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Biogenic silver nanowires for hybrid silver functionalized benzothiazolilthiomethanol as a novel organic–inorganic nanohybrid

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ABSTRACT

This article involves the synthesis of a new organic compound, benzothiazolilthiomethanol (BTTM) which derived from the reaction of 2-mercaptobenzothiazole with formaldehyde in ethanol. The resulting compound (BTTM) was diagnosed using the following techniques: FTIR, elemental analysis and ^{1}H NMR. The research also included the preparation of silver nanowires depending on using green method. To enhance the idea even further, both as-prepared materials were spatially used to prepare new nanocomposite of the type organic–inorganic nanohybrid where silver was used to form the core while the organic matter was the shell. The resulting organic–inorganic nanohybrid was characterized using FTIR, XRD, EDX, SEM and TEM. The results of characterization techniques prove that a stable and sufficient amount of BTTM can be attached to the metal surface.

1. Introduction

A heterocyclic compound is a cyclic compound that has at least two different elements as members of its ring(s) [1–4]. They are very widely distributed in nature and are essential to life in various ways [5]. Most derivatives of these compounds and their complexes are used in the industrial and medical field due to their industry and pharmacological efficacies [6–9].

Benzothiazole is a heterocyclic compound with a weak base effect, which contains in its composition sulfur and nitrogen atoms as well as carbon and hydrogen atoms, and consists of a thiazole ring fused with a benzene ring at the 5, 4 sites [10]. This molecule having various biological activities and still of great scientific interest now a day. Benzothiazole compounds and their derivatives have been used in pharmaceutical and agricultural chemistry [11]. Eventually, they were found to show pharmacological activities in the fields of antitumor [12,13], anticonvulsant [14], antimicrobial [15], anthelmintic [16], antileishmanial [17], antitubercular [18], schictosomicidal [19], antibacterial and antifungal [20,21], anti-inflammatory [22–24] antipsychotic [25] and antidiabetic activities [26].

Due to benzothiazole substitutes played an important role in the field of pharmacological chemistry [27], our aim in this research was to prepare a new derivative of benzothiazole that could have a biological effect by introducing a functional group on the 2-mercaptobenzaziazole. But the excessive use of antibiotics led to the resistance of some germs to these antibiotics, as well as these antigens may have a negative effect on the cells of the body. For these reasons, silver nanoparticles were used as a nanoparticle carrier of the organic compound benzothiazolilthiomethanol. The silver nanowires are prepared using the aqueous extract of the sidr leaves which act as a reducing agent. As silver nanoparticles were used to resist diseases [28,29], as antibacterial [30,31] and to treat wounds [32] because these nanoparticles are safer and do not possess toxicity to human and animal cells [33,34]. There are several methods for preparing nanoparticles, including chemical and physical methods, as well as thermal evaporation [35]. Researchers are studying the safest way to pre-

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pare these nanoparticles. Where the researchers used many plant materials in preparing the silver nanoparticles, including aloe vera [36] and sidr leaves [37], because extracts of plant leaves and seeds are safer, as the researchers stressed through their studies that the use of aqueous extract of plant leaves in preparing silver and gold particles may be Environmentally friendly [38]. This work aims to prepare a novel organic compound (benzothiazolilthiomethanol) which will use to make a novel organic–inorganic nanohybrid with green-synthesized nanosilver. Furthermore, our method does facilitate convenient use of nanomaterials by only minor modifications.

2. Experimental

2.1. Materials

2-mercaptobenzothiazole ($C_7H_5NS_2$, 98%), formaldehyde (CH_2O, 37.00%), silver nitrate ($AgNO_3$, 99.99%) and all solvents were supplied from Merck-Turkey.

2.2. Instrumentations

FTIR spectra of the prepared BTTM, silver nanowires and the organic–inorganic nanohybrid were collected in Shimadzu 800S. Elemental analysis was conducted on CHN-Leco-Truspec. ^{1}H NMR spectrum of BTTM were measured on Varian Unity spectrometer using DMSO d_6 solvent. XRD analysis of Ag nanowires and Ag-BTTM organic–inorganic nanohybrid were measured on Shemadzu-XR-6000. SEM and EDS of nano silver and its nano derivative were conducted using FESEM-EDAX Type Oxford instruments FESEM Tech.). Furthermore, TEM of organic–inorganic nanohybrid was conducted using JEOL 2010.

