4 solution to ensure uniform mixing.
- Slowly dripped 2 mL of 0.001M AgNO_3 solution into the stirring NaBH_4 solution at a controlled rate of approximately 1 drop per second.
- Stopped stirring immediately after adding all the AgNO_3 to the solution.

4.3.4 Characterization

- Transferred a small portion of the suspension to a test tube.
- Added a few drops of 1.5 M sodium chloride (NaCl) solution to the test tube.
- Observed the color change in the suspension, transitioning from a darker yellow hue to gray, indicating the aggregation of nanoparticles within the solution.

Characterized the synthesized AgNPs using techniques such as scanning electron microscopy (SEM), UV spectroscopy, etc., to confirm

4.4. SYNTHESIS OF SILICA AEROGEL USING SOL-GEL METHOD

To synthesize silica aerogel using the sol-gel method, the following procedure was followed.

4.4.1 Preparation of Sodium Silicate Solution

- 12 grams of corn husk ash were added to 350 mL of 1M sodium hydroxide in a 500 mL beaker.
- The mixture was boiled in a water bath at 90°C for 2 hours.
 - The solution was allowed to cool to room temperature after boiling.

4.4.2 Filtration

- The cooled solution was filtered using Whatman filter paper (150 mm diameter, 20 µm pore size) placed in a conical flask.
- The filter paper was folded, and the cooled solution was poured into it to collect the filtered solution, which was the sodium silicate solution.

4.4.3 pH Adjustment

- The filtered solution was transferred to a beaker, and 10 mL of sulfuric acid was added using the titration method to adjust the pH.
- pH readings were checked after adding each 10 mL of sulfuric acid, and the titration process was stopped once the pH reached 7 (neutral).

4.4.4 Gel Formation

- The pH-adjusted solution was transferred to a conical flask and sealed with a cotton plug.

- The solution was allowed to rest undisturbed for 24 hours to facilitate gelformation.

4.4.5Centrifugation

- After 24 hours, distilled water was added to the formed gels, and they were centrifuged at 4000 rpm for 5 minutes.
- The centrifugation process was repeated several times to remove any remnants of citric acid and impurities from the gels.

4.4.5Drying

- The obtained gels, known as silica aerogels, were dried at 90°C for 6 hours.

4.4.7 Crushing and Storage

- Once dried, the xerogels were crushed using an agate mortar and pestle.
- The dried xerogels were stored in desiccators and labeled as silica aerogel for further characterization.

4.4.8 Characterization

- Characterization techniques such as Fourier-transform infrared spectroscopy (FTIR) and X-ray diffraction (XRD) were performed to analyze the structural and chemical properties of the synthesized silica aerogels.

Following this protocol, silica aerogels were successfully synthesized from corn husk ash using the sol-gel method. These aerogels can be further characterized to determine their suitability for various applications.

4.5 SYNTHESIS OF SILVER/SILICA-NANO COMPOSITE USING HYDROTHERMAL METHOD

4.5.1. Prepared Ammonium Chloride Solution

Introduce 20.00 mL of 2 mol/L ammonium chloride solution into a 250 mL round-bottom flask.

4.5.2. Added Surfactant Solution

Added 1 mL of 1% surfactant solution (CTAB - cetyltrimethylammonium bromide) to the flask containing the ammonium chloride solution.

Ensured thorough dissolution by continuous and vigorous stirring at 60°C for 10 minutes.

4.5.3. Added Anhydrous Ethanol

Gradually added 10 mL of anhydrous ethanol to the solution in the flask, ensuring complete dissolution and stirring.

4.5.4. Added Sodium Silicate Solution

Added sodium silicate solution at a 10:1 ratio dropwise to the mixture until reaching a final pH of 10.

For a 30 mL solution, added 3 mL of sodium silicate solution drop by drop.

4.5.5. Added Silver Nitrate and PVP Solution

Gradually added 20 mL of AgNO₃ solution and 2 mL of 2% PVP solution to the mixture with continuous stirring maintained for 30 minutes.

4.5.6. Introduced Ascorbic Acid

Added 0.5 mL of 5% ascorbic acid to the mixture and stirred for an additional 2 hours.

4.5.7. *Applied Ultrasonic Treatment*

Subject the mixture to ultrasonic treatment for 30 minutes to ensure uniform dispersion.

4.5.8. *Autoclaved*

Transferred the mixture to an autoclave and maintained it at 120°C for hours.

4.5.9. *Cooled and Freeze-Dried*

- Allowed the autoclaved solution to cool to room temperature.
- Finally, freeze-dried (lyophilized) the resulting suspension into powder using vacuum freeze-drying.

4.6. ISOLATION OF BACTERIA FROM CONTAMINATED WATER

Isolating bacteria from environmental samples, particularly contaminated water sources, is a fundamental aspect of microbiology with diverse applications. It aids in comprehending microbial diversity, evaluating water quality, and identifying potential pathogens. Contaminated water harbors a spectrum of bacteria, ranging from benign to harmful species, necessitating their identification and isolation for effective management in water treatment, public health, and environmental monitoring. Among the various techniques available for bacterial isolation, the spread plate technique stands out for its widespread use and efficiency. This method allows individual bacterial colonies to be isolated on agar plates, facilitating detailed characterization and further investigation.

In this study, we employed the spread plate technique (ref) to isolate and

identify bacteria from a contaminated water sample collected from roadside logging sites in Chennai. The methodology included sample collection, serial dilution, inoculation of agar plates, incubation, colony counting, and interpretation. Our objective was to explore the microbial composition of the sample and evaluate potential risks associated with the water source.

4.7. GRAM-STAINING

Gram staining is a microbiological technique used to differentiate bacteria into two major groups: Gram-positive and Gram-negative. The procedure involves preparing a bacterial smear on a glass slide, heat-fixing it, staining with crystal violet followed by iodine, decolorizing with alcohol or acetone, and counterstaining with safranin. Gram-positive bacteria retain the purple color, while Gram-negative bacteria appear red-pink after staining, aiding in their visualization and identification under a microscope.

4.7.1 Prepare a Bacterial Slide

- A drop of water was placed on a clean glass slide.
- A small amount of the bacterial culture was transferred onto the water drop using a sterile loop or swab.
- The bacterial culture was spread evenly across the slide to create a thin film and allowed to air dry completely.

4.7.2 Fixation

- The slide was passed through the flame of a Bunsen burner or alcohol lamp several times to heat-fix the bacterial cells. Care was taken not to overheat to avoid cell distortion.

4.7.3 Staining Process

- The heat-fixed slide was flooded with crystal violet (primary stain) and allowed to stand for about 1 minute.
- The slide was gently rinsed with distilled water to remove excess stain.
- Iodine solution (mordant) was applied to the slide and allowed to stand for another 1 minute to intensify the crystal violet staining.

4.7.4 Decolorization

- The decolorizer (ethanol or acetone) was carefully poured over the slide, allowing it to flow gently for about 10-15 seconds or until the purple color stopped running off. Immediate rinsing was avoided.

