

ECO FRIENDLY SYNTHESIS AND CHARACTERIZATION OF MESOPOROUS SILICA NANOPARTICLES FROM HUSK OF ZEA MAYS WASTE

J. Arockiya Mashina¹, D.Carolin Jeniba Rachel^{2*}

PG Department of Chemistry, St. Mary's College (Autonomous), Thoothukudi
Affiliated to Manonmanium Sundaranar University, Tirunelveli, Tamil Nadu, India

ABSTRACT

This study presents a sustainable and eco-friendly method for the synthesis of mesoporous silica nanoparticles (MSNs) utilizing Zea Mays waste, an abundant agricultural byproduct. The synthesis process involves the extraction of silica from Zea Mays waste followed by a sol-gel approach to fabricate MSNs. Characterization of the synthesized MSNs was performed using various techniques including UV visible spectroscopy, scanning electron microscopy (SEM), Fourier-transform infrared spectroscopy (FTIR), (X-ray diffraction) XRD analysis, Energydispersive X-ray analysis (EDAX), Antibacterial Activity. The results demonstrate the successful formation of MSNs with well-defined mesoporous structures and high surface areas. The potential applications of these MSNs in drug delivery, catalysis, and environmental remediation are discussed. Moreover, the utilization of Zea Mays waste as a precursor for MSN synthesis highlights the importance of sustainable practices in nanomaterial production. This approach not only offers a solution for waste valorization but also contributes to the development of green and sustainable nanotechnologies.,

Keywords: Mesoporous Silica nanoparticles, *Zea Mays* waste, Antibacterial activity.

1. INTRODUCTION

Good control of the morphology, particle size, uniformity and disparity of mesoporous silica nanoparticles (MSNs) is of increasing importance to their use in catalyst, adsorption, polymer filler, optical devices, bio-imaging, drug delivery, and biomedical applications. This review discusses different synthesis methodologies to prepare well-dispersed MSNs and hollow silica nanoparticles (HSNs) with tunable dimensions ranging from a few to hundreds of nanometers of different meso structures. The methods include fast self-assembly, soft and hard

templating, a modified Stöber method, dissolving– reconstruction and modified aerogel approaches. In practical applications, the MSNs prepared by these methods demonstrate good potential for use in high-performance catalysis, antireflection coating, transparent polymer–MSNs Nan composites, and drug-release and theranostic systems. [1]In addition to their biocompatibility, key advantages of MSNs are associated with their high surface area and facile surface tenability. Often described as “nano sponges” MSNs can adsorb large amounts of molecules dissolved in solution thanks to this high surface area; on fact 0.5 grams of mesoporous silica nanoparticles possess the same area as an American football field. This highly ordered porosity can be controlled during the synthesis of MSNs. [2]Thanks to the versatility of the silica, it's possible to modify the surfaces of MSNs with specific functional groups that will interact selectively with certain categories of molecules. One can imagine coating the pores with a positive surface in order to attract negatively charged molecules, or using a hydrophobic surface to attract more hydrophobic molecules. The figure below shows an MSN in which the pores are modified to allow molecules with a specific surface functionality (represented as green circles) to adsorb inside the particles, whereas others (red pentagons) are excluded. This type of selective adsorption has numerous uses in drug encapsulation, environmental chemical removal, and various other applications. [3]. The organizational structure of the pores can also be modulated (MCM-41, MCM-48, radial, cubic, wormlike, etc.), as represented in the following figure. The use of one type over the others will depend on the application, as the pore structure controls the release/leakage of molecules loaded inside the pores.

