

SYNTHESIS AND STRUCTURAL CHARACTERIZATION OF NEW COMPLEXES BASED ON SILVER NANOPARTICLES, DITHIOOXAMIDE REAGENT, AND CETYLTRIMETHYLAMMONIUM BROMIDE

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Received 04.05.2025

Accepted 19.09.2025

Abstract: In this study, the green synthesis of silver nanoparticles (AgNPs) was carried out, and the physicochemical properties of the complexes formed between AgNPs, dithiooxamide (R) reagent, and cetyltrimethylammonium bromide (CTAB) were investigated. The formation and structural features of the resulting complexes were confirmed by ultraviolet-visible (UV-Vis) spectroscopy, infrared (IR) spectroscopy, and X-ray diffraction (XRD) analysis. UV-Vis spectral data revealed bathochromic shifts and variations in absorption intensity, indicating increased electron density upon complex formation. The IR spectra exhibited characteristic signals corresponding to coordination bonds formed between functional groups. XRD analysis demonstrated that the initial reagent possessed a highly crystalline structure; however, upon complexation with AgNPs, a reduction in particle size and a tendency toward amorphous structure were observed.

Keywords: binary complex, ternary complex, silver, nanoparticles, dithiooxamide

DOI: 10.65382/2221-8688-2026-1-138-146

1. Introduction

In recent years, the synthesis of nanomaterials, particularly metal-based nanoparticles, and their application in functional complexes have become one of the prominent research areas in analytical chemistry. Within this context, silver nanoparticles (AgNPs) have attracted significant attention due to their high specific surface area and unique optical and electrical properties [1–5]. Owing to their unique physicochemical properties, AgNPs have been extensively investigated for a wide range of applications. They demonstrate strong antimicrobial activity against bacteria, fungi, and viruses, making them promising candidates for antimicrobial coatings and medical devices. Additionally, AgNPs act as efficient catalysts in various chemical reactions, including oxidation and reduction processes. Their distinctive optical properties enable their use in optical and colorimetric sensors for detecting ions and biomolecules. Furthermore, due to their biocompatibility and functionalization potential,

AgNPs are explored in medical nanomaterials, such as drug delivery systems, imaging agents, and wound healing applications [6–9]. AgNPs possess a wide range of potential applications in fields such as antimicrobial systems, catalytic reactions, and the development of sensor technologies. Zhang, L et al. (2009) demonstrated that AgNPs not only exhibit strong antimicrobial activity but also show enhanced stability and functional performance when incorporated into systems with other functional materials [10]. The effective utilization of AgNPs strongly depends on the controlled modification of their surface properties. For this purpose, cationic surfactants—particularly cetyltrimethylammonium bromide (CTAB)—are frequently employed. CTAB plays a crucial role in stabilizing nanoparticles and promoting the formation of ordered coordination complexes. Its amphiphilic structure allows strong interactions with the silver surface, leading to bilayer formation and preventing particle aggregation.

Several studies have shown that surfactants not only reduce the surface energy of nanoparticles but also regulate their size, shape, and surface reactivity, which in turn expands their range of potential applications. For instance, Ghosh et al. (2012) reported that AgNPs stabilized with plant extracts and surfactants demonstrated synergistically enhanced biological activity compared to non-stabilized systems [11]. Among the reagents of interest in this field are dithiooxamide. Dithiocarbamide (R) ligands form complexes on the surface of silver nanoparticles (AgNPs) through coordination bonds. The sulfide (-S-) and amino (-NH₂) groups of the ligand strongly interact with silver ions. Cetyltrimethylammonium bromide (CTAB) acts as a stabilizing agent, providing steric and electrostatic stabilization, preventing nanoparticle aggregation, and ensuring homogeneous dispersion. As a result, the AgNP-dithiocarbamide-CTAB complex exhibits enhanced stability and functional properties. These structural features enable dithiooxamides to form strong and selective coordination bonds with soft metal ions such as Ag⁺, Cu⁺, and Hg²⁺. Tiekink et al. (2006) reported that dithiooxamide ligands are efficient chelating agents capable of producing stable crystalline complexes with characteristic spectroscopic signatures [12]. The ability of such ligands to create selective complexes with transition metals also makes them attractive in analytical chemistry, particularly in the design of sensitive and selective spectrophotometric methods [13]. Dithiocarbamate ligands exhibit superior stabilization of AgNPs compared to other ligands, such as thiols and amines. Previous studies have shown that thiol-capped AgNPs aggregate under certain conditions and have shorter stability, whereas dithiocarbamate ligands provide strong coordination and enhance the long-term stability of the complexes. This property makes dithiocarbamate ligands particularly effective for analytical, catalytic, and biomedical applications [14]. The size of AgNPs also directly affects the structural stability of the resulting complexes. Smaller nanoparticles have a higher surface-to-volume ratio, which increases ligand binding efficiency and promotes uniform complex formation. Consequently, complexes formed with smaller AgNPs exhibit greater structural stability, reduced aggregation, and