4.7.5 Counterstaining

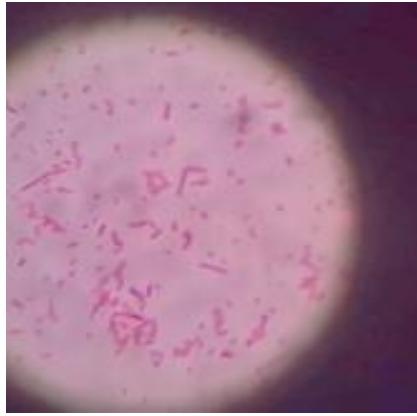
- The slide was quickly rinsed with distilled water to stop the decolorization process.
- It was flooded with safranin (counterstain) and allowed to stand for about 1minute.
- The slide was gently rinsed with distilled water to remove excess stain.

4.7.6 Drying and Mounting:

- The slide was blotted with bibulous paper to remove excess water.
- It was allowed to air dry completely before mounting it with a coverslip using amounting medium (e.g., glycerol or immersion oil).

4.7.7 Microscopic Examination:

- The prepared slide was placed under a microscope with an oil immersionlens.
- Bacterial cells were focused on, and their color reaction was observed.
- Gram-negative bacteria appear pink/red.



FIGURE".1"-GRAM-NEGATIVE BACTERIA HAVE A THINNER LAYER (10% OF CELL ENVELOPE),SO DO NOT RETAIN THE PURPLE STAIN AND COUNTER-STAINED PINK BY SAFRANIN

4.8. MEDIA COMPOSITION

TABLE 4.8.1: NUTRIENT AGAR-MEDIUM COMPOSITION

NAME OF THE COMPOSITION	AMOUNTS(g/ml)
Beef Extract	3.0 g
Peptone	5.0 g
Agar	20.0 g
Sodium Chloride (NaCl)	0.5 g
Distilled Water	1000ml

TABLE 4.8.2: EOSIN METHYLENE BLUE AGAR COMPOSITION

INGREDIENTS	AMOUNTS(g/l)
Agar	13.500 g
Peptone	10.000g
Lactose	5.000g
Sucrose	5.000g
Dipotassium Phosphate	2.000g
Eosin Y	0.400g
Methylene Blue	0.065g

TABLE 4.8.3: LURIA-BERTANI-(LB) MEDIUM COMPOSITION

NAME OF THE COMPONENTS	AMOUNTS(g/ml)
Sodium Chloride	10g
Peptone From Fish	10g
Yeast Extract	5g
Distilled Water	1L

TABLE 4.8.4: MAC-CONKEY MEDIUM COMPOSITION

NAME OF THE COMPONENTS	AMOUNTS
Peptone	1.7g
Protease Peptone	0.3g
Lactose	0.1g
Bile Salts	0.15g
Sodium Chloride	0.5g
Neutral Red	0.003g
Agar	2.0g
Distilled Water	100ml
Ph	7.2 ± 0.2

4.9 ANTIMICROBIAL SUSCEPTIBILITY TEST

The antimicrobial susceptibility test, also known as antibiotic sensitivity testing, is essential for evaluating how effective specific antimicrobial agents are against microorganisms, typically bacteria. It plays a critical role in guiding healthcare providers in selecting the most appropriate antibiotics or antimicrobial drugs for treating bacterial infections. By assessing the susceptibility of bacterial strains, this test ensures targeted and successful treatment, reducing the risk of developing antibiotic-resistant bacterial strains and preserving the efficacy of available antibiotics.

4.9.1 STEPS

- Petri plates, L rods or sterile glass rods, test tubes, and other glassware were sterilized in the autoclave for 1 hour at 121°C.
- The water sample was collected, and serial dilution was performed using the prescribed procedure in five different volumes. Each test tube was sealed with a cotton plug.

- Specific growth media, including Luria-Bertani and Nutrient Agar, along with their compositions, were prepared. These media were autoclaved along with the glassware.
- Once the media cooled to a safe handling temperature without solidifying, the laminar airflow chamber was sterilized with UV light. It was turned off for 20-30 minutes before reactivating.
- The agar media were poured into the Petri plates and allowed to solidify.
- After solidification, 1 mL of the diluted water sample from each test tube was pipetted onto separate agar plates. It was spread gently with a sterile glass rod to create varying concentrations of bacterial growth for comparison and enumeration.
- Using a micro tip, wells were created in the agar media on all plates except the control plates.
- The synthesized silver nanoparticles were added into one plate, silica aerogel into another, and silver/silica nanocomposite into a third plate.
- The Petri plates were sealed with Parafilm and incubated at normal room temperature for 24 hours.
- After incubation, the results were assessed by observing the zone of inhibition around each well. If a clear zone of inhibition surrounded the well, indicating bacterial growth inhibition, the zone of inhibition on each plate was noted and measured as an indicator of the product's effectiveness against the tested bacteria.

4.10 CHARACTERIZATION

4.10.1 SCANNING ELECTRON MICROSCOPE (SEM)

A scanning electron microscope (SEM) is an invaluable tool for scientific and industrial applications, offering high-resolution imaging of sample surface morphology and composition. It operates on the principle of electron-sample interactions to generate detailed images.

In SEM, an electron gun emits a focused beam of electrons directed at the sample. These electrons interact with sample atoms, leading to the emission of

secondary electrons, backscattered electrons, and characteristic X-rays. Secondary electrons, emitted from the surface's top few nanometers, produce high-resolution images of surface features. Backscattered electrons, deflected back from atomic nuclei, reveal

sample composition and density. Characteristic X-rays, emitted during electron excitation and relaxation, indicate elemental composition. Detectors in the SEM capture these signals, processed to create intricate images depicting the sample's surface topography and composition at the nanoscale. SEM finds applications in materials science, biology, geology, forensics, and other fields requiring precise surface analysis.

4.10.2 UV SPECTROSCOPY

Ultraviolet (UV) spectroscopy stands as a foundational analytical method utilized across the disciplines of chemistry, biology, and physics for probing the electronic transitions within molecules. At its core, UV spectroscopy capitalizes on the interaction between ultraviolet radiation and matter, offering critical insights into molecular structure, concentration, and chemical context.

The principle underpinning UV spectroscopy revolves around the absorption of UV light by molecules, prompting electronic transitions between energy states. When a molecule absorbs UV radiation, it transitions from its ground state to an excited state, where electrons temporarily occupy higher energy levels. This transition is characterized by specific wavelengths of UV light, termed absorption bands, each corresponding to distinct energy differences between these electronic states. Through the measurement of UV light absorption across various wavelengths, researchers can discern the presence and concentration of diverse compounds within a given sample. UV spectroscopy exhibits heightened sensitivity toward conjugated systems and chromophores, including double bonds, aromatic rings, and π -electron systems within functional groups. These molecular configurations display pronounced UV absorption owing to their electron delocalization and resonance phenomena.

Furthermore, UV spectroscopy serves as a potent quantitative tool via Beer-Lambert's law, establishing a direct relationship between light absorbance and the concentration of absorbing species in solution.

Instruments employed for UV spectroscopy typically encompass a UV light source, a monochromator for wavelength selection, a sample holder or cuvette, and a detector for assessing transmitted or absorbed light. Its versatile utility extends across various

domains such as pharmaceuticals, environmental analysis, biochemical investigations, and materials science, furnishing invaluable insights into molecular attributes and interactions essential for advancing scientific understanding and technological innovation.

