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## Synthesis and characterization of capped silver nanoparticles through green and chemical reduction methods study of their applications

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### ABSTRACT

Silver nanoparticles capped with N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene) benzamide were synthesized using a simple chemical reduction method. The process involved treating aqueous silver ions with a hot ethanolic solution of N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene) benzamide. The resulting silver nanoparticles were spherical, uniform in shape, and had an average size of 34 nm. Their characterization was performed using UV-visible spectroscopy, FTIR, XRD, SEM, and TEM techniques. The capped silver nanoparticles exhibited significant antibacterial activity against various human pathogenic bacteria at different concentrations (25,50,75, and 100 µg). Compared to the standard antibiotic ciprofloxacin, the synthesized nanoparticles demonstrated potent inhibitory effects.

**Keywords:** Antimicrobial, Capping Agent, Silver Nanoparticles, Triazole Derivatives

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#### 1. Introduction:

2-Iminothiazoline derivatives are an important class of five-membered heterocyclic compounds, known for their diverse biological activities. These compounds play a crucial role in drug development for conditions such as hypertension [1], inflammation [2], and cancer [3]. Due to their low toxicity and favorable pharmacokinetic properties, they are recognized as privileged structural motifs in medicinal chemistry. Recently, 2-iminothiazolines have been identified for their antifungal properties [4] and potential use in skin whitening treatments [5].

One notable example is pifithrin (Pft-), a lead compound containing the 2-iminothiazoline core, which was identified through chemical library screening. It has garnered

significant attention for its potential applications in treating neurodegenerative diseases such as Alzheimer's and Parkinson's, as well as stroke, cancer, and other disorders related to various signaling pathways [6-9]. In this study, the 2-iminothiazoline derivative N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene) benzamide is utilized as a capping agent to control the size and stabilization of silver nanoparticles. Additionally, this capping agent enhances the biological activity of the nanoparticles. Synthesis and Characterization of N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene) benzamide 2-Iminothiazoline derivatives are widely studied due to their broad applications [10]. They serve as key building blocks in natural products and are highly valuable scaffolds, particularly in combinatorial and

medicinal chemistry [11-15]. Furthermore, thiazolines are utilized in agriculture as acaricides, insecticides, and plant growth regulators [16,17].

The synthesis of 2-iminothiazoline derivatives is typically achieved through the reaction of thiourea derivatives with substituted  $\alpha$ -bromoketones in the presence of an appropriate base [18,19]. In this work, a similar method was employed for the synthesis of 4-amino-2-iminothiazole, using the reaction between aroylthiourea and bromobenzyl cyanide. Based on literature reports, triethylamine is considered the most effective base for this transformation [20], and it was therefore chosen for this synthesis. The thiourea derivatives were prepared using established procedures, and the final product was fully characterized using spectroscopic techniques, including IR,  $^1\text{H}$  NMR,  $^{13}\text{C}$  NMR, and mass spectrometry.

- IR spectrum: The characteristic  $\text{NH}_2$  stretching bands appeared at  $3340\text{--}3222\text{ cm}^{-1}$ , while the  $\text{C}=\text{N}$  bond was observed at  $1590\text{--}1550\text{ cm}^{-1}$ .
- $^1\text{H}$  NMR: The  $\text{NH}_2$  group appeared as a broad singlet at 3.5 ppm.

## 2. Materials and Methods

All reagents used in the experiment were of analytical reagent (AR) grade. Silver nitrate, procured from National Refinery Pvt. Ltd., was prepared as a 0.1M aqueous stock solution. Sodium borohydride was sourced from Merck, India. Throughout the experiment, organic-free water was used to ensure purity and accuracy.

The UV-visible spectra were recorded using a Shimadzu UV-visible spectrophotometer, with solutions placed in a 1 cm stoppered quartz cuvette. The Fourier Transform Infrared (FTIR) spectra were obtained using a Shimadzu FTIR spectrometer, with samples prepared as KBr pellets. Each spectrum was recorded over 45 scans, and the background spectrum was automatically subtracted. The X-ray diffraction (XRD) technique was used to confirm the formation of a single-phase compound. XRD patterns were recorded using an XPERT-PRO X-ray diffractometer at room temperature, employing  $\text{CuK}\alpha$  radiation ( $\lambda = 1.5406\text{ \AA}$ ) over a broad Bragg angle range ( $30^\circ \leq 2\theta \leq 85^\circ$ ).