more consistent functional properties, whereas larger nanoparticles may lead to weaker ligand coordination and lower stability [15]. Thus, both the advantages of dithiocarbamate ligands and the effect of AgNP size directly determine the stability and functionality of the complexes, enhancing the scientific value of our work for analytical and catalytic applications.

Recent works have further highlighted the growing interest in combining AgNPs with organic ligands and surfactants to create multifunctional nanocomplexes. For example, Kuliyeve et al. (2020) investigated silver complexes with thio- and azo-containing ligands for application in extraction-photometric determination of metal ions while other studies have demonstrated that hybrid systems based on AgNPs and surface-active molecules could serve as promising candidates for nano-sensors and catalytic platforms [16]. Zalov et al. (2025) demonstrated that quaternary ammonium salts formed from triethanolamine with hexadecanoic and heptadecanoic acids generate colloidal solutions in water and facilitate oil dispersion. These findings are directly relevant to our study, as CTAB stabilization in our AgNP systems similarly enhances nanoparticle dispersion and analytical performance [17]. Rashidova et al. (2025) investigated nickel, cobalt, and phosphorus-based catalysts and showed that metal-ligand interactions directly affect the catalysts' stability and catalytic properties. These results are directly related to our work, as dithiooxamide ligands in AgNP complexes similarly enhance nanoparticle stability and functional properties [18].

The main objective of the present study is the synthesis of novel complex compounds based on silver nanoparticles, dithiooxamide-structured R reagents, and CTAB, as well as the investigation of their structural characteristics using infrared (IR) spectroscopy, X-ray diffraction (XRD), and ultraviolet-visible (UV-Vis) spectroscopy. Preliminary studies have shown that stable complex structures are formed as a result of the interactions between AgNPs, the reagent, and the surfactant in these systems. The results obtained indicate that such complexes are promising for applications in selective analysis, sensor-based technologies, and the design of targeted nanomaterials.

2. Experimental part

2.1. Materials. Silver nitrate (AgNO_3 , PLC 141459, 98% chemically pure), soluble starch ($(\text{C}_6\text{H}_{10}\text{O}_5)_n$, PLC 121096, 98% chemically pure), β -D glucose ($\text{C}_6\text{H}_{12}\text{O}_6$, CAS No.50-99-7); sodium hydroxide (NaOH , PLC 1416x87), cetyltrimethylammonium bromide (CTAB, AB 117004), ethanol ($\text{C}_2\text{H}_5\text{OH}$, CAS No. 64-17-5, 95%), dithiooxamide ($\text{C}_2\text{H}_4\text{N}_2\text{S}_2$, CAS No. 79-40-3, 98% chemically pure).

2.2. Synthesis of Ag Nanoparticles. The synthesis of silver nanoparticles was carried out as follows: 100 mL of 0.01 M silver nitrate (AgNO_3) solution was mixed with 150 mL of 1% starch solution. In parallel, 100 mL of 0.07 M sodium hydroxide (NaOH) solution was combined with 100 mL of 0.2 M glucose solution. The NaOH -glucose mixture was then added to the AgNO_3 -starch mixture, and the resulting solution was stirred continuously for 30 minutes. A dark brown coloration appeared immediately, indicating the formation of a colloidal silver nanoparticle solution. Following synthesis, to remove unreacted ions and impurities, the obtained nanoparticle dispersion was purified by ultracentrifugation (Eppendorf R 5430, 12,000 rpm), followed by repeated washing with a water-ethanol mixture. It is worth noting that the starch in the reaction medium acted both as a reducing and stabilizing agent, while NaOH served as a catalyst to accelerate the reaction. Glucose functioned as the primary reducing agent, facilitating the conversion of silver ions into silver nanoparticles [19-22].