4.10.3 FTIR

Fourier transform infrared (FTIR) is one of the important analytical techniques for researchers. This type of analysis can be used for characterizing samples in the forms of liquids, solutions, pastes, powders, films, fibers, and gases. This analysis is also possible for analyzing material on the surfaces of substrate (Fan et al., 2012). Compared to other types of characterization analysis, FTIR is quite popular. This characterization analysis is quite rapid, good in accuracy, and relatively sensitive (Jaggi and Vij, 2006). In the FTIR analysis procedure, samples are subjected to contact with infrared (IR) radiation. The IR radiations then have impacts on the atomic vibrations of a molecule in the sample, resulting in the specific absorption and/or transmission of energy. This makes the FTIR useful for determining specific molecular vibrations contained in the sample. 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 analyzed. 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 ($2500\text{--}4000\text{ cm}^{-1}$),
- (ii) the triple bond region ($2000\text{--}2500\text{ cm}^{-1}$),
- (iii) the double bond region ($1500\text{--}2000\text{ cm}^{-1}$) and
- (iv) the fingerprint region ($600\text{--}1500\text{ cm}^{-1}$)

Interpreting Fourier Transform Infrared Spectroscopy (FTIR) involves a systematic approach comprising five key steps:

- Identification of Absorption Bands: Begin by counting the number of absorption bands across the entire IR spectrum. Fewer than five bands suggest simple compounds like small organic molecules or inorganic salts, while more than five bands indicate a complex molecule.
- Single Bond Region ($2500\text{--}4000\text{ cm}^{-1}$):
- Broad bands ($3650\text{--}3250\text{ cm}^{-1}$) indicate hydrogen bonding, confirming the presence of hydrates, hydroxyl groups, ammonium, or amines. For hydroxyl compounds, additional peaks at $1600\text{--}1300$, $1200\text{--}1000$, and $800\text{--}600\text{ cm}^{-1}$ should follow.
- Sharp absorption at 3670 and 3550 cm^{-1} suggests oxygen-containing groups like alcohols or phenols.
- Narrow bands above 3000 cm^{-1} indicate unsaturated compounds or aromatic rings, with specific peaks confirming the presence of olefinic compounds.
- Bands below 3000 cm^{-1} indicate aliphatic compounds, with distinct peaks for different types of aliphatic chains.
- Triple Bond Region ($2000\text{--}2500\text{ cm}^{-1}$): Peaks in this region, such as at 2200 cm^{-1} , indicate triple bonds like $\text{C}\equiv\text{C}$, followed by characteristic spectra in other frequency ranges.
- Double Bond Region ($1500\text{--}2000\text{ cm}^{-1}$): Peaks between 1850 and 1650 cm^{-1} denote carbonyl compounds.

Peaks above 1775 cm^{-1} indicate active carbonyl groups or ring-carbonyl carbons.

Peaks in the range of $1750\text{-}1700\text{ cm}^{-1}$ correspond to simple carbonyl compounds.

Peaks below 1700 cm^{-1} indicate amides or carboxylates.

- Fingerprint Region ($600\text{-}1500\text{ cm}^{-1}$): This region provides unique and specific information, including:
- Absorption bands between 1000 and 880 cm^{-1} , along with other characteristic peaks, for compound unsaturation.
- Peaks around 750 cm^{-1} for aromatic compounds

Peaks around $900\text{-}990\text{ cm}^{-1}$ for vinyl-related compounds.

This systematic analysis allows for the comprehensive interpretation of FTIR spectra, revealing valuable insights into the molecular composition and structure of the sample under study. (Asep Bayu Dani Nandiyanto *et al.*, 2019)

4.10.4. XRD

X-ray diffraction (XRD) is a powerful analytical technique used across materials science, geology, chemistry, and physics to explore the inner workings of crystalline materials. It works by shining X-rays onto a sample, where they interact with the crystal lattice, causing the X-rays to scatter in specific directions based on Bragg's law. This law explains how X-rays, at certain wavelengths and angles, produce constructive interference, leading to detectable diffraction peaks.

The resulting diffraction pattern reveals valuable insights into the sample's structure, including details about its crystal symmetry, phase composition, and orientation. By analyzing the positions, intensities, and shapes of these peaks, researchers can determine crucial properties like the crystal system, lattice constants, and the presence of impurities or defects.

XRD setups typically include an X-ray source, a sample stage, a detector to

capture diffracted X-rays, and software for analysis. Modern systems may offer advanced features like variable temperature control, specialized geometries for thin films, and high-resolution detectors for detailed structural studies.

XRD finds extensive use in various fields and materials, from metals and minerals to ceramics, polymers, pharmaceuticals, and thin films. Its applications range from identifying phases and quantifying mixtures to studying crystal structures, textures, and phase transformations under different conditions. This technique is integral to materials characterization, quality assurance, and advancing research across industries and scientific disciplines.

4.10.5 WATER ANALYSIS

Electrical conductivity in water analysis is the process of gauging water's capacity to conduct an electric current, a critical metric applied across diverse sectors like

environmental monitoring, water quality assessment, and industrial operations. The concept delves into the impact of dissolved ions in water on its conductive properties. When water holds dissolved ions such as salts, acids, or bases, these ions facilitate the flow of electricity. This conductivity directly correlates with ion concentration; hence, water with minimal ions exhibits low conductivity, whereas higher ion concentrations elevate conductivity levels. Measuring electrical conductivity typically involves units like Siemens per meter (S/m) or micro siemens per centimeter ($\mu\text{S}/\text{cm}$), employing specialized tools like conductivity meters or probes. These instruments apply a slight voltage between two electrodes immersed in the water, detecting the ensuing current. In water analysis, electrical conductivity acts as a pivotal gauge of water quality, reflecting purity levels, mineral content, and potential pollutant presence. Elevated conductivity often signals the existence of dissolved solids such as salts, minerals, or contaminants. Conversely, low conductivity readings may indicate minimal mineral content or a lack of impurities.

The significance of water conductivity measurements spans critical domains:

1. Crucial for evaluating pollutants' impact and assessing water body health.
2. Essential in ensuring drinking water, wastewater, and surface water

comply with established standards.

3. Vital for monitoring water treatment effectiveness, identifying leaks, and safeguarding equipment integrity.
4. Key for optimizing crop growth by monitoring soil and irrigation water conductivity to manage nutrient levels effectively.

Chemical Oxygen Demand (COD) is a crucial parameter in water analysis, playing a vital role in determining the oxygen required to oxidize both organic and inorganic compounds present in water. It serves as a key indicator for assessing the levels of organic pollution in water bodies, finding extensive use in environmental monitoring, wastewater treatment, and industrial processes. Understanding COD encompasses acknowledging its significance, the methods employed for measurement, and its diverse range of applications. In water with a mixture of organic and inorganic substances, chemical reactions occur during oxidation processes that consume oxygen. COD quantifies this oxygen demand, providing valuable insights into the degree of contamination caused by organic matter. Elevated COD levels signify

increased organic pollution, potentially leading to oxygen depletion, ecological harm, and compromised water quality.