For example, MSNs with the hexagonal pore arrangement can release cargo from both ends of a specific channel whereas in the cubic arrangement, molecules can travel freely inside the intricate network and be released from any pore outlet. In the radial pore arrangement, the molecule can only access a single pore and thus must exit through the pore it entered, making this particular system less prone to premature leaking (in the case of drug encapsulation). [4]Compared to the synthesis of regular silica nanoparticles that generally follow the Stöber process, MSNs are synthesized in a water-based solution in the presence of a base catalyst and a pore forming agent more widely known as a surfactant. Surfactants are molecules the present the particularity to have a hydrophobic tail (alkyl chain) and a hydrophilic head (charged

group, such as a quaternary amine for example). As these surfactants are added to a water-based solution, they will coordinate to form micelles with increasing concentration in order to stabilize the hydrophobic tails as own below. During the early stage of the hydrolysis and condensation of the silica precursor, oligomeric forms of silica appear. Like the final material, these oligomers possess silanol groups (Si-OH) that are deprotonated under the basic conditions of the reaction, forming negatively charged oligomers that can in turn condense on the surface of the positively charged micelles. As the reaction proceeds, the oligomers grow larger around the micelles ultimately forming a hybrid organic/inorganic silica network templated by surfactant molecules. After the reaction, the removal of the template will create the porosity that defines mesoporous silica nanoparticles. [5]

2. MATERIALS AND METHODS

MATERIALS

- Sweet corn husk
- NaOH (LR grade purchased from ISOICHEM).
- HCl (LR grade purchased from ISOICHEM).
- Silica Crucible

METHODS

Preparation of Mesoporous Silica Nanoparticles Sweet Corn Husk

Husk of *Zea mays* Convar is collected washed well with distilled water and dried at 100°C in hot air oven. The dried sample is allowed to cool and crushed into small pieces. The crushed Husk of *Zea mays* Convar is combusted for approximately eight hours at a temperature of roughly 600 °C. After that, it is purified with con. HCl, thoroughly cleaned to get rid of any surplus acid, dried in a hot air oven for about two hours and chilled. Using a mortal pestle, the sample is now thoroughly crushed into finer particles. It was named as SC-H.[6]

3. RESULTS AND DISCUSSION

3.1 UV-Analysis

The UV-Visible spectra for SC-H are observed in the 200-900 nm region. The particle exhibits three absorption peaks at 287nm, 364nm, for SC-H The synthesized mesoporous silica nanoparticles show $n-\pi^*$ due to Si-O-Si Stretching and $\pi-\pi^*$ due to

the Si-OH stretching and π - π^* due to the Si-O group and n- π^* due to Si-O-Si bending transition states of SC-H are indicated by the UV-visible absorption peak.[7][8]

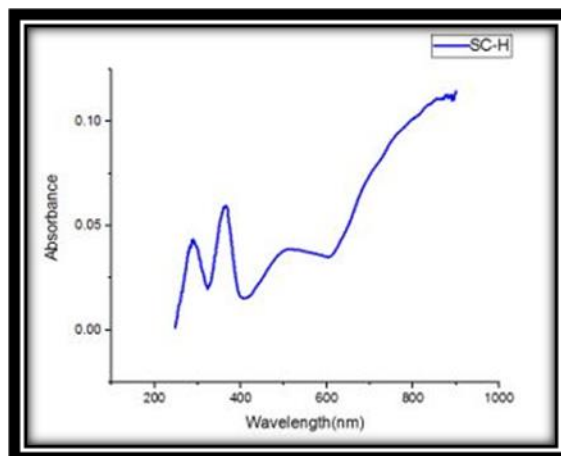


Fig 3.1. UV-Vis spectrum of SC-H(Absorbance Vs Wavelength)

3.2 BAND GAP ENERGY

The Band gap energy for SC-H

The absorption maxima in a nanoparticle could provide information about its band gap energy. From calculation the band gap energies for SC-H are 4.3205 eV, and 3.4065 eV. As a result, the mesoporous silica nanoparticle that were created are Insulators.

3.3 Fluorescence Spectroscopy

The fluorescence graph for synthesised mesoporous silica nanoparticles recorded between the range 200nm-900nm. For SC-H sample, the high intense emission peak at 468nm with 1960.14 intensity. [9] [10]

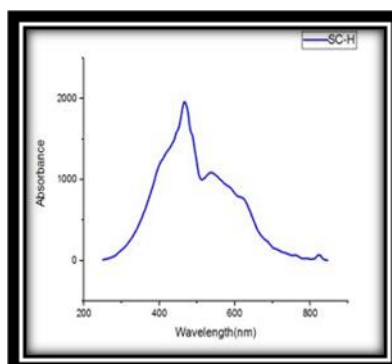


Fig 3.3 Emission spectrum of SC-H(Absorbance Vs Wavelength)

