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SYNTHESIS AND CHARACTERIZATION OF SILICA NANOPARTICLES USING *EICHHORNIA CRASSIPES*

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Abstract

Silica nanoparticles were synthesized by utilizing biological waste *Eichhornia crassipes* from water bodies. Different parts of the weed were used to synthesize the nanoparticles by using two methods. One of the methods employed uses TEOS as precursor for the silica nanoparticles. The biologically synthesized silica nanoparticles (SN1, SN2, SN3) were characterized by Fourier Transform infrared spectroscopy X-ray diffraction analysis, Scanning electron microscopy, and Nano sizer. Silica nanoparticles synthesized using all the three processes showed the characteristic silanol groups in the FTIR spectra. The characteristic absorption bands at 1094cm^{-1}, 1063cm^{-1} & 1058cm^{-1}of SN1, SN2 & SN3 samples showed characteristic Si-O-Si group vibrations.SN1 was selectively analyzed further due to smooth texture obtained. The crystalline & spherical nature of the sample was confirmed from XRD pattern & SEM studies respectively. The particle size of the nanoparticles was found to be in the range of 10-15nm.The SN1 sample was found to exhibit -24.1 mv zeta potential owing to the uniform dispersibility of the synthesized nanoparticles.

***Index Terms*- Silica nanoparticles, *Eichhornia*, silanol group, FTIR**

I. INTRODUCTION

Water hyacinth is a weed that results in thick mats on slow moving water bodies affecting aquatic ecosystems. The dense mats formed restrict commercial activities, production of hydroelectricity, tourism, and transport within a short time due to its rapid dispersal and widespread expansion in the surroundings (Obeid et al. 1984). The rapid growth of water hyacinth poses a great threat to the biodiversity in water bodies (Patel et al, 2012). Water hyacinth (*Eichhornia crassipes*) is a perennial herb that belongs to the family Pontederiaceae. It consists of mature rosette of leaves around a broad stem, on top of which is an inflorescence which consists of violet flowers. It consists of fibrous roots (Williams et al, 2005). There is a negative impact on ecology, economy as well as human welfare caused due to water hyacinth (Degaga et al 2018). The mats formed due to rapid spread of water hyacinth lowers the dissolved oxygen concentration which is lethal to aquatic dwellers. It also results in

obstructing water flow thus increasing flooding in water bodies (Makhanu 1997). The mats also provide a subtle environment for breeding mosquitos (Ecoport 2011). The water hyacinth consists of different minerals in varying proportions (Lara-Serrano et al, 2016). The weed constitutes 95% water and 5 % dry matter. There are reports on the composition of dry matter of which silica is 50%, potassium is 30%, nitrogen constitutes 15% & protein 5% (Degaga AH 2018). Green synthesis of nanomaterial is highly advantageous compared to physical and chemical methods (Fatimah et al, 2018). The present work focused on synthesis of silica nanoparticles (SNPs) by using a blend of chemical and green approach. Water hyacinth extract was used for the synthesis of SNPs. Green synthesis of nanoparticles was carried out in our earlier work successfully using *Tagetes erecta* flower extract (Vakkanti Venkata Sridevi et al, 2022) The silica nanoparticles are synthesized successfully using organic waste like rice husk & sugarcane bagasse (Jansomboon et al, 2016, Lu P et al, 2012) The exclusive properties of SNPs like high surface area, high thermal stability attracts wide application in drug delivery, catalysis, and electronics. Owing to the ease of incorporation of Silica as well as silica nanoparticles into the formulation these are widely employed in different pharmaceutical formulations (Gao et al, 2022) not only as drug carriers, but engineered nanostructures are widely employed in nano pesticide research (Kah et al, 2014). There is high potential in multifunctionalization of nonporous silica nanoparticles which facilitates their widespread applications in varied fields like photo theranostics (Mochizuki C et al 2021). Hence the present work tried to utilize the biological waste causing a menace in water bodies to produce useful nonporous silica nanoparticles which can be widely employed in varied fields like nanomedicine (Li Tang et al, 2013).

II. MATERIALS & METHODS

2.1 Plant Collection and Authentication

Fresh leaves and roots of *Eichhornia crassipes* were collected from Dundigal and Pochampally lake Hyderabad, Telangana. The collected herb was identified and authenticated by Department of Pharmacognosy, Marri Laxman Reddy institute of Pharmacy, Hyderabad, Telangana, India.

