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Eco-friendly Synthesis and Antibacterial Properties of Silica Nanoparticles from Rhus coriaria Extract

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Abstract: Due to its sustainable, economical, and environmentally benign methods, the green synthesis of nanoparticles has drawn a lot of attention. Given their distinct physicochemical characteristics and extensive range of uses, silica nanoparticles (SiO_2) are one of the most widely used nanomaterials. In this study, silica nanoparticles (SiO_2) were synthesized via both chemical and green synthesis methods and compared in terms of their structural, chemical, and antibacterial properties. X-ray diffraction (XRD) analysis, performed within a 2θ range of 20–80°, confirmed the amorphous nature of both chemically and green synthesized silica nanoparticles, with a broad peak centered at 2θ = 22°. The average particle sizes were 3.46 nm for chemically synthesized silica nanoparticles and 3.48 nm for green synthesized silica nanoparticles, confirming successful nanoscale synthesis. Fourier-transform infrared (FTIR) spectroscopy revealed characteristic bands for siloxane (Si–O–Si) and adsorbed water molecules, with minor shifts between samples. Antibacterial activity was evaluated against multidrug-resistant Streptococcus sp. (Gram-positive), methicillin-resistant Staphylococcus aureus (MRSA, Gram-positive), and Klebsiella pneumoniae (Gram-negative) using a nanoparticle concentration of 40 mg/mL chemically silica nanoparticles exhibited inhibition zones of 18 mm, 14 mm, and 17 mm, respectively, while green silica nanoparticles showed zones of 16 mm, 15 mm, and 16 mm against the same pathogens. DMSO, used as a negative control, showed no antibacterial activity. Despite slightly smaller inhibition zones, green silica nanoparticles demonstrated comparable antimicrobial efficacy to chemically silica nanoparticles, highlighting the potential of green synthesis as an effective and sustainable route for producing bioactive silica nanoparticles for biomedical applications.
Keywords: Green synthesis, Silica nanoparticles, XRD, FTIR and antibacterial activity.

1. Introduction

Recently, the distinctive characteristics of nanoparticles (NPs) such as their small size, varied morphology, high surface-to-volume ratio, and flexible shape have drawn a lot of interest to nanotechnology [1]. These characteristics make NPs extremely adaptable for a wide range of applications, such as chemical sensing, electronics, diagnostic imaging, medicines, medicinal therapies, and catalysis [2]. Consequently, NPs have emerged as a viable area of study in many different scientific fields.

Metal NPs has garnered attention worldwide because of its diversity in many daily life applications uses. Different synthesis methods were utilized for synthsis of metal NPs, however green synthesis has emerged as a successful and environmentally responsible strategy [3]. Utilizing biological sources like microbes or plant extracts (such as seeds, leaves, or flowers), this process creates NPs in an environmentally responsible and sustainable way. The ease of use, affordability, and minimal environmental impact of the biosynthesis process make it a viable substitute for traditional chemical synthesis.

Silica (SiO_2) nanoparticles have drawn more interest in recent years because of their low toxicity, great biocompatibility, dependability, and controllable particle size [4]. Since silica nanoparticles' performance has been greatly enhanced, they have found extensive use in the food, construction, technology, medical, and agricultural sectors [5].

This work uses extract from *Rhus coriaria* (sumac) as a natural capping and reducing agent to create silica nanoparticles in an environmentally friendly manner. Sumac, a wild plant in the *Anacardiaceae* family, is indigenous to the Mediterranean region, extending from the Canary Islands to Iran and Afghanistan. In addition to being widely used as a spice in cooking, it has long been known to have therapeutic benefits, especially in the treatment of wounds [6]. According to literature, *Rhus coriaria* is a good option for green synthesis because of its high bioactivity including antifungal, antibacterial, anti-inflammatory, antiviral, anticancer, and hypoglycemic activities.

The objective of this study is to synthesis silica NPs from *Rhus coriaria* extract and assess their physicochemical characteristics as well as assess their antibacterial efficacy against Gram-positive and Gram-negative bacterial strains.

2. Materials and methods

2.1 Materials:

The following materials were used in the study without any purification. *Rhus coriaria* (Sumac) seeds were purchased from local market, Sodium metasilicate nonahydrate ($Na_2O_3Si\ 9H_2O$,

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molecular weight 284.2 g/mol, purity ≥98%, Sigma-Aldrich), distilled water, 0.1 M NaOH with a Molecular weight 40 g/mol, Ethanol 96% , and HCL with Molecular weight 36.46 g/mol.