The scanning electron microscopy (SEM) micrograph of N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene)benzamide-capped silver nanoparticles was obtained using a NOVA-230 SEM with an operating voltage of 10 kV. For SEM analysis, the sample was suspended in distilled water, sonicated to disperse individual particles, and a few drops of the suspension were deposited onto holey carbon-coated copper grids, followed by drying under an infrared lamp.

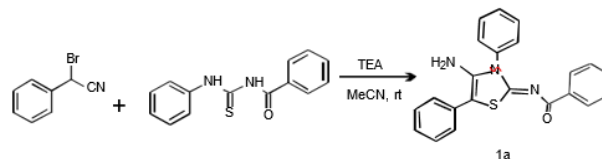
### Synthesis Procedure:

A thiourea derivative (10 mmol) was dissolved in acetonitrile, and triethylamine (10.50 equiv.) was added. The mixture was stirred at room temperature ( $25\text{--}30\text{ }^\circ\text{C}$ ) for 10 minutes, resulting in a pale yellow solution. Then, bromobenzyl cyanide (10.10 mmol)—pre-dissolved in 1 mL of acetonitrile—was added dropwise over 10 minutes. After 15 minutes, a yellow precipitate formed. The solvent

was removed under reduced pressure, and the solid product was filtered and washed with hexane to remove excess triethylamine. The resulting yellow crystalline compound was purified and characterized.

### Characterization of N-(4-Amino-3,5-diphenyl-3H-thiazol-2-ylidene)benzamide (1a):

- **Melting Point:**  $78\text{--}80\text{ }^\circ\text{C}$
- **IR (KBr,  $\text{cm}^{-1}$ ):** 3220, 1670, 1598, 1560, 1219
- **$^1\text{H}$  NMR ( $\text{CDCl}_3$ , 400 MHz):**  $\delta$  6.0 (s, 2H,  $\text{NH}_2$ ), 6.9–8.0 (m, 15H, aromatic)
- **$^{13}\text{C}$  NMR ( $\text{CDCl}_3$ , 100 MHz):**  $\delta$  95.3, 124.1, 126.7, 127.2, 129.8, 131.2, 133.7, 135.5, 136.8, 137.6, 167.0, 174.4, 178.5



Scheme-I: Synthetic approach

## Synthesis of Silver Nanoparticle Assembly

### N-(4-Amino-3,5-diphenyl-3H-thiazol-2-ylidene)benzamide-Capped Silver Nanoparticles

To prepare the silver nanoparticles, 2.5 mL of  $10^2\text{ M}$   $\text{AgNO}_3$  was added to 75 mL of triply distilled organic-free water. Then, 5 mL of  $10^{-2}\text{ M}$  N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene)benzamide (dissolved in hot ethanol) was introduced as a stabilizer under continuous stirring. After 10 minutes of mixing, 2.5 mL of  $10^{-2}\text{ M}$  KI was added dropwise to the solution until a green-yellow  $\text{AgI}$  colloid was formed. The final silver colloid was obtained by adding 20 mg of  $\text{NaBH}_4$  to the  $\text{AgI}$  colloidal solution, followed by continuous stirring for approximately 20 minutes. Throughout the reaction, the color of the colloidal solution changed progressively—initially from green-yellow to nut-brown, then to brown, and finally to black, indicating the formation of silver nanoparticles.

**Formation, Morphology, and Biological Activity of Capped Silver Nanoparticles:** The synthesis, morphology, photophysical properties, and biological activity of N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene)benzamide-capped silver nanoparticles were studied. These nanoparticles, characterized by uniform shape and size, were synthesized via a simple chemical reaction between silver iodide ( $\text{AgI}$ ) and sodium borohydride.

This method offers several advantages, including ease of preparation, user convenience, and uniform particle size and shape, which are crucial parameters for biological evaluation. The formation of stable silver colloids was optimized using UV-visible spectroscopy, while time-dependent UV-visible spectroscopy and scanning electron microscopy (SEM) were employed to monitor nanoparticle formation. A noticeable change in nanoparticle shape was observed during synthesis, providing insights into their growth mechanisms and opening new possibilities for controlled nanoparticle morphology.