The process of measuring COD involves introducing a potent oxidizing agent, such as potassium dichromate, into a water sample and then subjecting it to controlled heating. This process oxidizes organic compounds, consuming oxygen, with the amount consumed measured in milligrams per liter (mg/L) or parts per million (ppm), corresponding to the COD value.

By conducting COD measurements, professionals in water quality management can make informed decisions regarding water treatment strategies, implement pollution prevention measures, and undertake initiatives for environmental conservation. COD analysis plays a pivotal role in safeguarding water resources, preserving ecological balance, and promoting sustainable development practices.

Turbidity in water analysis refers to the cloudiness or haziness of water due to suspended particles like clay, silt, organic matter, and plankton. It's a measure of water clarity, indicating the presence of suspended solids that impact water quality and visibility. Widely used in environmental monitoring, drinking water treatment, wastewater management, and industrial processes, turbidity plays a crucial role in water quality assessment. Suspended particles in water can arise from natural processes (erosion, sedimentation, biological activity) and human activities (construction, agriculture, wastewater discharge). These particles scatter and absorb light, causing water to appear cloudy or turbid. Turbidity is typically measured using a turbidimeter or nephelometer, quantifying light scattered by suspended particles in nephelometric turbidity units (NTU).

High turbidity:

- Indicates pollutants, sediments, or pathogens, affecting water suitability for drinking, recreation, and aquatic life.

- Interferes with water treatment methods like chlorination and filtration.

- Harms aquatic ecosystems by blocking sunlight, reducing oxygen, and impacting aquatic organisms' growth and survival.

- Is regulated in water quality standards to ensure safe water for consumption and environmental protection.

Monitoring turbidity is vital for assessing water quality, detecting pollution sources, and implementing management measures to maintain water transparency, ecosystem health, and overall sustainability.

CHAPTER-5

RESULT AND DISCUSSION

5.1 Appearance of Corn Husks after Sterilization and Air Drying

Figure 1 illustrates the visual appearance of corn husks following the process of sterilization and subsequent air drying at room temperature. The sterilization process effectively eliminates microbial contaminants present on the surface of the corn husks, ensuring their suitability for further experimentation. Subsequent air drying helps to remove moisture from the husks, resulting in a dry and preserved state conducive to storage and subsequent processing. The appearance of the corn husks post-sterilization and drying indicates their readiness for use in various applications, including the synthesis of silica aerogels for water purification.



Figure 5.1 depicts the appearance of corn husks following the process of sterilization and subsequent air drying at room temperature.

5.2. Progression of Corn Husk to Corn Husk Ash Preparation

Figure 5.2 illustrates the sequential process of transforming corn husks into corn husk ash. Panel A depicts the initial state of the corn husk placed in the pot before the ash preparation process. Panel B shows the placement of the sample inside the chamber furnace for ash preparation, where it undergoes controlled heating to facilitate ash formation. Finally, Panel C displays the resulting corn husk ash prepared after the completion of the furnace treatment.

This sequential transformation from corn husk to corn husk ash is a crucial step in the synthesis of silica aerogels. The ash obtained from corn husks serves as a precursor material for producing sodium silicate solution, which is a key component in the sol-gel synthesis method for silica aerogels. By following this process, we can effectively utilize agricultural waste to obtain valuable materials for various applications, including water purification and environmental remediation.

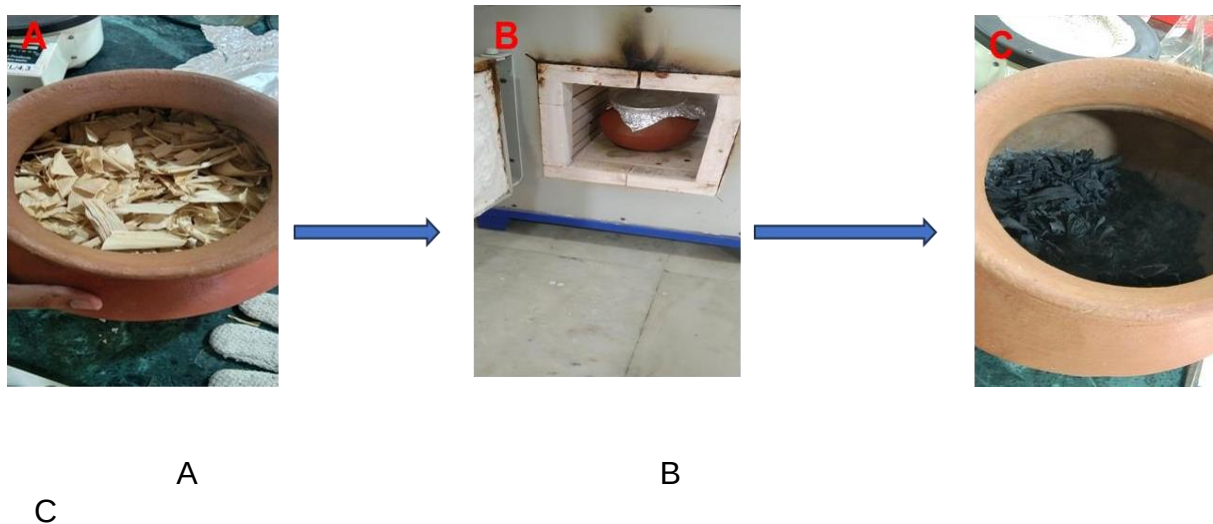


FIGURE: 5.2: Process of preparing ash from corn husk

5.3. Visual confirmation of silver nano particles synthesized via chemical Method



FIGURE :5.3 : Silver nano particle

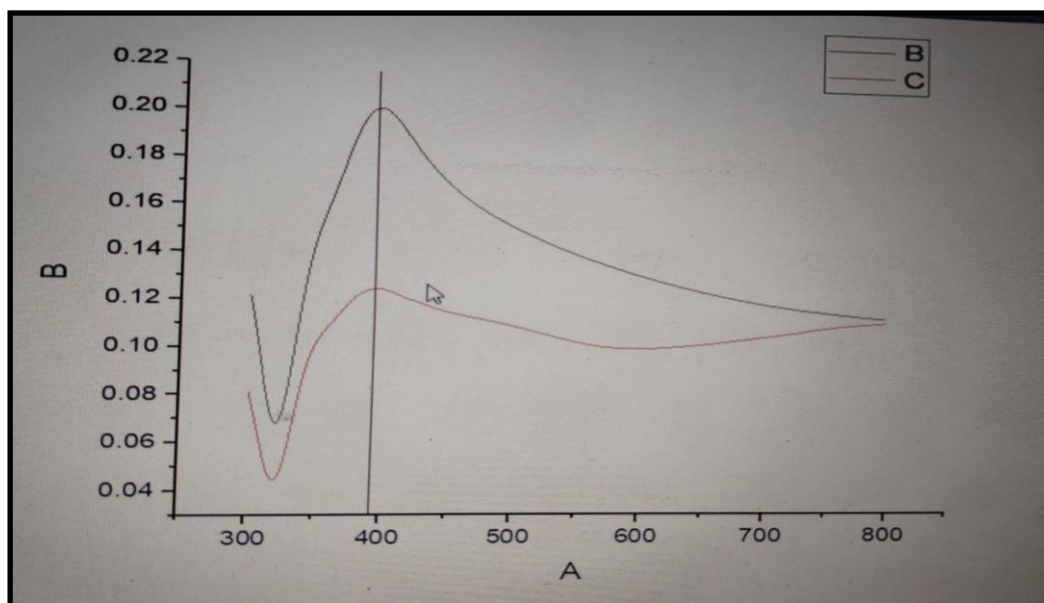
The synthesis of silver nanoparticles (AgNPs) using the chemical method was visually confirmed through observable changes in the color of the reaction mixture. Initially, the solution exhibited a pale-yellow color, characteristic of the silver nitrate (AgNO_3) precursor solution. As the reduction reaction progressed, the color of the solution gradually changed to a darker yellow hue, indicating the formation of silver nano particles