2.2 Methods

2.2.1 Preparation of Rhus coriaria Extract

Rhus coriaria seeds were purchased from local market, dried in a shaded area at room temperature, and then ground using a mixer. A total of 50 g of crushed *Rhus coriaria* seeds were heated in 200 mL of distilled water while being stirred magnetically until boiling for 40 minutes. The mixture was then filtered through filter paper, and the obtained extract was stored in a refrigerator for further use.

2.2.2 Biosynthesis of Silica NPs

A 10 mL solution of 0.1 M sodium metasilicate nonahydrate was added dropwise to 50 mL of freshly prepared *Rhus coriaria* extract while continuously stirring at 60°C. The pH of the solution was adjusted using 0.1 M NaOH, leading to the synthesis of silica NPs, indicated by a color change from colorless to dark green in the solution. The mixture was left to age overnight and then filtered. The obtained precipitate was thoroughly washed with distilled water and methanol four times to remove any impurities. The residue was separated by centrifugation at 4000 rpm for 20 minutes and then dried at 100°C for 6 hours in a furnace to ensure complete removal of residual water [7]. The biosynthesis SiO2 Nanoparticles were prepared using the mentioned method by R. Y. Chiya. *et al.,*[8] with a little modification.

2.3 Chemical Synthesis of Silica NPs

Sol-gel method was used in the silica nanoparticles chemically synthesized [9]. Sodium metasilicate nonahydrate ($Na_2O_3Si \cdot 9H_2O$, molecular weight 284.2 g/mol, purity ≥98%, Sigma-Aldrich) was used as a precursor for the chemical synthesis of silica nanoparticles. Compared to tetraethyl orthosilicate, sodium metasilicate-based silica synthesis has been less studied. Using a molarity calculator, the required mass of sodium metasilicate nonahydrate was weighed and dissolved in distilled water to prepare a 0.1 M solution in a total volume of 100 mL. The solution was maintained under magnetic stirring and heated to 60°C for 1 hour. Hydrochloric acid (0.1 M HCl) and sodium hydroxide (0.1 M NaOH) were added dropwise until the pH reached 6.0. The solution was stirred continuously for 2 hours until it formed a gel-like appearance. The product was allowed to age overnight. To remove any formed sodium salts, the precipitate was washed multiple times with ethanol and distilled water. The purified precipitate was collected by centrifugation at 4000 rpm for 20 minutes (repeated four times). The final product was dried at 100°C in an oven for 6 hours and stored in vials for further characterization and modification.

2.4 Characterization of Synthesized Silica NPs

2.4.1 X-ray Diffraction (XRD)

The crystalline structure of the synthesized silica NPs was analyzed using an X-ray diffractometer (D8 Advance with DAVINCI design, Bruker, Germany) at Sohag University. The measurements were conducted with Cu Kα radiation (λ = 1.5418 Å) at an operating voltage of 40 kV and a current of 40 mA. The diffraction patterns were recorded over a 2θ range of 20–80° with a step size of 0.02° and a time per step of 0.6 s. A silicon zero-background sample holder was used to minimize interference. Data analysis and peak identification were performed using DIFFRAC Measurement Center Version V7.3.0 (32-bit) software, with reference to the Powder Diffraction Files (PDF) from the Crystallography Open Database (COD).

2.4.2 Fourier-transform Infrared Spectroscopy (FTIR)

FTIR analysis was performed to identify functional groups and chemical bonds present in the silica NPs. The spectra were recorded using an FT-IR Alpha spectrometer (Bruker) equipped with a Platinum-ATR module at Sohag University. The measurements were taken over a wavenumber range of 400–4000 cm^{-1} with a resolution of 4 cm^{-1}. Potassium bromide (KBr) pellets were used for sample preparation to enhance spectral quality.