2.2 Reagents

All the chemicals used for the present work were obtained from Sigma-Aldrich (Hyderabad, Telangana) through a local chemical supplier. All the reagents received were used as such without further purification. Double distilled water was used for the experiments in the study.

2.3 Synthesis of silica nanoparticles

Silica nanoparticles were synthesized by using 3 methods.

Method I

The collected leaves were separated from the whole plant and dried at 60^0C .30 g of dried leaves were powdered and kept in a muffle furnace in a crucible at 750^0C for 4 hrs. The ash thus obtained was treated with HCl at 60°C under continuous stirring for about 30 min to remove the impurities. The slurry was subjected to several steps of washing using double distilled water until pH is neutral. The obtained mixture is subjected to acid precipitation and alkali extraction. Sodium silicate was obtained by adding NaOH at 80^0C under continuous stirring for 2 hrs. Silica sol was obtained upon acidification (Music S et al 2011) The sol obtained was centrifuged and washed with double distilled water. The formed silica (**SN_1**) was dried completely and preserved for further studies.

Method II

About 20 g of thoroughly washed fresh leaves were boiled for about 30 mins. At 60^0C the extract was filtered. The filtrate was subjected to react with TEOS precursor for about 10 mins with continuous stirring (Stöber et al,1968). The mixture was acidified with continuous stirring. The obtained jelly was heated at 70^0 C for 4 hrs. The white powder obtained was stored in an airtight container until further studies.

Method III

Silica nanoparticles were obtained by using thoroughly washed roots of *Eichhornia crassipes* by employing a procedure similar to Method II. The obtained silica powder was stored in an airtight container for further studies.

2.4 Characterization of Silica Nanoparticles:

Various functional groups present on the surface of synthesized SNPs by all three methods were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) (Bruker Germany) in the scan range of 400–4000 cm^{-1} at a resolution of 4 cm^{-1}. Based on the texture and FTIR spectra obtained further characterization of sample SN1 was carried out. SEM (Scanning electron microscopy) analysis was carried out to visualize the topographical features, crystalline structure, and orientation of the synthesized nanoparticles in the sample using SEM- JEOL Ltd., (Japan) at an accelerated voltage of 15 KV. The zeta potential of the prepared SNPs was determined using MALVERN. XRD patterns of the selected sample of silica nanoparticles was determined by using SHIMADZU -XRD instrument operated at a voltage of 40 kV at a current of 30 mA with Cu-Kα radiation (λ=0.15418nm). The scanning was done in the region of 2θ from 10° to 80°. The size of the nanoparticles was calculated through Scherrer's equation.

III. RESULTS & DISCUSSION

3.1 Physical appearance

Silica nanoparticles of nonporous nature were synthesized using the ash of *Eichhornia crassipes* leaves, fresh leaf extract and root extract and labelled as SN1, SN2 & SN3 respectively. The synthesized SNPs are seen as depicted in figure 1.The physical examination shows different color & texture patterns as represented in table 1

table 1: visual examination of synthesized silica nanoparticles

S.No	Sample	Color	Texture
1	SN1	Grey	Smooth
2	SN2	White	Little gritty
3	SN3	Cream white	gritty

figure 1: synthesized silica nanoparticles

3.2 FTIR Analysis of Silica Nanoparticles

All three samples of synthesized SNPs were subjected to FTIR spectral analysis. Different peaks were obtained in all the three samples as depicted in figure 2,3,4. The sharp peaks at 1094cm^{-1}, 1063cm^{-1} & 1058cm^{-1} in SN1, SN2 & SN3 represent Si-O-Si asymmetric stretching of siloxane group indicating the synthesis of silica nanoparticles. The appearance of absorption bands at 795 cm^{-1}, 792 cm^{-1} & 793cm^{-1} represent the Si-O-Si symmetric stretching vibrations in the samples SN1, SN2 & SN3 respectively. Appearance of a broad peak at around 1630cm^{-1} in SN2 & SN3 samples show the presence of O-H bending vibration due to water molecule. The absorption band at 671cm^{-1} can be assigned to OH deformation. The peak at 3355 cm^{-1} in SN1 is due to the OH stretching of water molecules. The peak observed at 1631 cm^{-1} in SN2 sample is due to the bending vibration of water molecules. The sharp peak at 949 cm^{-1} in SN2 sample is due to silanol groups. The sharp bands at 439 cm^{-1} & 442cm^{-1} of SN2 & SN3 samples respectively due to O-Si-O bending vibrations. (Music S et al 2011).