2.3. Methods

2.3.1. Synthesis of silver nanowires (Ag NWs)

Silver nanowires were synthesized using water extract of *zizyphus Spina Christi L* leaves as a green reducing agent by weighing 25 g of fresh sidr leaves after washing with distilled water. Thereafter, sidr leaves were mixed with 100 ml distilled water and cut into very small pieces by an electric mixer. The resulting mixture was filtered to obtain a yellowish-green solution. In a separate vessel, 0.22 g of silver nitrate was dissolved in 400 ml distilled water and heated to a temperature of 50–60 °C. The last step includes mixing of the silver nitrate solution with the aqueous extract solution. The addition of the extract solution was very slowly (5 drops per minute) under vigorous stirring at 50–60 °C. After the two addition cycles, the solution was turned to pale yellow then yellow and red which indicates the successful formation of silver nanoparticles.

2.3.2. Synthesis of benzothiazolilthiomethanol (BTTM)

To a solution of 2-mercaptobenzothiazole (0.0059 mol, 1 g) in ethanol (40 ml), formaldehyde (5 ml) was added. The resulting mixture was mixed for 5 min at room temperature before refluxing for 5 h on a steam bath. Thereafter, the mixture was left to evaporate at room temperature to half of its initial volume, cooled, filtrated and washed with ethanol.

White solid. Yield: (77%). Anal. calc. for $C_8H_7NOS_2$: C, 48.71; H, 3.58; N, 7.10, Found: C, 49.01; H, 3.78; N, 7.25%. Molar conductivity in DMSO (1.8 Ω^{-1} cm^{-1} mol^{-1}). FTIR (KBr, cm^{-1}): 3388 broad m ν (O–H), 3054w, ν(=C–H), 2933 m, 2898 m, ν(–C–H), 1600vs, ν(C=N), 1559vs, 1484 s, ν(C=C), 1355 ν(C–O–H), 612 m ν(C-S). ^{1}H NMR (400 MHz, δ ppm, J Hz, DMSO d_6) δ = 7.80 (d, J^3 = 7.8, 1H), 7.74 (d, J^3 = 7.9, 1H), 7.50 (td, J^3 = 7.9, J^4 = 4.2, 1H), 7.38 (td, J^3 = 7.9, J^4 = 4.2, 1H), 5.09 (broad s, 1H), 4.39 (s, 2H). Melting point: 98–100 °C.

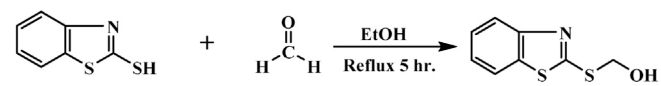
2.3.3. Synthesis of nanohybrid (Ag NWs-BTTM)

A solution containing 1 g of BTTM in 100 ml hot ethanol was added to a fresh-prepared solution of nanosilver (0.22 g of silver nitrate in 400 ml distilled water). The resulting mixture was mixed, sonicated by sonication device for 1 h and then refluxed for 1 h. The resultant mixture was centrifuged at 6000 rpm to separate the yellow precipitate of Ag NWs-BTTM. The precipitate of Ag NWs-BTTM was re-dispersed in hot ethanol to remove unreacted BTTM and centrifuged to take Ag NWs-BTTM as a pure product.

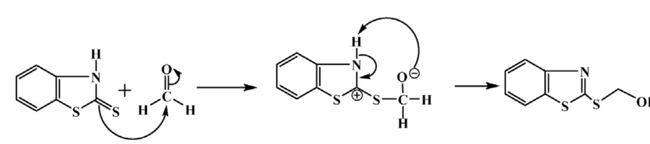
3. Results and discussion

3.1. Synthesis and characterization of benzothiazolilthiomethanol (BTTM)

BTTM was prepared by the reaction of 2-mercaptobenzothiazole with formaldehyde in the presence of ethanol as a solvent, as in the following equation:



Equation 1: Reaction equation for the synthesis of BTTM.
This reaction occurs depending on the mechanism (1)



Mechanism 1: The suggested mechanism for the synthesis of BTTM.