2.4.3 Antibacterial Activity

The antibacterial activity of the synthesized silica nanoparticles was evaluated using agar well diffusion method against two Gram-positive bacterial strains, *Streptococcus sp.* (ATCC 19615) and methicillin-resistant *Staphylococcus aureus (MRSA,* ATCC 43300*),* as well as one Gram-negative strain, *Klebsiella pneumoniae* (ATCC 700603) [10]. The pathogenic bacterial strains were obtained from Department of Medical Microbiology & Immunology, Faculty of Medicine, Sohag University and subculture on nutrient agar plates. Bacteria were identified routinely by morphological and biochemical tests. A single colony from each strain was transferred to 10 mL of sterile nutrient broth and incubated at 37°C for 24 hours under continuous shaking to ensure active bacterial growth. The bacterial suspensions were then adjusted to 0.5 McFarland standard (~1.5×10^8 CFU/mL) using sterile saline to standardize bacterial concentration for the assay [11] (McFarland, 1907).

For the agar well diffusion assay, nutrient agar plates were prepared by pouring 20 mL of sterile molten agar into petri dishes and allowing them to solidify. The standardized bacterial suspensions were uniformly spread onto the agar surface using a sterile cotton swab to ensure even lawn formation. Wells of 6 mm diameter were punched into the agar using a sterile cork borer. Into each well, 100 µL of a 40 mg/mL suspension of either chemically synthesized (CS) or green synthesized (GS) silica nanoparticles (dispersed in DMSO) was added. For controls, 100% DMSO was used as a negative control, while Gentamicin (10 µg per well) was used as a positive control to compare the antimicrobial effectiveness of the nanoparticles with a standard antibiotic. The plates were then incubated at 37 °C for 24 hours to allow diffusion and interaction between the test agents and the bacteria. After the incubation period, the zone of inhibition around each well was measured using a digital caliper to determine the antibacterial efficacy of the silica NPs.

3. Results and Discussion:

3.1 Silica NPs characterization

3.1.1 XRD characterization

XRD analysis was conducted to determine the crystal structure of the synthesized silica nanoparticles within a 2θ range of 20–80°. As shown in Figure.1, the diffraction patterns confirmed the amorphous nature of both chemically synthesized (CS) and green synthesized (GS) silica nanoparticles, as no sharp crystalline peaks were observed the same result mentioned in [12,13]. A broad peak centered at 2θ = 22° was detected, which is characteristic of amorphous silica. The absence of additional peaks further indicated the high purity of the synthesized nanoparticles. The observed peak was identified based on reference COD 9013393 for silica [14]. To estimate the average particle size (D) of the synthesized silica NPs, the Debye-Scherrer equation [15] was applied:

$$D = \frac{0.9\lambda}{\beta Cos\theta} \quad (1)$$

where K is the shape factor (typically 0.9), λ is the X-ray wavelength (1.5418 Å for Cu Kα radiation), β is the full width at half maximum (FWHM) of the peak in radians, and θ is the Bragg angle.

The calculated average particle sizes of the silica (SiO_2) NPs were calculated using the Debye-Scherrer equation, as calculated in [8, 16]. The results indicated that the particle size was 3.46 nm for the chemically synthesized (CS) SiO_2 NPs [17], and 3.48 nm for the green synthesized (GS) SiO_2 NPs. These values highlight the nanoscale dimensions of both sets of NPs, confirming their successful synthesis and consistency in size across both methods.

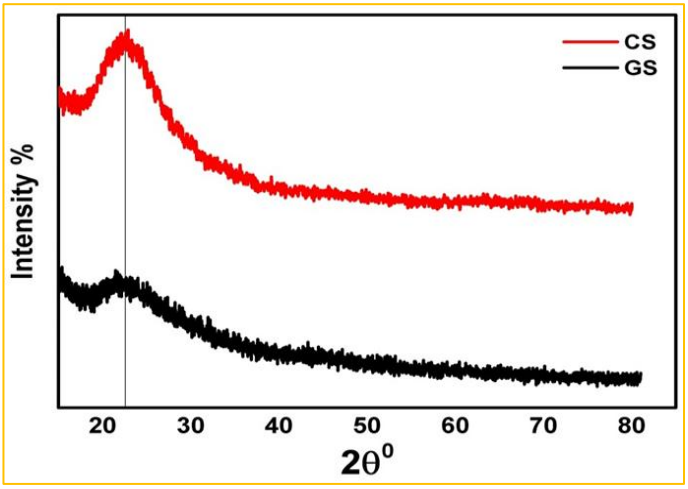

Fig.1: XRD spectra of chemically and green synthesized silica NPs.