### Antimicrobial Studies of 2-Imino-4-Amino Thiazole and Its Silver Nanoparticles

**Tested Human Pathogenic Bacteria:**

The antibacterial activity was evaluated against *Salmonella typhi*, *Vibrio cholerae*, *Shigella dysenteriae*, and *Staphylococcus aureus*, obtained from the Department of Microbiology, S V University, Tirupati, India.

**Preparation of Bacterial Inoculum:**

The bacterial strains were inoculated in Müller-Hinton (MH) broth and incubated at  $35 \pm 2$  °C for 4 hours. The resulting suspensions were diluted with MH broth to achieve a 25% transmittance at 580 nm, measured using a Bausch & Lomb Spectrophotometer (Model Spectronic 20). This turbidity level corresponds to 1 McFarland standard, equivalent to approximately  $3.0 \times 10^8$  CFU/mL, and was used as the inoculum for antimicrobial studies.

**Agar-Well Diffusion Assay**

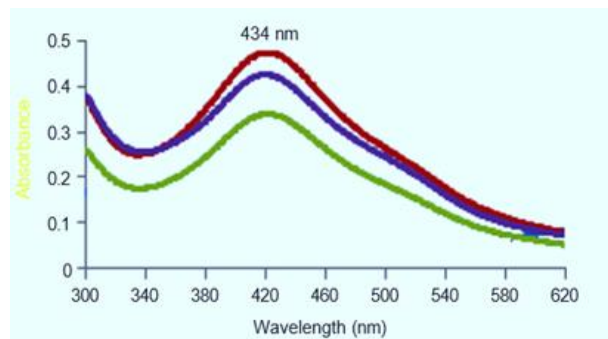
The modified agar-well diffusion method described by Perez et al. [21] was utilized for this study. Each selective medium was inoculated with the test microorganism suspended in Muller Hinton broth. Once the agar solidified, 6 mm diameter wells were punched into it and filled with the required concentrations of the test compounds. Ciprofloxacin was used as a positive control (standard antibiotic), while pure solvents served as negative controls. The results were evaluated based on the size of the inhibitory zone formed around the wells containing the test extract, in comparison with the standard and blank. The diameter of the inhibition zones (in mm) was measured using a Hi-Media zone reader.

**Minimum Inhibitory Concentration (MIC)**

**Determination:** The minimum inhibitory concentration (MIC) of the synthesized silver nanoparticles was evaluated using the broth dilution assay. A series of dilutions ( $10^{-1}$ ) were prepared, with compound concentrations ranging from 100 mg/mL to 100 $\mu$ g/mL in the growth medium. Following inoculation of the bacterial culture, the tubes were incubated at 37 °C for 24 hours. The MIC for each sample was determined by measuring the optical density (OD) at 580 nm using a spectrophotometer (Electronics India). The results were compared with those of the non-inoculated broth (blank). A control was prepared using only the medium and inoculum, without the test compounds.

**3. Results and Discussion****UV-Visible Spectroscopy Analysis**

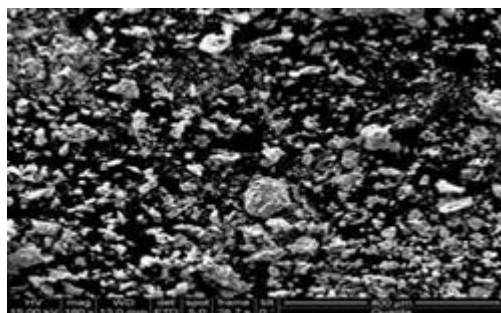
Figure 1 presents the UV-visible spectra of the synthesized silver colloids. The surface plasmon resonance (SPR) band appears broad, indicating the presence of polydispersed nanoparticles. A distinct and narrow absorption band at 434 nm is observed for N-(4-amino-3,5-diphenyl-3H-thiazol-2-ylidene)benzamide-capped silver nanoparticles. The optical absorption spectra of metal nanoparticles are primarily influenced by surface plasmon resonances (SPR), which tend to shift toward longer wavelengths as the particle size increases. The position and shape of the plasmon absorption band of silver nanoclusters are significantly affected by factors such as particle size, surrounding dielectric medium, and surface-adsorbed species. Additionally, the surface plasmon absorption of silver nanoparticles exhibits a short-wavelength band in the visible region, approximately at 409 nm, attributed to transverse electronic oscillations.