The mechanism concludes (i) a nucleophilic attack of C=S bond on the carbon atom of carbonyl group of formaldehyde molecule and this resulted in (ii) change of hybridization of the carbon of carbonyl group from sp^2 to sp^3 to form unstable intermediate which suffer from an intermolecular reaction to give BTTM, as shown above. The evidences for the success of BTTM formation were came from the following techniques: FTIR, ^{1}H NMR and CHN. Moreover, melting point was giving a good confirmation for the formation of a different material, as it shows a difference in melting point between starting material and the produced one (BTTM). Further evidence is coming from FTIR, as the spectrum of BTTM showed new bands at 3388 and 2898 cm^{-1} which attributed to the stretching vibrations of OH and CH_2 groups, respectively, proving the formation of BTTM through the suggested mechanism. ^{1}H NMR is the most powerful technique for the characterization of organic molecules, therefore, it was used to diagnose the afforded molecule. The spectrum shows the aromatic protons signals in the range 7.82–7.34 ppm as two doublets, triplet of doublet and triplet signals, ^{1}H NMR spectrum (Fig. 1) shows broad singlet and singlet signals at 5.09 and 4.39 ppm. These signals are due to the presence of OH and CH_2 protons, respectively, indicating that BTTM was successfully synthesized. Finally, elemental analysis gives good effort for the formation of BTTM, as it

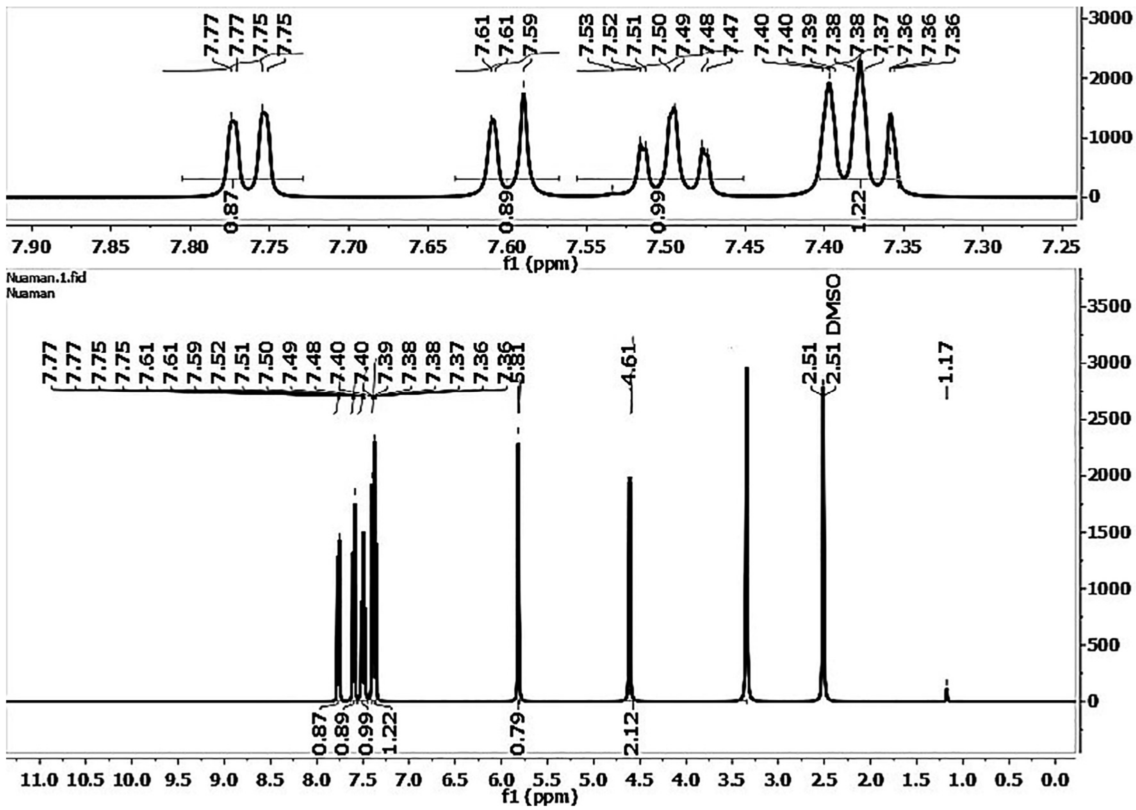


Fig. 1. ^{1}H NMR of BTTM.

shows a good association between theoretical values of C, H and N elements and calculated one.