2.3. Synthesis of the Ag-R-CTAB-Based Complex. For the synthesis of the Ag-R-CTAB-based complex, 0.012 g (10^{-3} mol) of the R reagent was accurately weighed using an

analytical balance. For the preparation of the R reagent solution, an accurately measured quantity of the substance was dissolved in ethanol and diluted to a final volume of 100 mL using a volumetric flask. Subsequently, 50 mL of the prepared R solution was measured and transferred into a 100 mL beaker, followed by the addition of 10 mL of 0.01 M silver nanoparticle (AgNPs) solution. The mixture was stirred on a magnetic stirrer for 2 hours. During the stirring process, initial color changes were observed, indicating the formation of a binary Ag^+ -R complex. Next, another 50 mL portion of the R solution was treated again with 10 mL of 0.01 M AgNPs solution and stirred under the same conditions. Then, 5 mL of CTAB solution was added, and the stirring was continued for an additional 2 hours. At this stage, further color changes were recorded. CTAB acted as a stabilizing agent, playing a crucial role in stabilizing the complex formed between the silver nanoparticles and the R reagent. As a result, a ternary Ag-R-CTAB complex was successfully synthesized. The obtained complexes were isolated by drying in a muffle furnace at 100°C for 2 hours.

2.4. Research methods. X-ray diffraction (XRD) patterns of the silver nanoparticles and their complexes with the reagent were recorded at room temperature using a Rigaku Mini Flex 600 diffractometer.

Ultraviolet-visible (UV-Vis) absorption spectra were obtained at room temperature using a Specord 210 spectrophotometer in the wavelength range of 200–900 nm. Infrared (IR) spectra were recorded using a Varian 640-IR spectrometer at room temperature in the wavenumber range of 400–4000 cm^{-1} .

3. Results and Discussion

Fig. 1 shows the X-ray diffraction (XRD) pattern of the synthesized silver nanoparticles. The analysis confirms that the obtained nanoparticles possess a well-defined crystalline structure, with no evidence of an amorphous phase. Distinct diffraction peaks were observed at 2θ values of 38.10° (111), 44.43° (200), 64.36° (220), 77.33° (311), 81.28° (222), 110.25° (331),

and 114.81° (420), which correspond to the characteristic crystallographic planes of metallic silver. These peaks are consistent with the standard face-centered cubic (fcc) structure of silver and serve as confirmation of the material's phase purity. To determine average crystallite size, the Debye-Scherrer relationship was utilized, accounting for peak broadening

observed in the XRD pattern. The equation incorporates variables such as the wavelength of the X-ray source (Cu K α , $\lambda = 1.5406 \text{ \AA}$), the shape factor K, FWHM (β), and the diffraction

angle (θ). The average crystallite size, calculated from the XRD diffractogram using the Debye–Scherrer formula (1), was found to be 20 nm.