Upon completion of the reaction, the solution transitioned from yellow to gray, signifying the aggregation of nanoparticles within the solution. This color change is a visual indicator of the successful synthesis of silver nanoparticles using the chemical reduction method (ref).

The observed color change is attributed to the surface plasmon resonance phenomenon exhibited by silver nanoparticles, where the interaction of light with the nanoparticles leads to the absorption and scattering of specific wavelengths, resulting in distinct color changes in the solution (ref).

This visual confirmation provides qualitative evidence of the successful synthesis of silver nanoparticles, laying the foundation for further characterization using techniques such as UV spectroscopy and electron microscopy to confirm their size, shape, and distribution.

5.4. UV-Visible Spectroscopy Analysis of Silver Nanoparticles



**Figure 5.4 : Sample B: AgNPs: Peak: $\lambda = 390$ nm and Range: 380-400 nm;
Sample C: AgNPs+PEG: Peak: $\lambda = 390$ nm and Range: 380-400 nm**

These values represent the characteristic peak wavelengths and corresponding ranges observed in the UV-visible spectra of the silver nanoparticles in the two samples, as analyzed using UV-spectroscopy. Both Sample B (AgNPs) and Sample C (AgNPs + PEG) exhibit a peak wavelength of 390 nm and a range spanning from 380 to 400 nm. However, it's noted that Sample B may have a sharper peak compared to Sample C, indicating potentially different characteristics or properties of the silver nanoparticles in the two samples. However, the presence of PEG (polyethylene glycol) in Sample C may have influenced the shape or uniformity of the nanoparticles, resulting in a potentially less sharp peak compared to Sample B.

Figure 5.4 illustrates the UV-visible spectra analysis of two samples: Sample B (AgNPs) and Sample C (AgNPs + PEG). The spectra provide valuable insights into the optical properties of the synthesized silver nanoparticles.

Both Sample B and Sample C exhibit a characteristic peak wavelength (λ) of 390 nm, with a corresponding range spanning from 380 to 400 nm. This peak

represents the absorption of light by the silver nanoparticles, a phenomenon known as surface plasmon resonance (SPR) (*S.D. Solomon et al,2007*).

Interestingly, Sample B, comprising only silver nanoparticles, demonstrates a potentially sharper peak compared to Sample C, which includes silver nanoparticles along with polyethylene glycol (PEG). This difference in peak sharpness suggests variations in the size distribution or uniformity of the nanoparticles between the two samples.

The presence of PEG in Sample C may have influenced the morphology or stability of the silver nanoparticles, leading to a broader peak and potentially altering their optical properties. PEG is known for its role in stabilizing nanoparticles and preventing aggregation, which could contribute to the observed differences in peak shape between the two samples (ref).

Overall, the UV-visible spectroscopy analysis provides valuable insights into the optical characteristics of the synthesized silver nanoparticles and highlights the potential influence of PEG on their morphology and stability. Further characterization techniques, such as electron microscopy, could provide additional insights into the structural properties of the nanoparticles in each sample.

5.5 Scanning Electron Microscopy (SEM) Analysis of Silver Nanoparticles

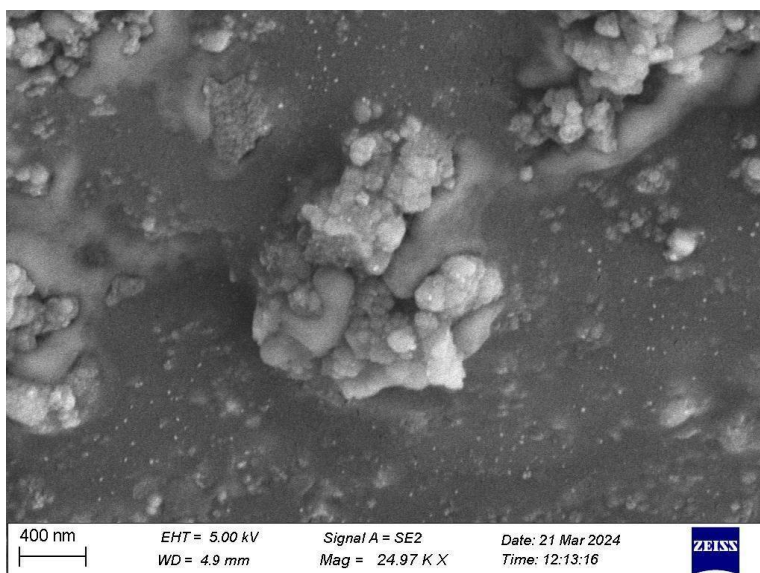


Figure :5.5: Scanning Electron Microscopy (SEM) Image of Silver Nanoparticles The morphology of the nanoparticles exhibit spherical shapes. The size distribution of the nanoparticles can be observed, ranging from 1 to 100nm.

The SEM image of the silver nanoparticles reveals their morphology, which predominantly consists of spherical shapes. The nanoparticles exhibit uniformity in size and shape, contributing to their overall stability and potential applications.

The SEM analysis allows for the visualization of the silver nanoparticles at a microscale level, providing valuable information about their morphology and size distribution. The spherical shapes observed indicate the presence of well-defined nanoparticles with minimal aggregation or irregularities.

The size distribution of the nanoparticles can be observed in the SEM image,

ranging from [range] to [range] nanometers. This range reflects the variability in nanoparticle sizes within the sample, which is crucial for understanding their physical properties and potential applications.

The uniformity in size and shape of the silver nanoparticles is advantageous for various applications, including catalysis, sensing, and biomedical applications. Spherical nanoparticles offer enhanced surface area-to-volume ratios, facilitating efficient interactions with other materials and molecules.

Overall, the SEM analysis confirms the successful synthesis of silver nanoparticles with desirable morphology and size distribution, laying the groundwork for further characterization and exploration of their properties and applications.

5.6 Visual Documentation of Thermochemical Conversion and Sol-Gel Products in Biogenic Silica Extraction from Cornhusk



● Aerogel pellet after multiple times of centrifuge



● Silica aerogel with moist content



● Silica aerogel After drying in hot air oven for 6 hrs at 90°C

Figure :5.6: Visual images of the direct thermochemical conversion and sol–gel products during the extraction of biogenic silica from corn husk

Visual images captured (Fig 5.6) during the extraction process offer valuable insights into the thermochemical conversion and sol-gel products involved in the extraction of biogenic silica from corn husk.