3.2. Characterization of Ag nanowires (Ag NWs)

Leaves extract of *zizyphus spina christi L* was used extensively as a reducing agent to convert silver ions into nanoscale silver [37]. However, using this extract, nanosilver produces a different and distinct nanostructure. Although leaves sidr extract was used extensively in the synthesis of nanoscale silver, it has not been diagnosed strongly (no SEM and/or TEM), except in a few studies [39]. The first study involved the formation of nanostructures with irregular sizes and shapes. Whereas, our previous study involved the formation of silver with two distinct morphologies (nanohexagones and nanosquares) [37]. In this study, different nanoscale forms are formed (silver nanowires).

3.2.1. Electronic spectrum and FTIR

The electronic spectrum is a very effective diagnostic technique for the characterization of silver nanoparticles because of silver nanostructures possesses a distinct band at approximately 400 nm which is attributed to the electron cloud around silver metal (surface plasmon resonance) [40–44]. UV–Vis spectrum of the prepared silver (Fig. 2) showed a broad band at a λ max. of 405 nm, demonstrated the successful formation of nano silver [40–44].

In the case of FTIR, the spectrum (Fig. 3) shows many peaks at 3431, 1645, 1425, 1030, 628, 584, 547 and 451 cm^{1-} which attributed to ν(O–H), ν(C=C)/ deformation of OH, ν(C–O–C) which attributed to the trace amount of phenols and terpenoids in the extract while the other peaks can be attributed to the substituted

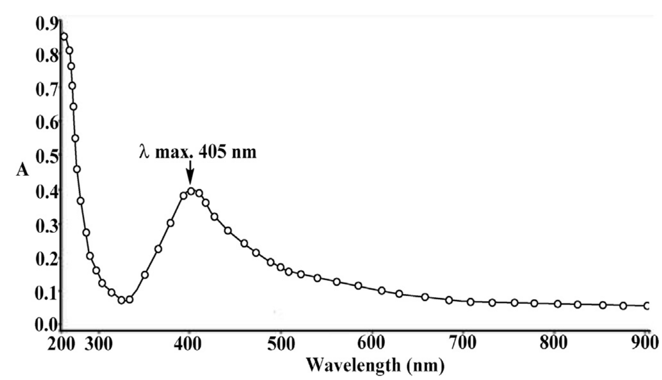


Fig. 2. UV–Vis of Ag nanowires.

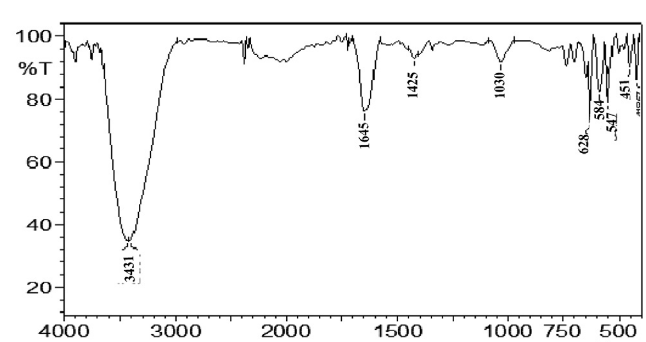


Fig. 3. FTIR of Ag nanowires.

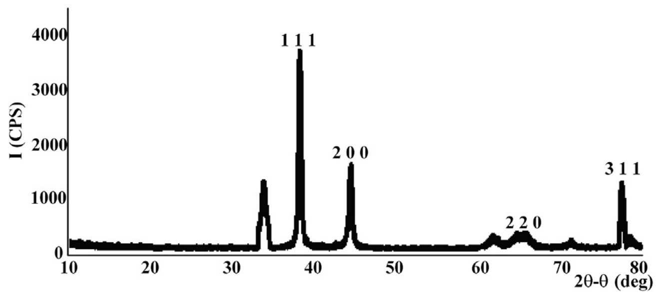


Fig. 4. Powder XRD of Ag nanoparticles.

aromatic ring in the extract of sidr, respectively [40–42]. To be more clear, this technique cannot be considered a feasible technique in the characterization of nanosilver, as insufficient IR energy to make the metallic bonds susceptible to vibration. Therefore, it only gives information about organic compounds capped with silver or adsorbed water not about nanosilver.