**Fig.1.** UV-visible absorbance spectra of 2-imino-4-amino thiazole capped silver nanoparticles in DMSO

**Scanning Electron Microscopy (SEM) Analysis**

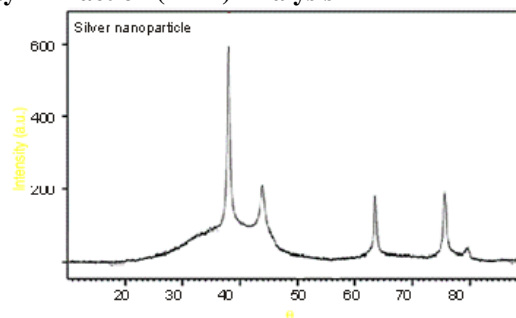
The SEM images of the synthesized 2-imino-4-amino thiazole-capped silver nanoparticles, as shown in Figure 2, reveal that the nanoparticles exhibit a nearly crystalline and spherical morphology.



**Fig.2.** SEM images of synthesized 2-imino-4-amino thiazole capped silver nanoparticles

**Scanning Electron Microscopy (SEM) Analysis**

The SEM images of the synthesized 2-imino-4-amino thiazole-capped silver nanoparticles, as shown in Figure 2, reveal that the nanoparticles exhibit a nearly crystalline and spherical morphology. The variation in particle size is likely due to the different formation times of the nanoparticles during synthesis.

**X-ray Diffraction (XRD) Analysis**

**Fig. 3.** XRD patterns 2-imino-4-amino thiazole capped silver nanoparticles

The XRD spectra of the silver nanoparticles, recorded over both small and wide-angle regions (Figure 3), confirm their crystalline nature. The small-angle XRD spectrum suggests a tendency for the nanoparticles to form organized structures, as indicated by the distinct peaks. The

broadening of these peaks is attributed to the nanocrystalline nature of the silver nanoparticles. When compared with standard database values, the diffraction peaks correspond to a face-centered cubic (fcc) silver crystal structure. Notably, three characteristic peaks at  $2\theta$  values of  $38.299^\circ$ ,  $64.853^\circ$ , and  $77.542^\circ$  correspond to the (111), (200), and (220) planes, respectively, confirming the crystalline phase of the silver nanoparticles.

**X-ray Diffraction (XRD) Analysis:** From Figure 3, it is evident that 2-imino-4-amino thiazole-capped silver nanoparticles predominantly exhibit an amorphous nature with broad diffraction peaks. The diffraction patterns of nanometer-sized particles are significantly influenced by their size and structural characteristics. Smaller particles exhibit distinct diffraction patterns, which vary with both size and structural type. As the particle size increases, the diffraction profiles evolve steadily, revealing more structural details without abrupt transitions. This principle forms the basis for diffraction pattern analysis, which helps extract structural insights from experimental data. The presence of diffraction peaks at scattering angles ( $2\theta$ ) around  $38\text{--}39^\circ$ , corresponding to the (111), (200), (220), and (311) planes, confirms the crystalline nature of the silver nanoparticles.

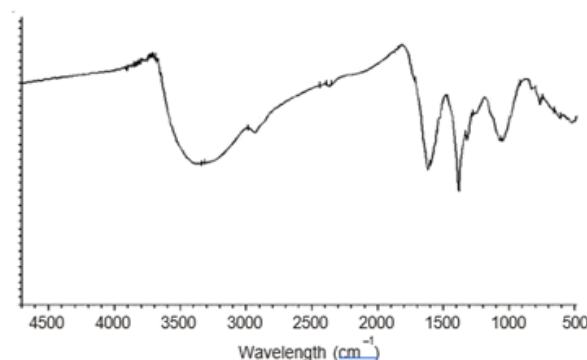
It is well known that as crystallite size decreases, XRD patterns exhibit peak broadening and overlapping reflections. This broadening is inversely proportional to the crystallite size, as described by Scherrer's equation, where the diffraction angle ( $2\theta$ ) influences the calculated size. The total diffracted intensity of a Bragg reflection results from the sum of independent diffraction contributions from individual unit-cell columns within the crystallite. Importantly, the Scherrer formula provides an estimate of the apparent crystallite size, representing a volume-weighted average column length rather than the absolute crystallite size. The difference between experimental and theoretical values further supports this approximation, reinforcing the structural interpretation of the nanoparticles.