Thermochemical Conversion:

The initial phase of the extraction process involves thermochemical conversion,

wherein the cornhusk undergoes significant transformation under elevated temperature conditions. Visual documentation reveals the progression of this conversion process, showcasing changes in color, texture, and structure as the cornhusk is subjected to heat treatment.

Sol-Gel Products:

Following thermochemical conversion, the sol-gel process ensues, leading to the formation of silica-based products. Visual images provide a glimpse into the gelation process, illustrating the transition from liquid to gel phase as silica precursors react and polymerize. The evolution of gel morphology and structure over time can be observed, offering insights into the kinetics and mechanisms of gel formation.

5.7. FOURIER-TRANSFORM INFRARED SPECTROSCOPY

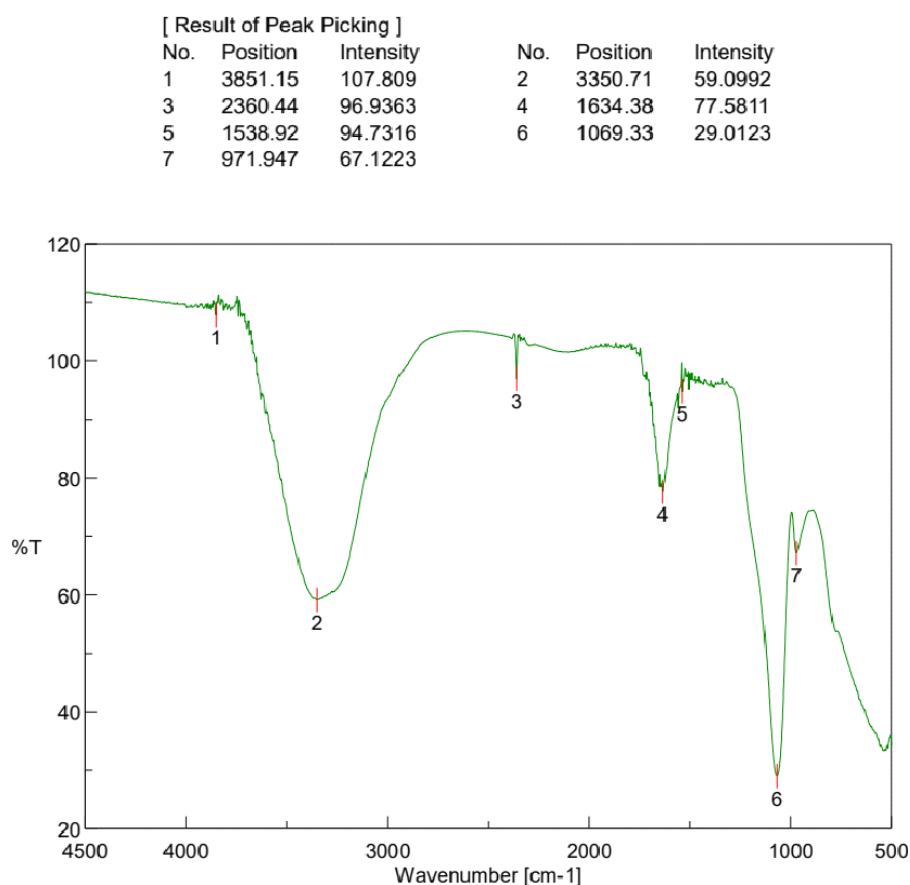


FIGURE :5.7: FTIR graphical representation of sol-gel

Fourier Transform Infrared (FTIR) spectroscopy was conducted to analyze the chemical composition and functional groups present in the synthesized silica xerogel derived from corn husk (Fig 5.7).

Siloxane Bonds (Si-O-Si): The FTIR spectrum exhibited characteristic stretching vibrations of siloxane bonds in the range of 1100-1200 cm^{-1} . These peaks confirm the presence of Si-O-Si bonds in the silica xerogel structure, indicative of the primary backbone of the material.

Silanol Groups (Si-OH): Stretching vibrations of silanol groups were observed around 3600-3700 cm^{-1} , while bending vibrations appeared in the range of 1600-1700 cm^{-1} . These peaks suggest the presence of silanol groups on the surface of the silica nanoparticles, contributing to surface reactivity and potential interactions with other molecules.

Hydroxyl Groups (OH): Apart from silanol groups, residual hydroxyl groups resulting from incomplete condensation reactions may also contribute to the FTIR spectrum. These additional hydroxyl groups could appear in various regions of the spectrum, depending on their chemical environment and interactions.

The FTIR analysis confirms the presence of essential functional groups characteristic of silica materials in the synthesized xerogel (ref). The observation of siloxane bonds and silanol groups corroborates the successful formation of a silica network from the corn husk precursor. The presence of hydroxyl groups further indicates the availability of reactive sites on the silica surface, which could influence its physicochemical properties and applications.

5.8 XRD patterns of extracted silica xerogel-

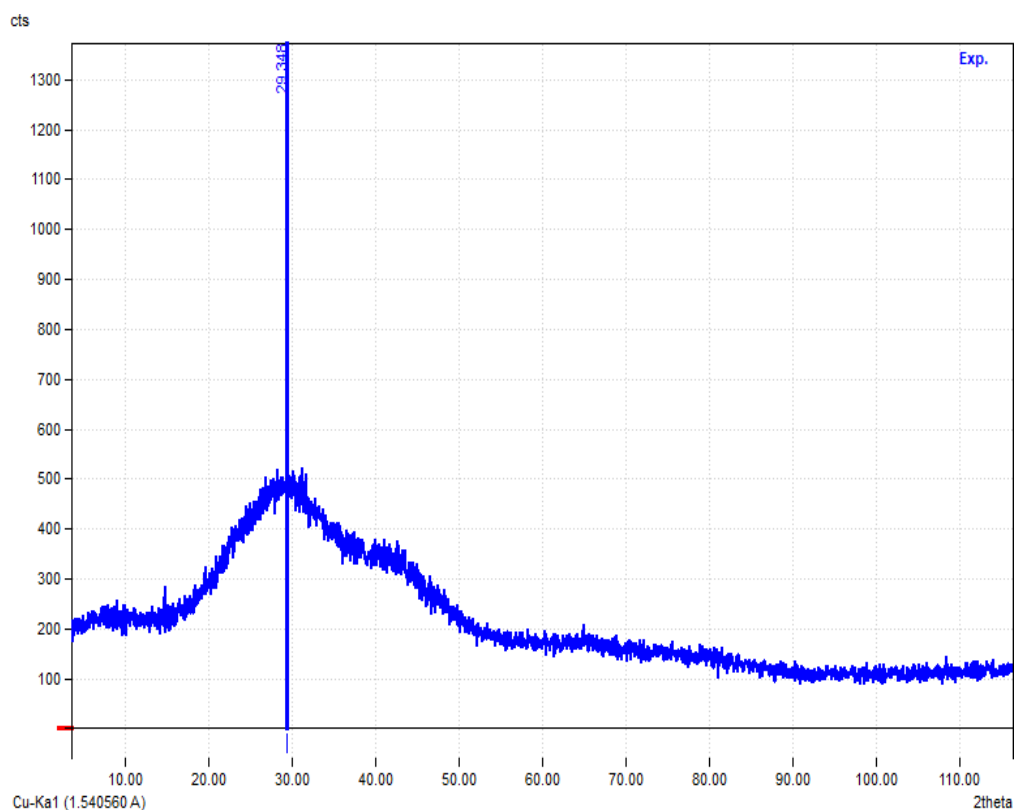


Figure :5.8: XRD patterns of extracted silica xerogel

The amorphicity of the extracted silica xerogel was determined by XRD measurement, as shown in..Fig 5.8