3.2.2. XRd

Fig. 4 represents the XRD pattern of as-prepared Ag nanowires. The pattern clearly shows only four major peaks of face centered cubic silver at (2θ) 38.25° for (1 1 1), 44.43° for (2 0 0), 64.58° for (2 2 0) and 77.52° for (3 1 1). The pattern shows another peaks at 33.78°, 62.14° and 70.64° proving the presence of a capping agent onto the surface of silver nanowires.

3.2.3. SEM, TEM and EDX

SEM measurement of the prepared nanosilver showed the presence of regular wire nanostructures with diameters ranging from 77 to 80 nm with no other structures as shown in Fig. 5a. TEM image shows that a clear nanowire was formed with a dimeter of 80 nm Fig. 5b.

Debye-Scherrer equation was not fit to determine the average crystallite diameter of Ag nanowires, as this equation can be gave an accurate average crystalline size in the case of spherical particles. Therefore, the calculated crystal size was 35 nm while it was 77–80 nm in diameter according to SEM. Finally, the longitudinal growth of silver to form its nanowire morphology is usually attributed to the mechanism of strain restriction onto decahedron nucleation [45].

EDX was also conducted for as-prepared silver nanowires to determine if there are pure silver nanoparticles or a combination of silver and silver oxide and however, XRD also showed no peaks for the silver oxide. The measurement displayed a high intense signal of silver at 2.98 KeV which proves the high purity of the resulted nanosilver as shown in Fig. 5c. Furthermore, the signals of carbon, iron and sodium can be attributed to the absorbed organic molecules from the extract or from the sampling process in SEM sample holder.

3.3. Characterization of silver nanowires-benzothiazolilthiomethanol (Ag NWs-BTTM)

3.3.1. Electronic and FTIR spectra

The UV–Vis spectrum of Ag NWs-BTTM (Fig. 6) revealed two major broad bands at 311 and 419 nm, attributable to pi-pi of