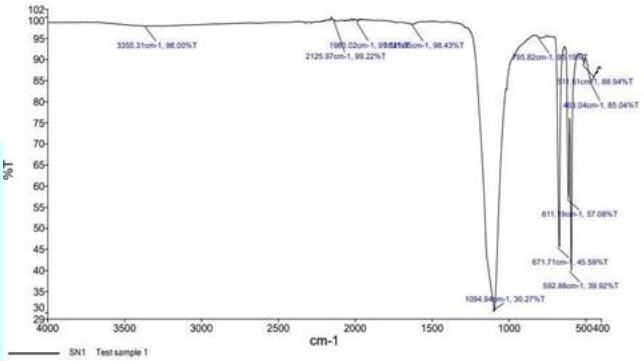

figure 2: FTIR spectrum of SN1

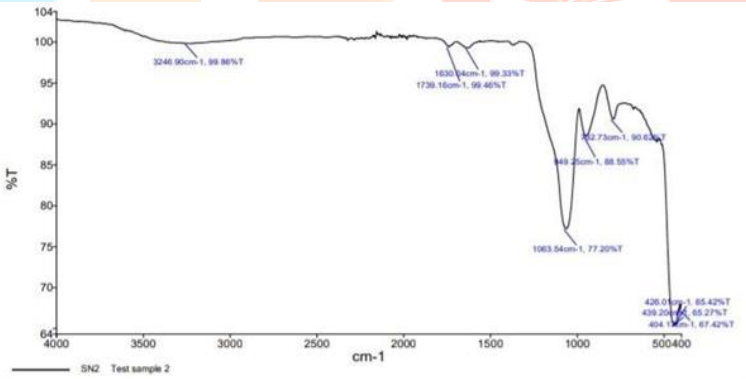

figure 3: FTIR spectrum of SN2

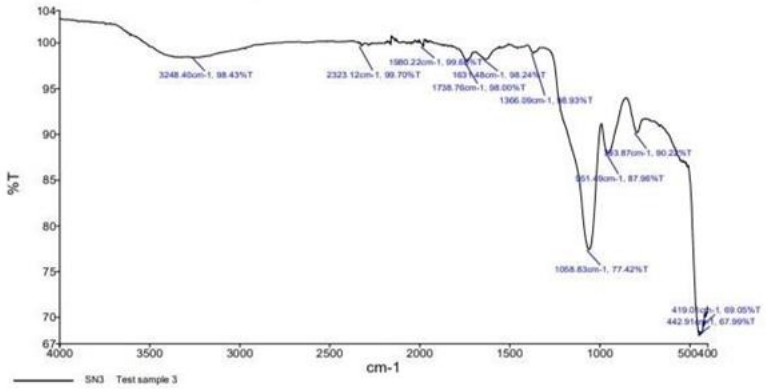

figure 4: FTIR spectrum of SN3

3.3 SEM Analysis

SEM image of selected sample (SN1) was obtained at a magnification of 2000 times. The image obtained represents the crystalline nature of the silica nanoparticles. All the particles represented in the SEM image are spherical in their shape as shown in figure 5. the particles distributed in the size range of 10-15 nm. The SEM image shows not much particle agglomeration representing discrete particles.