$$D = \frac{K}{\beta \cos \theta} \quad (1)$$

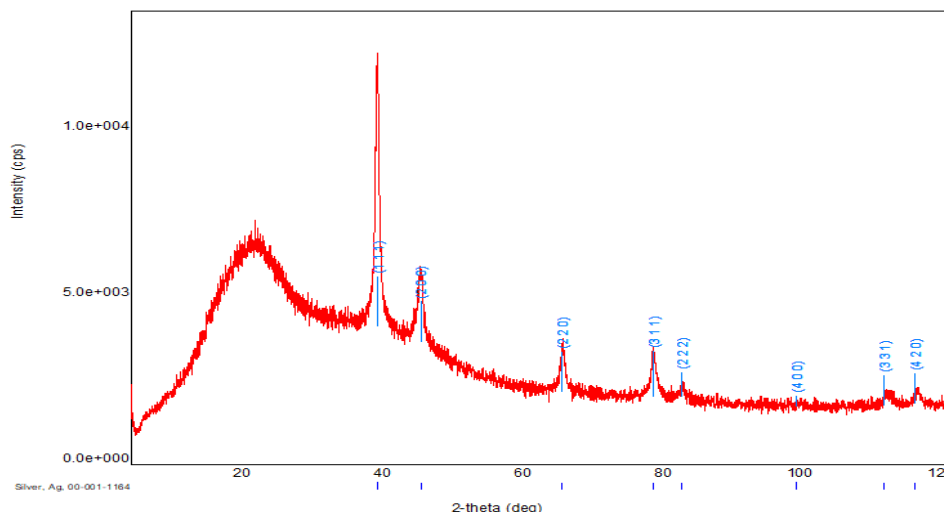


Fig. 1. XRD pattern of the synthesized silver nanoparticles

Fig. 2 presents the UV-Vis spectra of Ag nanoparticles and their complexes formed with the dithiooxamide reagent and CTAB. The UV-Vis spectrum of the R-type ligand demonstrates distinct absorption bands within 240–300 nm and 360–400 nm intervals, which are attributable to

electronic transitions of $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ types, indicating conjugated system activity. Upon complex formation, the solution color changes from light orange to deep red, which is accompanied by the appearance of a new absorption band in the 390–420 nm region.

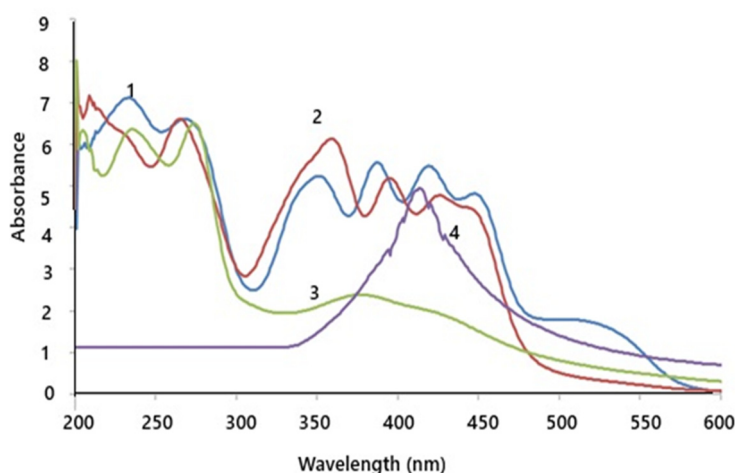


Fig. 2. UV-Vis spectra of R (1), Ag–R (2), Ag–R–CTAB (3), and silver nanoparticles (4)

This shift is attributed to an increase in electron density and delocalization of π -electrons, characteristic of a bathochromic shift [23–25]. The darkening of the color visually confirms the formation of a more symmetric complex and the delocalization of electrons

within the system. The addition of CTAB causes the Ag–dithiooxamide solution to gradually transition to lighter tones, accompanied by a decrease in absorption intensity. The spectral bands become smoother and more symmetric, indicating enhanced structural stability of the

complex. This effect can be explained by CTAB creating a stabilizing environment around the complex, preventing aggregation of colloidal particles, and maintaining a more monodisperse system. A protective layer forms between Ag^+ ions and dithiooxamide, reducing particle clustering and causing only minor energy changes in electronic transitions [26-30].

The observed darkening of the color is associated with the formation of the complex and the delocalization of π -electrons. Introduction of CTAB induces a notable shift in the color of the Ag–dithiooxamide solution, gradually transitioning to lighter tones. This is paralleled by a decrease in absorbance intensity and refinement of spectral band shapes, suggesting improved structural stability. Simultaneously, the absorption intensity decreases, and the spectral peaks become smoother and more symmetric, indicating stabilization of the

complex structure. This phenomenon can be explained by CTAB creating a stabilizing environment around the complex, preventing aggregation of colloidal particles and maintaining the system in a more monodisperse state. During CTAB addition, a protective layer forms around the complex between Ag^+ ions and dithiooxamide reducing aggregation; that is, particles do not cluster together, causing only minor energy changes in electronic transitions. AgNPs exhibit a broad absorption profile between 400 and 450 nm, attributed to surface plasmon resonance (SPR). SPR arises from the collective oscillation of free electrons on the metal nanoparticle surface in response to incident light waves. The presence of a plasmon resonance peak around 420 nm confirms the formation of AgNPs and their typical optical properties.