3.4 FTIR studies

3.4.1 FT-IR Analysis for SC-H sample:

The stretching frequency involved in SC-H Nanoparticles were detected by FT-IR Analysis. The IR spectra of the sample were recorded in the range 400-3800 cm^{-1} . [56]The stretching of the peaks at 1512.19 cm^{-1} , 478.35 cm^{-1} , 432.05 cm^{-1} could be designed Si-O-Si Stretching. 1111.09 cm^{-1} , 540.07 cm^{-1} could be designed Si -O-Si bending. 648.08 cm^{-1} , 609.08 cm^{-1} , 586.36 cm^{-1} could be designated Si – OH Stechting. 725.23 cm^{-1} Symmetry stretching.[11][12]

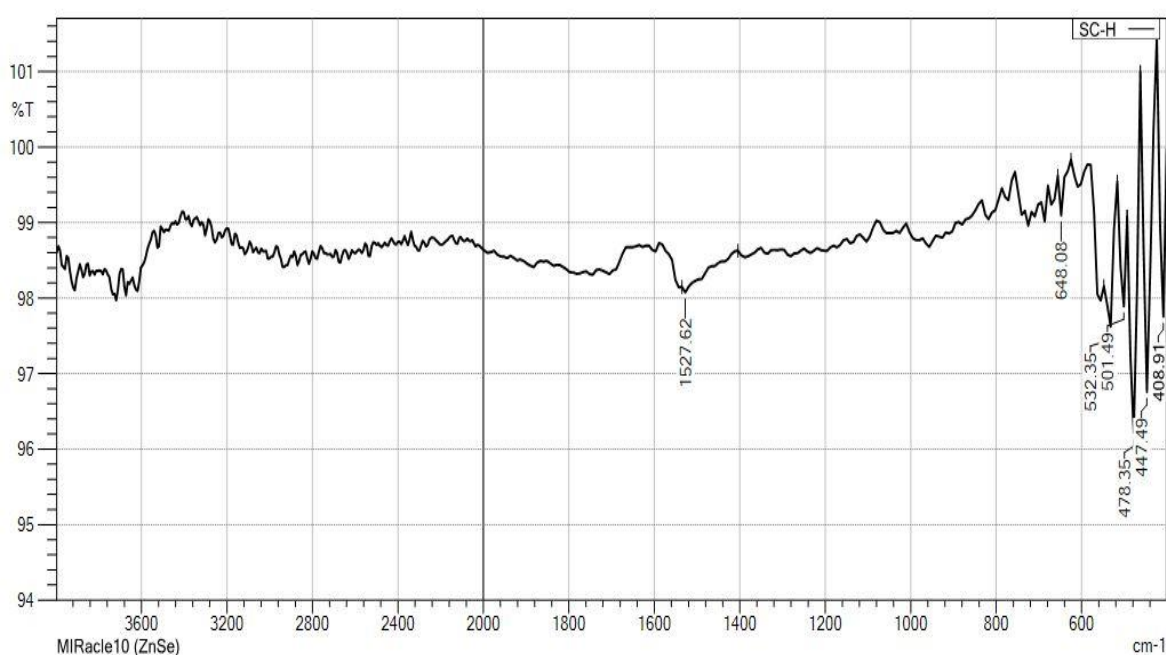


Fig 5.3.2 FT-IR spectra of SC-H

3.5 XRD Calculation

3.5.1 XRD Analysis of SC-H

XRD diffraction with a 2θ ranges 20° to 70° of SiO_2 nanoparticles, using $\text{Cu K}\alpha$ (1.540596Å).The 2θ formed are 28.74°,31.14°, 30.11°,and 50.53°. Based on the XRD diffractogram showing based on JCPDS Card No. 9006289 regarding hkl ,(400),(323), (420), (712).[62]The crystal phase of this type of silica is tetragonal with a lattice parameter of a axis-10.21700Å°, b axis – 7.95790Å° and c axix – 4.95650Å°,

respectively , the addition of high temperature influences the crystallinity and structural phase of material.[13] [14]