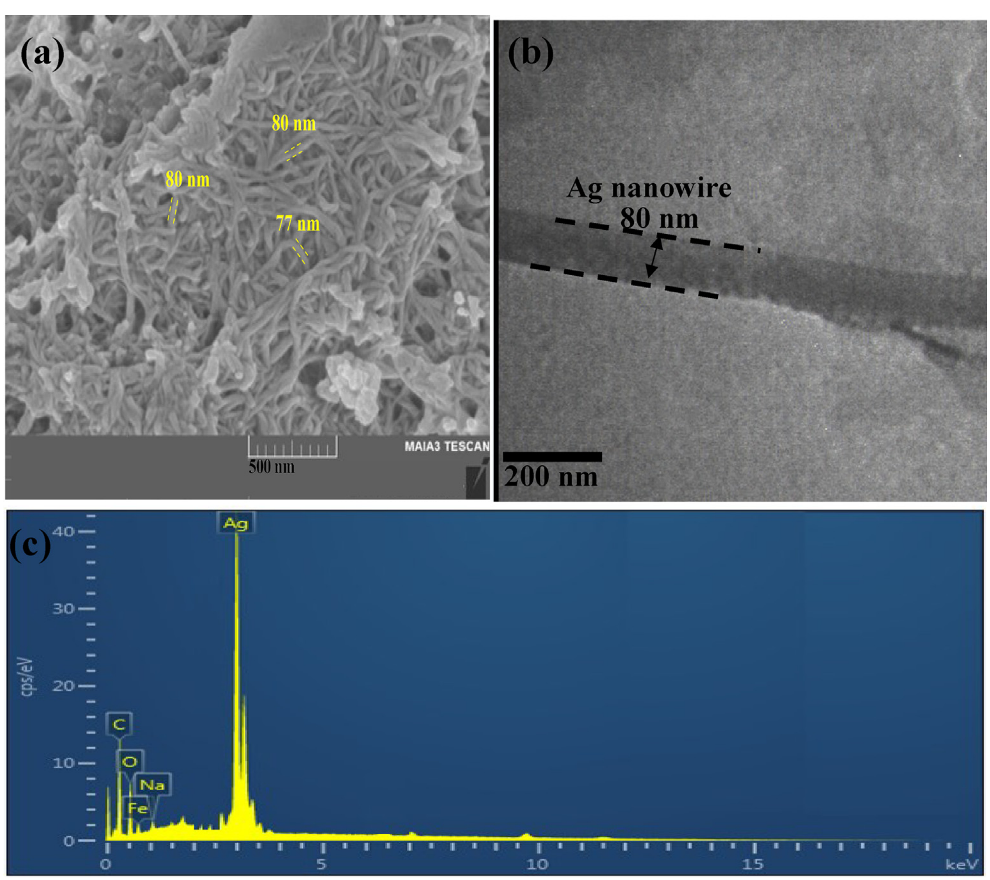


Fig. 5. (a) SEM, (b) TEM and (c) EDX of Ag nanowires.

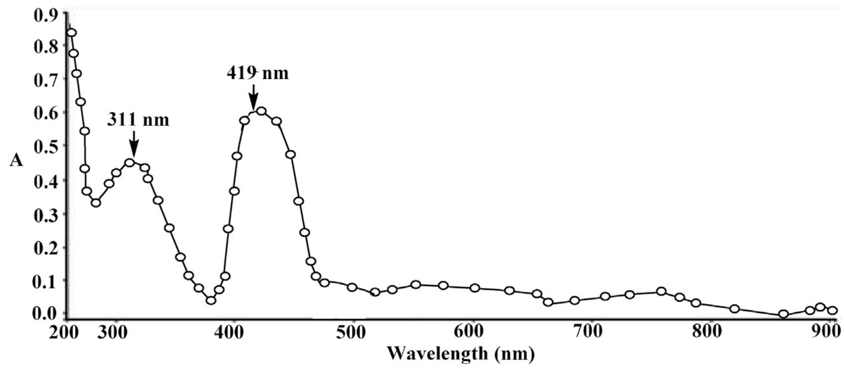


Fig. 6. UV–Vis of Ag NWs-BTTM.

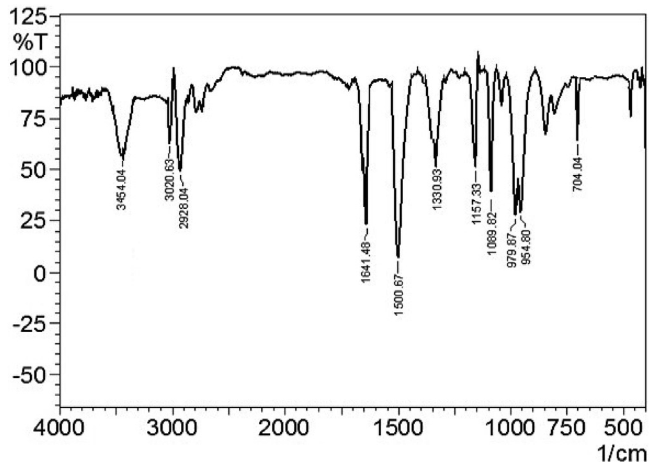


Fig. 7. FTIR of Ag NWs-BTTM.

BTTM and SPR of Ag nanowires, respectively. The shift of silver band as red shift compared to pure silver, evidence of the success of the reaction between silver and BTTM.

The FTIR spectrum of Ag NWs-BTTM (Fig. 7) showed the following characteristic peaks at 3454, 3020, 2928, 1641 and 704 cm^{1-}, attributed to (O–H), (=C–H), (–C–H), (C=C) and (C–S) of BTTM, respectively. The presence of these peaks directly indicates the success of the reaction and the association of the organic compound with nanosilver through weak forces.

3.3.2. XRd

Fig. 8 represents the XRD pattern of as-prepared Ag NWs-BTTM. The pattern seems like amorphous material. Even though less crystallinity of this material, the pattern showed two main peaks for nanosilver at (2θ) 38.00° for (1 1 1), 44.39° for (2 0 0). Additionally, the pattern showed two other peaks at 16.85° and 50.00° which attributed to the organic molecule (BTTM) anchored onto the surface of nanosilver. The disappearing of nanosilver peaks at the planes 2 2 0 and 311 proves that BTT is linked tightly to 1 1 1 and 2 0 0 planes.