3.1.2 Fourier-transform infrared spectroscopy (FTIR):

The FTIR spectra of the chemically synthesized (CS) and green synthesized (GS) silica NPs were obtained after grinding the samples with KBr pellets. Figure.2 presents the FTIR spectra for both samples of NPs. A broad band observed at 3450 cm-1 for CS NPs and 3443 cm-1 for GS NPs corresponds to the stretching vibrations of the O-H group, which are attributed to water molecules on the surface of the NPs. Similarly, the bands at 1636 cm-1 (CS) and 1631 cm-1 (GS) are associated with the bending vibrations of water molecules, further confirming the presence of adsorbed water.

The peaks at 1094 cm-1 and 801 cm-1 in the CS silica NPs are indicative of the asymmetric stretching vibrations of siloxane (Si-O-Si) bonds. For GS silica NPs, these vibrations appear at 1090 cm^{-1} and 795 cm^{-1}, showing a slight shift but consistent with the siloxane bonding. Additionally, the bending vibrations of O-Si-O were observed at 469 cm-1 (CS) and 466 cm-1 (GS), further confirming the silica NPs network.

Overall, the FTIR results confirm the successful synthesis of silica NPs, with the characteristic peaks corresponding to silica and water, verifying the presence of the silica network in both CS and GS NPs. The same FTIR results also described by Jaya V., and Arpita B. [12], and M. S. Emie and S. Masaru [18].

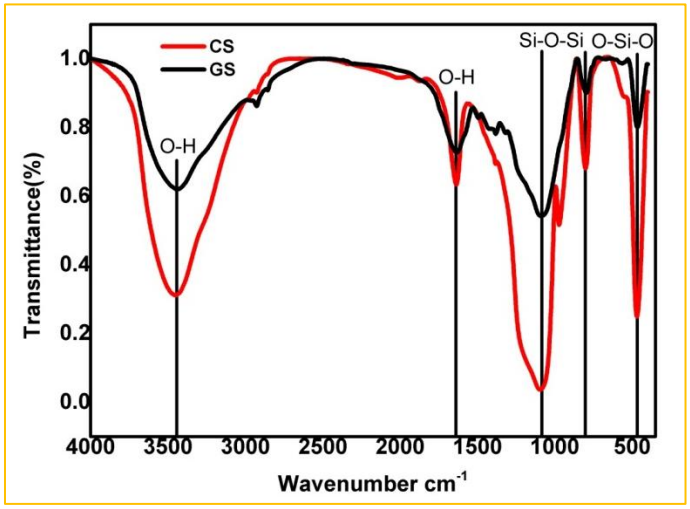

Fig.2: FTIR spectra of chemically and green synthesized silica NPs.

3.2 Antibacterial activity

The antibacterial activity of chemically synthesized (CS) and green synthesized (GS) silica NPs was evaluated against three multidrug-resistant bacterial species: *Streptococcus sp.* (Gram-positive), *Methicillin-resistant Staphylococcus aureus* (*MRSA*) (Gram-positive), and *Klebsiella pneumoniae* (Gram-negative), Figure. 3. The results, as summarized in Table 1, demonstrate that both CS and GS silica NPs exhibit significant antibacterial activity against all three bacterial strains, with inhibition zones ranging from 16 to 18 mm for the tested concentrations (40 mg/mL).

The CS silica NPs showed the largest inhibition zones, with 18 mm for *Streptococcus sp.*, 14 mm for *MRSA*, and 17 mm for *Klebsiella pneumoniae*. The GS silica NPs, on the other hand, exhibited slightly lower but still notable activity, with inhibition zones of 16 mm for *Streptococcus sp.,* 15 mm for *MRSA*, and 16 mm for *Klebsiella pneumoniae*.

For comparison, Gentamicin (10 µg) was used as a positive control and exhibited inhibition zones of 20 mm for *Streptococcus* sp., 19 mm for *MRSA*, and 21 mm for *K. pneumoniae*. These results validate the antibacterial activity of the synthesized silica NPs, which, although slightly less potent than the antibiotic, still showed promising efficacy. DMSO, the negative control, exhibited no antibacterial activity, confirming that the observed effects were due to the nanoparticles and not the solvent.