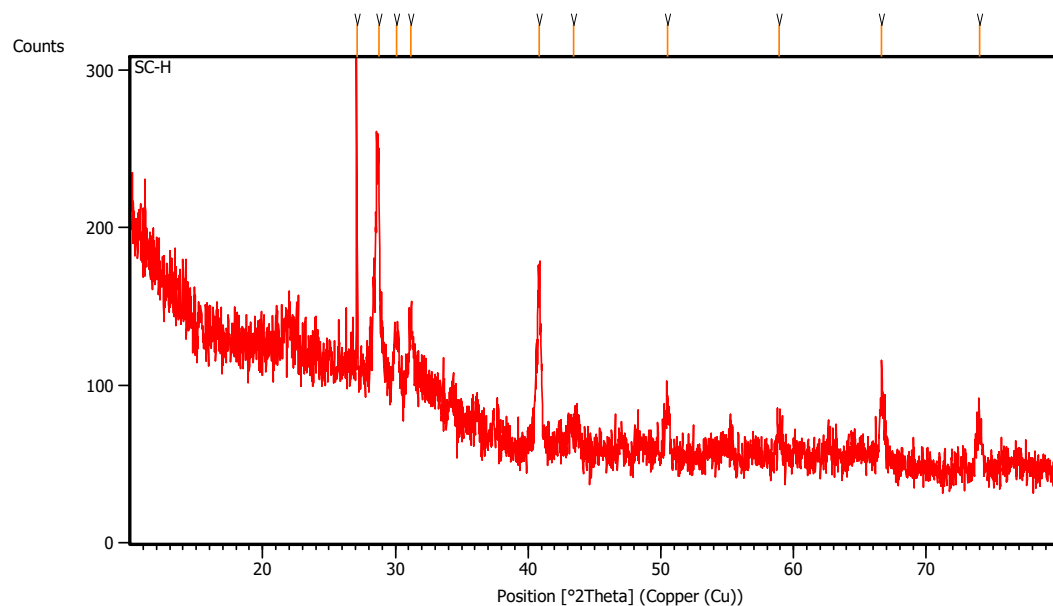


Fig 3.5.2.b XRD spectrum of SC-H

3.5.2.c Calculation of the lattice value

$$a = K\lambda / 2\sqrt{k}$$

$$a = 0.94 \times 1.5406 / 2\sqrt{0.0102}$$

$$a = 1.4481 / 0.2059$$

$$a = 7.0330$$

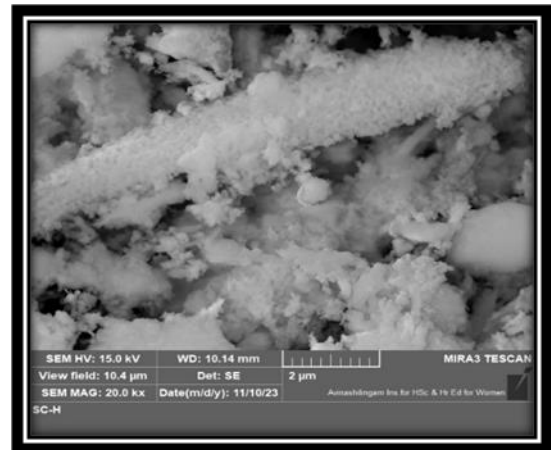
3.6 FIELD EMISSION - SCANNING ELECTRON MICROSCOPY

3.6.1 SEM Analysis for SC-H

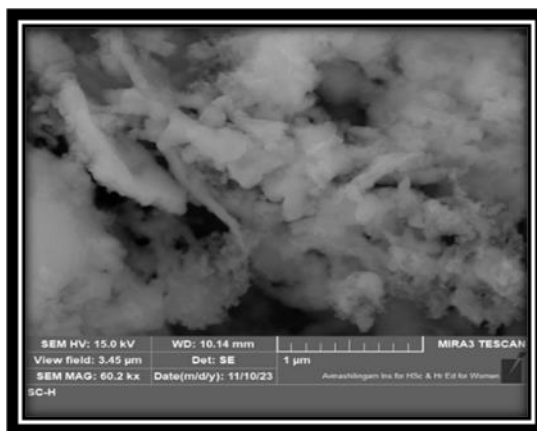
The morphology and size distribution of the synthesized SC-S Nano particles can be measured by the Field Emission – Scanning Microscopy (FE-SEM). The average particle size ranges from (2μm – 500nm).The figure (a,b,c,d,) shows the difference magnification. [15][16]