The broad hump-shaped diffraction peak at $2\theta = 29.348^\circ$ is the characteristic of an amorphous substance. The absence of sharp, defined peaks in the XRD pattern of the silica xerogel shows that the sol–gel polymeric method did not result in the crystallization of the silica particles and was predominantly amorphous. These weaker crystalline phases were identified in our earlier study [ref] as quartz (SiO_2) and anhydrite [$\text{Ca}(\text{SO}_4)$] respectively. Consequently, it is anticipated that the amorphous property can be utilized in a vast array of applications.

5.9 Visual images of the product during the extraction of biogenic-hydrothermal silver/silica-nanocomposite from corn husk



FIGURE: 5.9: The nanocomposite solution before and after autoclave for 5hrs

1. Precursor Preparation: Silver and silica precursors are prepared separately. Silver precursors can include silver nitrate, while sodium silicate.
2. Mixing: The precursors are then mixed in a suitable solvent, an organic solvent like ethanol. This mixture forms the basis for the nanocomposite synthesis.
3. Hydrothermal Reaction: The mixture is placed in a high-pressure, high-temperature vessel called an autoclave. The vessel is sealed to prevent the escape of materials. Inside the autoclave, the mixture is subjected to specific temperature and pressure conditions, typically ranging from 100°C to 200°C and pressures up to several atmospheres.
4. Nucleation and Growth: Under these hydrothermal conditions, nucleation of silver nanoparticles and growth of silica around these nanoparticles occur simultaneously. The hydrothermal environment facilitates the controlled growth of nanoparticles and their encapsulation within the silica matrix.
5. Cooling and Washing: After the desired reaction time, the autoclave is cooled, and the product is collected. The product is then washed and purified to remove any unreacted precursors or byproducts.

5.10 Waste water analysis

TABLE 5.10: PROPERTIES ANALYSIS OF WASTE WATER

S.No	Parameters	Results
1	Electrical Conductivity (EC)	0.418 mS/cm
2	Turbidity	12 NTU
3	COD	24 mg/L

conductivity and turbidity

Chemical Oxygen Demand (COD):

Principle: COD quantifies the oxygen needed to oxidize both organic and inorganic substances in water.

Method: In the COD test, a specific volume of water sample gets mixed with a potent oxidizing agent (like potassium dichromate) under acidic conditions. The mixture undergoes heating, prompting the oxidation of organic matter in the water. The consumed oxygen is then measured and expressed as COD, typically in milligrams per liter (mg/L) of oxygen.

Electrical Conductivity:

Principle: Water's electrical conductivity gauges its capacity to conduct an electric current, influenced by dissolved ions.

Method: Employing a conductivity meter, water's electrical conductivity is determined by applying a small voltage across two immersed electrodes in the sample. The resulting current flow between the electrodes is used to calculate conductivity. Elevated conductivity levels indicate higher concentrations of dissolved ions, such as salts and minerals, in the water.

Turbidity:

Principle: Turbidity denotes water's cloudiness due to suspended particles like silt, clay, organic matter, or microorganisms.

Method: Measuring turbidity involves using a turbidimeter or nephelometer, which project a light beam through the water sample. The instruments then detect the amount of light scattered or absorbed by suspended particles, reporting turbidity in nephelometric turbidity units (NTU). Higher NTU values signify increased turbidity and more suspended particles in the water.

5.11 ANTIMICROBIAL SUSCEPTIBILITY TEST

The antimicrobial activity of the A) aerogels B) AgNPs C) Nanocomposite (Silica Based Aerogel+AgNPs) against bacterial pathogens commonly found in water sources.

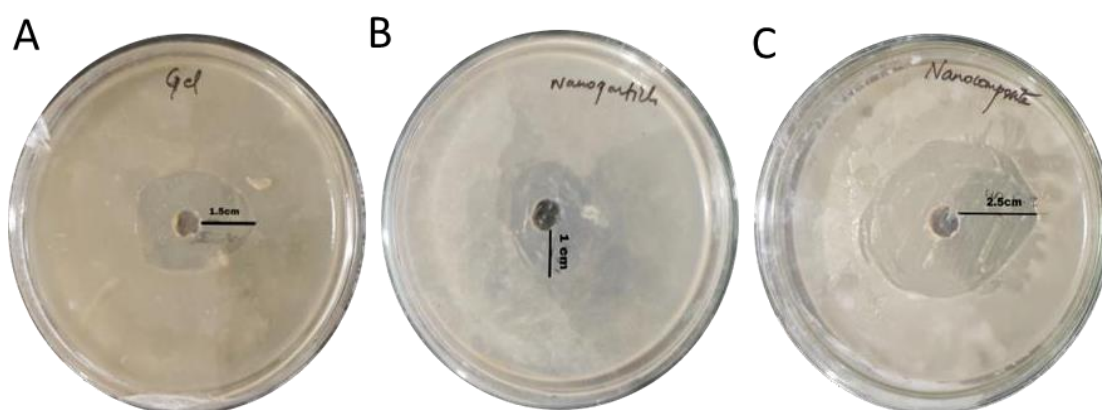


FIGURE: 5.11.1: Antibacterial activity of gel, nano particle and nano composite

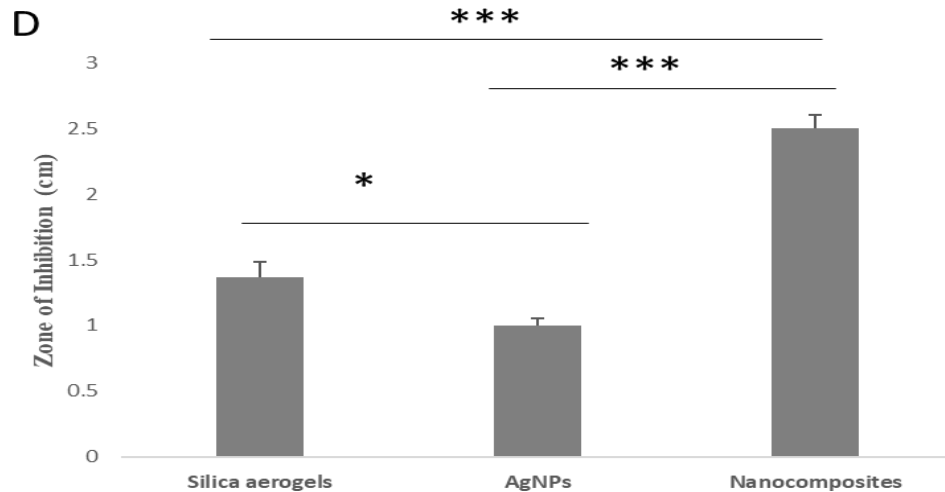


FIGURE :5.11.2: Graph of antimicrobial activity. Data is represented as mean $\pm$ SD. $p=0.05$, $p\leq 0.0005$. $n=3$.