This study suggests that the green synthesis method, despite the absence of toxic chemicals, is equally capable of producing silica NPs with antimicrobial efficacy comparable to the conventional chemical synthesis method.

The similarity in the antibacterial efficacy between the chemically synthesized and green synthesized NPs can be attributed to the common mechanism of action of silica NPs, which are known to disrupt the bacterial cell membrane, generate reactive oxygen species (ROS), and interfere with cellular processes. The slight differences in inhibition zones between CS and GS NPs might be due to variations in the surface charge, morphology, and size of the particles, which can influence their interaction with bacterial cells. Despite these small differences, the results demonstrate that green synthesized silica NPs can serve as an effective, environmentally friendly alternative to chemically synthesized ones in antimicrobial applications.

These findings are in line with previous studies that have demonstrated the antimicrobial potential of silica NPS Both synthesized chemically and biologically. Where the antibacterial of Silica nanoparticles was examined by Abdu Saeed, et al., [19] against *Pseudomonas aeruginosa, E. coli, Klebsiella pneumonia, Salmonella Typhimurium and Enterococcus Faecalis*, the results showed the highest antibacterial activity against *E. Coli* at 23 ± 2.16 mm and the lowest activity against *Enterococcus Faecalis* at 15 ± 0.94 mm. Also as exmined by Rajiv. B, *et al.,* [16], the antibacterial effects of silica nanoparticles (GS) against the human pathogens (*E. coli* and *Salmonella*), the maximum zone of inhibition on *E. coli* (25 mm) and *Salmonella* (26 mm) was observed. Furthermore, the green synthesis method offers the added advantage of being more environmentally sustainable, as it avoids the use of toxic chemicals and reduces the ecological impact of the production process.

Table.1: antibacterial activity of chemically and green synthesized silica NPs at concentration 40 mg/ml.

Cnocentration 40mg/ml	**Inhibition zone (mm)**		
Sample	***Streptococcus sp.***	***MRSA***	***K.pneumoniae***
Silica NPs (CS)	18	14	17
Silica NPs (GS)	16	15	16
Gentamicin (10 µg)	20	19	21
DMSO	0	0	0

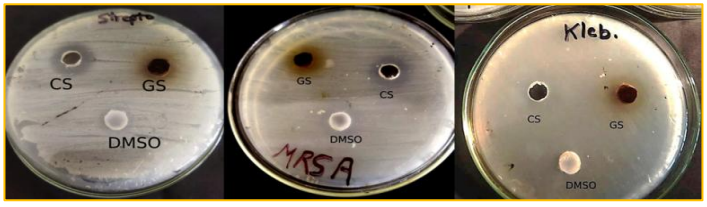

Fig.3: The inhibition zone of chemically and green synthesized silica NPs.

4. Conclusion

This study concluded to compare the synthesis of silica NPs using two different methods, highlighting the effectiveness of the biosynthesis process. The biosynthesis method is an eco-friendly and cost-effective approach that uses *Rhus coriaria* extract as a capping, reducing, and stabilizing agent for the preparation of silica NPs. The XRD analysis confirmed the amorphous nature of the silica NPs, with an average particle size of approximately 3 nm. The FTIR analysis provided insights into the presence of key functional groups in the NPs, further confirming the successful synthesis.

The antibacterial assay demonstrated that the antimicrobial efficacy of the biosynthesized silica NPs was comparable to that of the chemically synthesized NPs. Based on these findings; we conclude that biosynthesis is a viable and effective method for preparing silica NPs, offering an environmentally friendly alternative to the traditional chemical synthesis process. By utilizing a plant extract instead of hazardous chemicals, this method not only reduces the environmental impact but also yields NPs with promising properties for various applications.

CRediT authorship contribution statement:

Conceptualization, Aml Mahmoud Mohammed; methodology, formal analysis and investigation, Aml Mahmoud Mohammed; resources, Alaa Hassan Said; data curation, Aml Mahmoud Mohammed.; writing—original draft preparation, Aml Mahmoud Mohammed; writing—review and editing, Alaa Hassan Said.; visualization, Alaa Hassan Said.; supervision, A.M. Ahmed, H. F. Mohamed, Alaa Hassan Said. All authors have read and agreed to the published version of the manuscript.

Data availability statement

The data used to support the findings of this study are available from the corresponding author upon request.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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