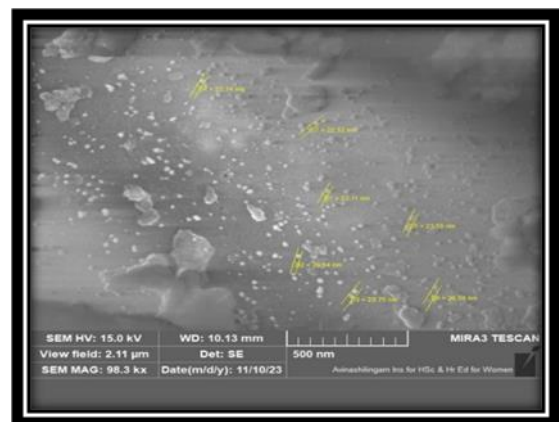
(a)



(b)



(c)



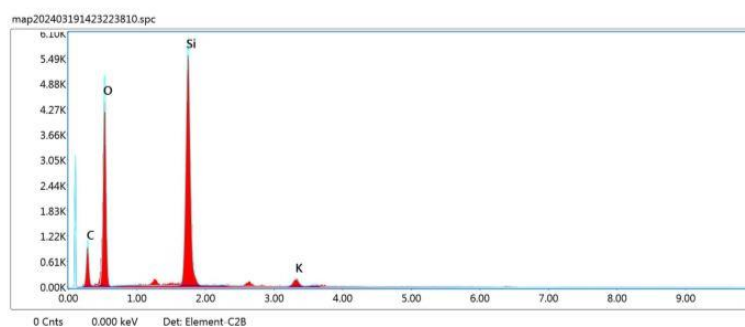
(d)

Fig 3.6.1..a, b, c, d SEM images of various magnifications such as 2µm, 1µm, and 500nm.

3.7 EDAX Analysis

3.7.1. EDAX Analysis of SC- H

Energy dispersive x-ray analysis was used to confirm the elemental composition of SC-H nanoparticles. The figure a,c shown below indicates the presence of silica, oxygen respectively in synthesized SiO_2 nanoparticles.[17][18]



3.7.1. EDAX spectrum of SC- H

4. CONCLUSION

The synthesized mesoporous silica nanoparticles using agricultural wastes viz., corn wastes are characterized using several techniques such as Using EDAX, XRD, and UV, FTIR, and SEM, mesoporous silica nanoparticles was analyzed. Additionally, use antibacterial activity (Disc diffusion method) to ascertain the silica nanoparticle's microbial activity

- ✓ The $\pi\text{-}\sigma^*$ and $\pi\text{-}\pi^*$ and $n\text{-}\pi^*$ transition states of SC-H are indicated by the UV-visible absorption peak.
- ✓ All the three samples show emission properties. The emission wavelength exhibited by the SC-H at 272nm, 290nm, 367nm, respectively.
- ✓ The FT-IR studies reveal Si-O-Si bending, Si-O group, Si-OH stretching, and in the sample SC-H and the band gap energy reveal that all the three synthesised mesoporous silica nanoparticles are act as a insulator.
- ✓ XRD reveals that the mesoporous silica nanoparticles that were generated are crystalline in nature. SC-H have crystal sizes 5.8069nm respectively. SC-H in the orthorhombic phase.
- ✓ The SEM results show that SC-H have a structure that resembles rock and needle.
- ✓ EDAX analysis confirms that the SC-H sample contains silica 58% and oxygen 31% , in were detected.
- ✓ The mesoporous silica nanoparticles that was created was utilized in a variety of applications such as drug delivery, tissue regeneration, adhesion to moist tissues, prevention of bleeding, imaging contrast, shielding organs or tissues from radiation, and enhancing the biocompatibility of medical implants.

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