3.3.3. SEM, TEM and EDX

The SEM measurement of the capped silver (Fig. 9a) showed an irregular morphology, as it was of a bundle tube-like agglomerated structure and covered with another substance, which is the BTTM. When comparing the SEM measurements for the non-functionalized and functionalized silver, we can clearly notice the success of the reaction, as it gave two completely different structures while maintaining the tubular structure but in a lumpy manner. The TEM measurement (Fig. 9b) showed a very clear evidence for the presence of silver nanowire of 80 nm in diameter which covered with BTTM which was about 37 nm thick. The nanosilver morphology was also demonstrated by XRD, as it showed the presence of the planes at 1 1 1 and 2 0 0. The disappearance of the other planes clearly indicates a change in the morphology after the addition of the organic matter. EDX was also conducted for Ag NWs-BTTM (Fig. 9c) and the measurement displayed two high intense signal for silver at $K\alpha = 2.980$ KeV and sulfur at $K\alpha = 2.307$ KeV. The presence of sulfur signal fully proves the existence of BTTM in the prepared organic–inorganic nanohybrid.

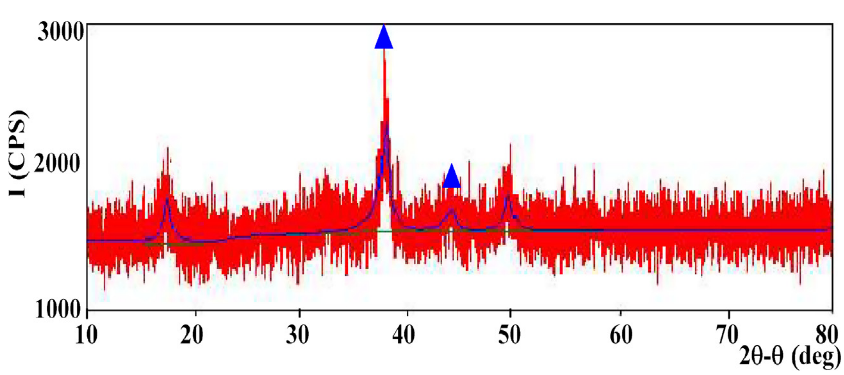


Fig. 8. XRD of Ag NWs-BTTM.