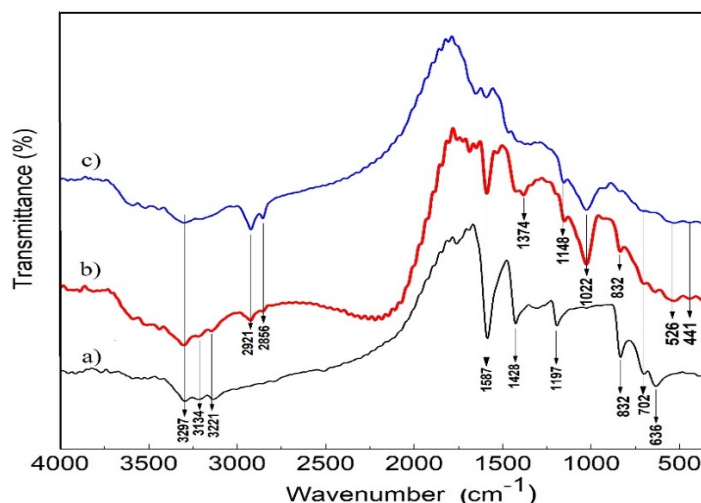


Fig. 3. IR spectra of R (a), Ag–R (b), and Ag–R–CTAB (c) complexes

To investigate the formation of the complex and the interactions between functional groups, the samples were analyzed using infrared (IR) spectroscopy. The spectrum of the dithiooxamide (a) displays a broad band near 3297 cm^{-1} , which is attributed to the stretching vibrations of hydrogen-containing functional groups such as N–H or O–H. Stretching vibrations of C–H groups appear at 2924 cm^{-1} and 2855 cm^{-1} . Intense bands observed at 1587 cm^{-1} and 1428 cm^{-1} correspond to the presence of C=N and C–N bonds, respectively. Significant changes were observed in these bands in the spectrum of the R–AgNPs complex (b). Notably,

the shift and attenuation of the signal at 1587 cm^{-1} indicate coordination between the C=N functional group of the reagent and the silver nanoparticles. Vibrational bands emerging near 1197 cm^{-1} and 1022 cm^{-1} are consistent with C=S stretching and C–S–Ag bonding interactions, respectively, confirming successful coordination between the ligand and metal center. In the spectrum of the dithiooxamide–AgNPs–CTAB complex (c), new bands appear at 832 cm^{-1} , 702 cm^{-1} , and 526 cm^{-1} [19–21]. These signals correspond to vibrational modes resulting from interactions and adsorption of CTAB molecules on the surface of AgNPs. Furthermore, a band

near 636 cm^{-1} is attributed to Ag–S or Ag–N coordination bonds. Overall, the IR spectroscopic analysis confirms the formation of

a complex between the reagent and silver nanoparticles, as well as the stabilization of this complex by CTAB.