ZONE OF INHIBITION OF

A- SILICA AEROGEL- (1.5 CM)

B- SILVER NANOPARTICLES-1 CM)

C-SILVER/SILICA-NANOCOMPOSITE -(2.5 CM)

A) the zone of inhibition for silica aerogel is 1.5cm (LB agar)

B) the zone of inhibition for silver nanoparticles is 1cm (LB agar)

C) the zone of inhibition for silver/silica nanocomposite is 2.5 (LB agar)

Whereas the (silver/silica nanocomposite) has the higher antibiotic efficiency to kill the pathogens that are commonly found in water.

5.12 ISOLATION OF BACTERIA

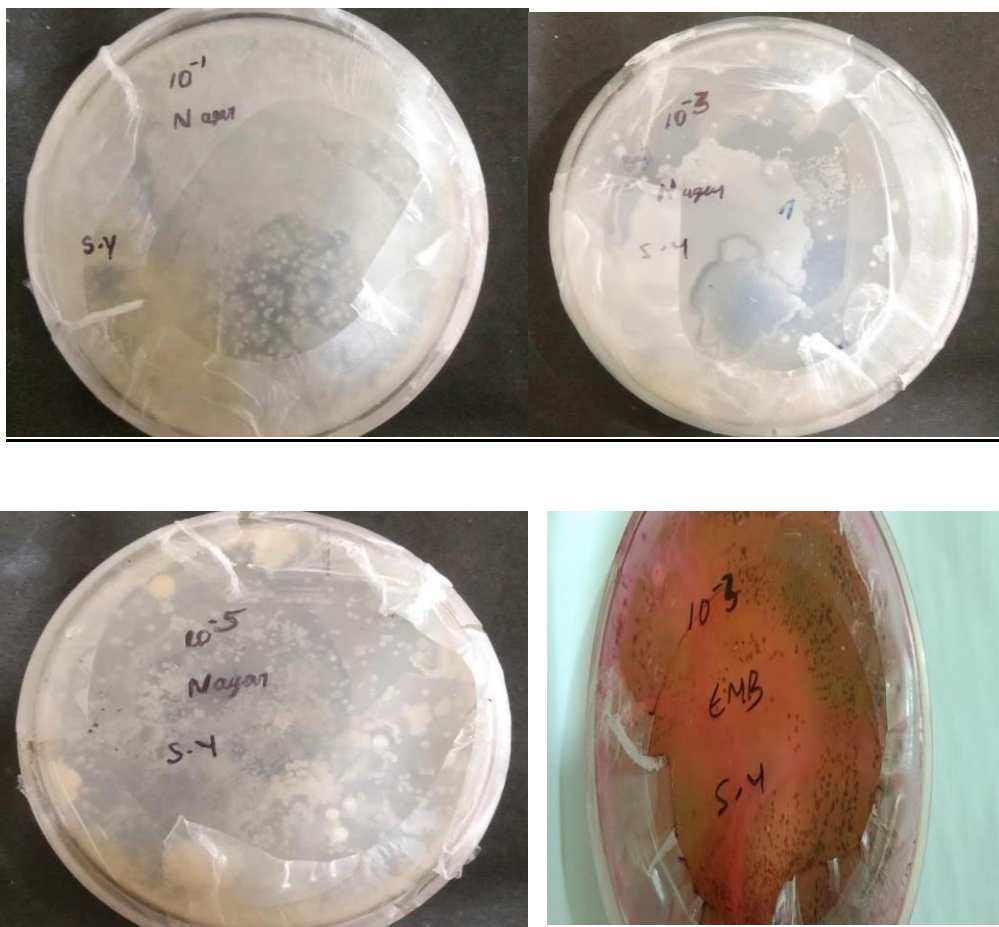


Figure :5.12: Isolation of bacteria from wastewater in different agars

The number of colonies can give an indication of the initial bacterial load or the efficiency of bacterial growth. A high number of colonies presented in EOSIN METHYLENE BLUE Agar proves its efficiency in bacterial growth, while other bacterial plates show low number growth by antibiotics or other factors.

The SEM image of the silver nanoparticles reveals their morphology, which predominantly consists of spherical shapes. The nanoparticles exhibit uniformity in size and shape, contributing to their overall stability and potential applications.

The SEM analysis allows for the visualization of the silver nanoparticles at a microscale level, providing valuable information about their morphology and size distribution. The spherical shapes observed indicate the presence of well-defined nanoparticles with minimal aggregation or irregularities.

The size distribution of the nanoparticles can be observed in the SEM image, ranging from [range] to [range] nanometers. This range reflects the variability in nanoparticle sizes within the sample, which is crucial for understanding their physical properties and potential applications.

The uniformity in size and shape of the silver nanoparticles is advantageous for various applications, including catalysis, sensing, and biomedical applications. Spherical nanoparticles offer enhanced surface area-to-volume ratios, facilitating efficient interactions with other materials and molecules.

Overall, the SEM analysis confirms the successful synthesis of silver nanoparticles with desirable morphology and size distribution, laying the groundwork for further characterization and exploration of their properties and applications.

CHAPTER-6

SUMMARY AND CONCLUSION

The development and examination of metal nanoparticle-infused silica aerogels for water purification signify a notable stride in combating water pollution. This project aimed to devise innovative materials capable of efficiently eliminating contaminants from water, aligning with the global drive to secure clean and safe drinking water universally. The synthesis process involved integrating metal nanoparticles, such as silver, into the silica aerogel structure using a hydrothermal method, leveraging the distinct properties of both silica aerogels and metal nanoparticles. Advanced techniques like scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) were utilized to comprehensively characterize the synthesized metal nanoparticle-infused silica aerogels, providing detailed insights into their structural, morphological, and chemical attributes. The evaluation of these materials' performance in water purification encompassed a series of experiments. Adsorption studies were conducted to assess the aerogels' ability to absorb various waterborne contaminants. Microbial disinfection tests were carried out to evaluate the antimicrobial effectiveness of the metal nanoparticles embedded within the aerogel matrix. Furthermore, crucial water quality parameters such as turbidity, chemical oxygen demand (COD), and heavy metal content were analyzed before and after treatment with the aerogels. The study findings highlighted the promising water purification capabilities of metal nanoparticle-infused silica aerogels. They demonstrated significant adsorption capacities for contaminants, effective antimicrobial properties, and the capability to improve water quality metrics. These outcomes suggest a substantial potential for these materials in water treatment applications, offering a sustainable and efficient approach to addressing water pollution concerns. In summary, the synthesis and assessment of metal nanoparticle-infused silica aerogels represent a significant advancement in developing state-of-the-art materials for water purification and environmental restoration, making a significant contribution to the global mission of ensuring widespread access to clean and potable water resources.

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