#### FT-IR Analysis

The FT-IR spectra (Figure 4) of 2-imino-4-amino thiazole-capped silver nanoparticles closely resemble those of the free 2-imino-4-amino thiazole molecule, confirming the incorporation of organic molecules into the nanoparticle structure. However, a notable difference in peak intensity is observed between the free molecule and the capped nanoparticles.

This difference is attributed to the formation of a closely packed amino layer around the nanoparticles, which constrains molecular motion. The steric hindrance primarily affects transverse vibrational modes (such as rocking and wagging motions) more significantly than longitudinal modes (such as stretching vibrations).

Additionally, the C–N stretching mode shifts due to the proximity of the C–N bond to the silver nanoparticle surface, suggesting the formation of a chemical bond between nitrogen (N) and silver (Ag) atoms. This further confirms the successful capping of silver nanoparticles by 2-imino-4-amino thiazole.



**Fig.4.** FT-IR spectra of 2-imino-4-amino thiazole capped silver nanoparticles

#### FT-IR Analysis

The FT-IR spectrum of free 2-imino-4-amino thiazole exhibits an azo group absorption in the  $1620\text{--}1597\text{ cm}^{-1}$  region. Two distinct bands observed between  $1384\text{ cm}^{-1}$  and  $1319\text{ cm}^{-1}$  correspond to the C–N stretching vibrations, while the band at  $767\text{ cm}^{-1}$  is attributed to C–S stretching mode. Overall, the vibrational spectrum of silver nanoparticles capped with 2-imino-4-amino thiazole closely resembles that of the free molecule, confirming successful capping.

#### Particle Size Analysis Using TEM

Transmission electron microscopy (TEM) was employed to examine the synthesized silver nanoparticles. A sample was prepared by drying a small drop of a freshly synthesized yellow sol on a carbon-coated 200-mesh copper grid. The TEM image, shown in Figure 5, reveals that the silver nanoparticles are spherical in shape with an average size of  $34 \pm 2\text{ nm}$ .

<b>Table-1.</b> Antibacterial Activity of Compounds Against Human Pathogens				
	Zone of inhibition (mm)*			
	S. typhi	V.cholera	S.dynteriae	E.faecaulit
Compound Ia	6	8	6	7
AgNPs+	13	11	13	12
DMSO	8	7	7	7
Ciprofloxacin	17	17	15	15
+50 $\mu\text{g}$ of compound (1 $\mu\text{g}/\mu\text{L}$ concentrated), *6 mm is the well size				

Type of human pathogenic bacterial sps.	Causing disease	Zone of inhibition (mm) at different doses			
		25 µg	50 µg	75 µg	100 µg
Salmonella typhi	Typhoid fever	11	14	16	17
Vibrio cholera	Cholera	15	14	16	18
Shigella dysenteriae	Dysentery	12	14	15	13
Enterococcut faecalit	Gastro, intestinal infections	13	12	13	15

#### 4. Conclusion

N-(4-Amino-3,5-diphenyl-3H-thiazol-2-ylidene)-benzamide capped silver nanoparticles with a uniform size and shape were synthesized using a simple and efficient method. Their morphology, photophysical properties, and biological activities were thoroughly investigated. The 2-imino-4-amino thiazole-capped silver nanoparticles exhibited notable antibacterial activity against several human pathogenic bacteria, including *Salmonella typhi*, *Vibrio cholerae*, *Shigella dysenteriae*, and *Enterococcus faecalis*. The compound demonstrated a dose-dependent inhibitory effect, with the zone of inhibition increasing as the concentration of the compound increased. Notably, the synthesized silver nanoparticles proved to be a potent antibacterial agent, exhibiting superior inhibitory activity compared to the standard antibiotic.

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