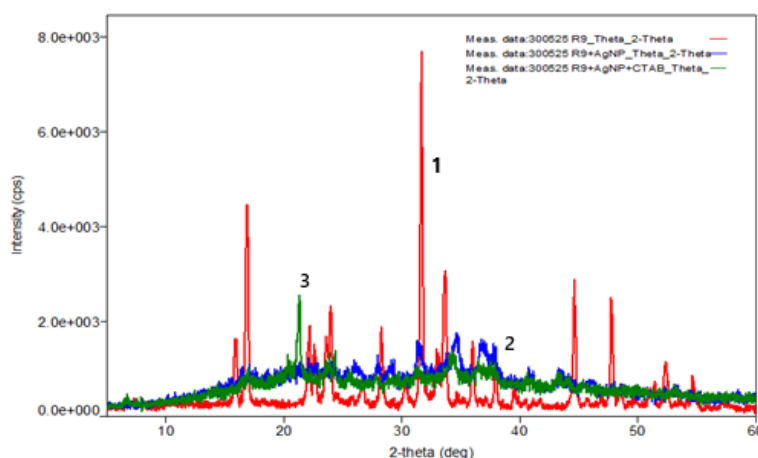


Fig. 4. XRD analysis of R(1), R+Ag (2), and R+Ag+CTAB (3) complexes

The structural integrity and phase composition of the samples were examined through X-ray diffraction analysis. Fig. 4 presents the XRD diffractograms recorded over the 2θ range of 5° to 60° for dithiooxamide ((1) red line), its complex with silver nanoparticles (R+AgNPs, (2) blue line), and the CTAB-stabilized form of this complex (R+AgNPs+CTAB, (3) green line). The XRD analysis of the dithiooxamide reagent (1) revealed multiple sharp and high-intensity peaks, indicative of a pronounced crystalline phase. The main peaks are around $2\theta \approx 15.9^\circ$, 22.1° , 26.7° ,

30.3° , 33.6° , 39.5° , 45.6° , 54.7° , and 57.6° . The crystallite sizes calculated using the Debye–Scherrer equation range from 18.13 nm to 42.58 nm, with an average size of 30.47 nm. These results confirm that the reagent possesses a well-ordered crystalline structure.

In the blue diffractogram (dithiooxamide +AgNPs), some of these peaks decrease in intensity, and new peaks appear near $2\theta \approx 16^\circ$, 23° , 31° , 34° , 37° , 43° , and 53° . The Debye–Scherer analysis revealed crystallite sizes between 1.89 nm and 15.28 nm, with an average size of 8.43 nm.

Table 1. Data of binary R, Ag+R and ternary Ag+R+CTABr complexes estimated by XRD method

R			Ag+R			Ag+R+CTAB		
2θ	β	D, nm	2θ	β	D, nm	2θ	β	D, nm
15.8^0	0.264	30.38	16.8^0	2.3	3.49	16.92^0	0.30	26.77
22.1^0	0.262	30.90	23.4^0	4.3	1.89	21.31^0	0.25	32.33
26.71^0	0.35	23.33	31.37^0	0.5	15.28	23.90^0	0.91	8.92
30.27^0	0.34	24.21	34.4^0	0.73	11.39	34.16^0	0.70	11.87
33.6^0	0.28	28.82	37.0^0	1.72	4.87	36.82^0	1.54	5.44
39.4^0	0.23	36.70	43.4^0	0.70	12.22	43.27^0	1.18	7.24
45.6^0	0.22	39.18	53.3^0	0.9	9.88			66.83
54.6^0	0.21	42.58						
57.5^0	0.5	18.13						

This significant reduction in particle size indicates complexation between the reagent and AgNPs, leading to a more amorphous and dispersed structure. These changes confirm the formation of new phases due to complex

formation between silver nanoparticles and dithiooxamide. After the addition of CTAB, the diffractogram of the resulting sample (dithiooxamide+AgNPs+CTAB, green line) largely preserves the main structural features of

the previous complex. However, some peak intensities decrease and broaden slightly, with peak positions shifting to around 16°, 21°, 23°, 34°, 36°, and 43°. Crystallite sizes for the ternary complex were calculated to range from 5.44 nm to 32.33 nm, with an average size of 15.43 nm. This indicates that CTAB enhances the

dispersion of the complex while also providing a certain degree of structural stabilization. Overall, the XRD results demonstrate that dithiooxamide forms new crystalline phases upon complexation with AgNPs and that CTAB acts as a stabilizer for these structures.

4. Conclusion

In this study, complexes of silver nanoparticles (AgNPs) were synthesized based on dithiooxamide -containing reagents, and their structural and crystallinity characteristics were investigated using various instrumental techniques. UV-Vis spectroscopy revealed the emergence of new absorption bands due to the interaction between the reagent and AgNPs, indicating complex formation, π -electron delocalization, and an increase in electron density within the system. IR spectral analysis confirmed the formation of coordination bonds such as C=N, C=S, and C-S-Ag, while the

presence of CTAB facilitated stabilizing adsorption on the AgNPs surface via Ag-S and Ag-N bonds. XRD results demonstrated that the dithiooxamide reagent possesses a crystalline structure, which upon complexation with AgNPs leads to a reduction in particle size and the formation of amorphous phases. Based on the Debye-Scherrer equation, it was determined that CTAB not only increases the dispersity but also enhances the stability of the complex structure. Such nanocomplexes can be considered promising materials for sensor applications and selective analytical methods.

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