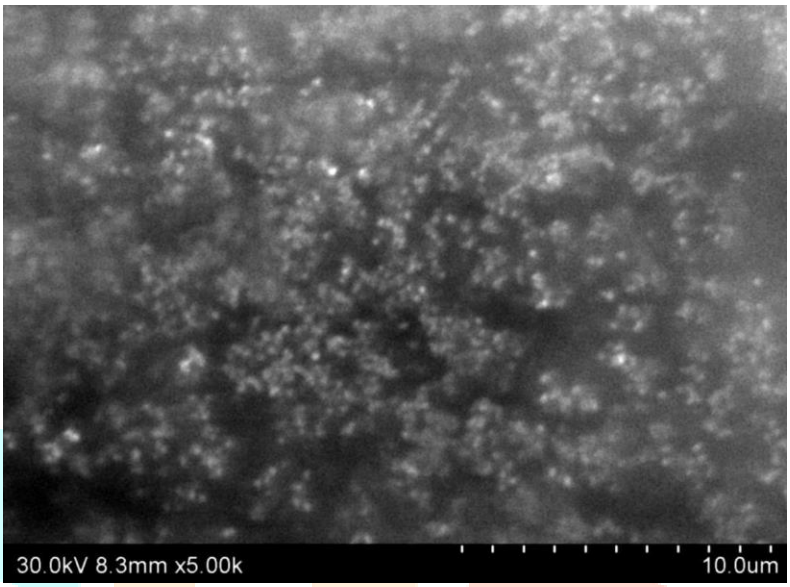

figure 5: SEM image of synthesized SNPs

3.4 XRD

The nature of the silica nanoparticles was further analyzed by XRD studies as depicted in figure 6. Different peaks observed in the XRD pattern at 2Θ values of 19.240, 23.650, 26.45 which shows different diffraction faces of the synthesized SNPs. The XRD peaks obtained represent crystalline nature of silica nanoparticles. The crystal size was determined using Scherrer equation ($D = (0.94 \times \lambda) / (\beta \times \cos \theta)$ where D=Average Crystallite size, β = full width at half maximum (FWHM) in radians, θ =Bragg angle, λ = X-Ray wavelength. The mean particle size of the synthesized silica nanoparticles was observed to be 12.45 nm.

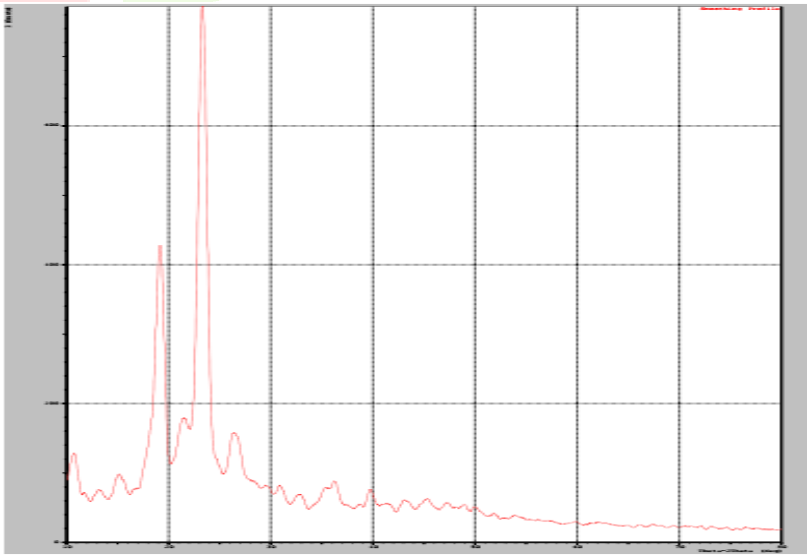

figure 6: XRD Pattern of synthesized SNPs

3.5 ZETA POTENTIAL

The zeta potential of the synthesized silica nanoparticles using Malvern was observed to be -24.1 mv as shown in figure 7. The obtained potential indicates stability of the synthesized nanoparticles limiting the formation of aggregates, agglomerates, or floccules.

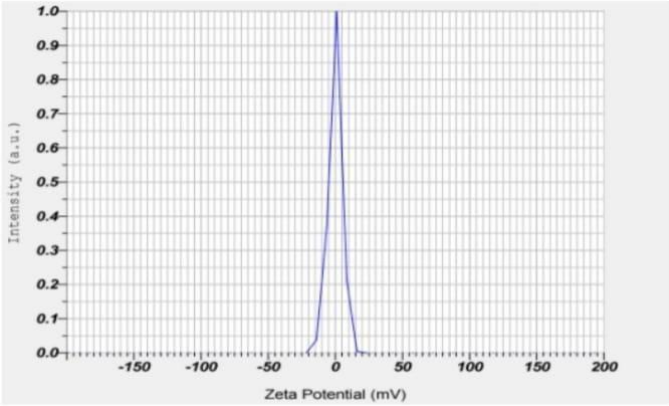

figure 7: zeta potential of synthesized SNPs

IV. CONCLUSION

Silica nanoparticles were successfully synthesized from an invasive weed water hyacinth using different parts of the weed. Owing to the wide applications of silica nanoparticles the green approach to synthesize them from a weed is a promising novel alternative to solve the environmental menace caused by *Eichhornia crassipes*. Further studies can be carried out by using these nanoparticles as carriers in various formulations.

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