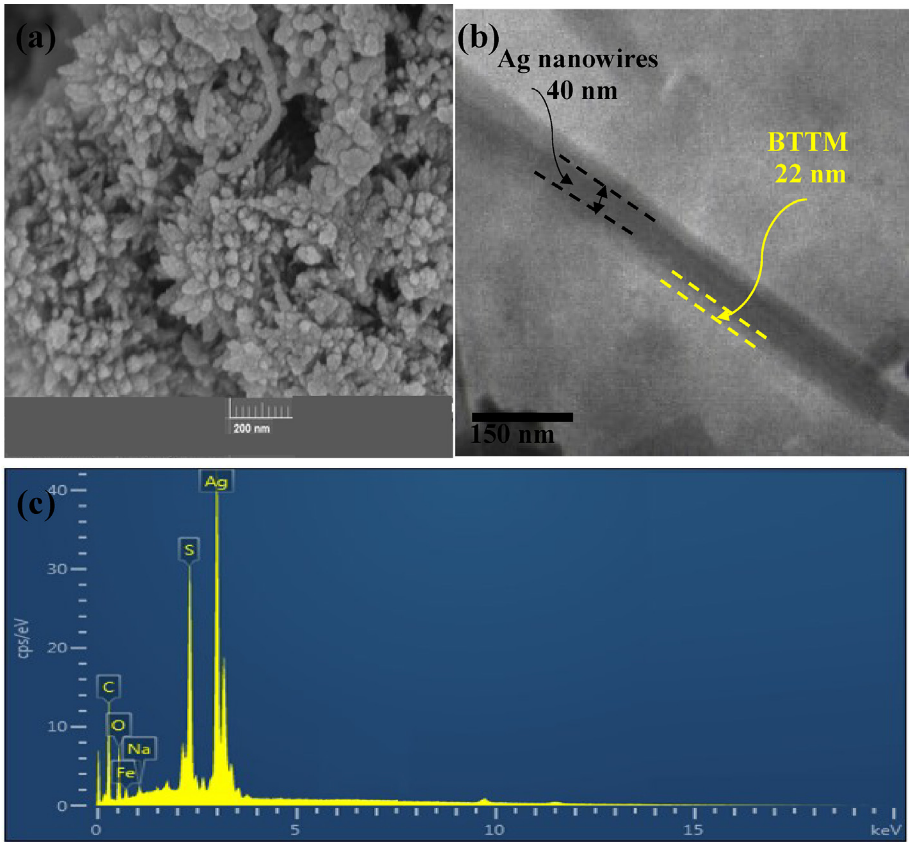


Fig. 9. (a) SEM, (b) TEM and (c) EDX of Ag NWs-BTTM.

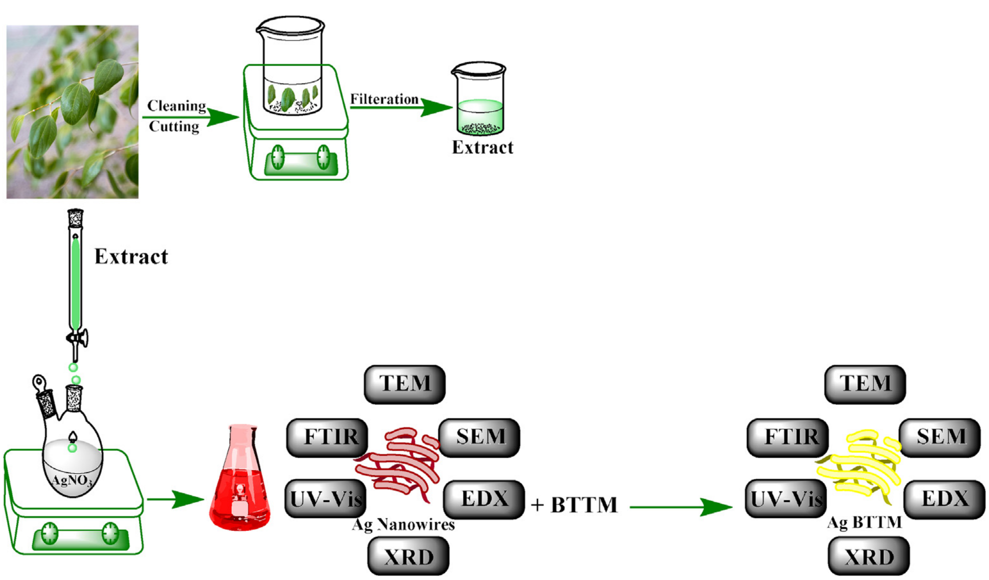


Fig. 10. Illustration diagram for the synthesis of Ag NWs-BTTM nanohybrid.

4. Conclusion

The novelty of this work is concerned about the synthesis of new silver nanostrutures (nano wires) using *zizyphus spina christi L* leaves extract as a green reducing agent. The results show that a pure nanosilver can be produced with a diameter of 77–80 nm with long length. Additionally, this work concludes the synthesis of new organic molecule (BTTM) which the characterization techniques prove its purity and the suggested mechanism. The resulted nanowires were successfully coated by with BTTM by a simple reaction to form Ag NWs-BTTM as in Fig. 10. We suggest that organic substrate (BTTM) was attached to the Ag nanowire by the interaction between the electron pairs of sulfur and/or oxygen atoms and silver nanowires. TEM measurement proved this type of interaction through the presence of uniform interaction of BTTM along the nanosilver, which does not strongly support the interaction with weak forces. This new organic–inorganic nanohybrid is now under consideration to examine its bioactivity.

CRediT authorship contribution statement

Nuaman F. Alheety: Supervision, Conceptualization, Investigation, Visualization. **Maysoon A. Hamad:** Supervision, Conceptualization, Investigation, Visualization. **Abdullah Z. Kalif:** Supervision, Conceptualization, Investigation, Visualization. **Mustafa A. Alheety:** Data curation, Methodology, Investigation, Writing - review & editing. **Modher Y. Mohammed:** Data curation, Methodology, Investigation, Writing - review & editing. **Hüseyin Akbaş:** Data curation, Methodology, Investigation, Writing - review & editing.

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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