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PREPARATION OF GOLD NANOPARTICLES USING CHEMICAL METHODS

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Abstract:

Nanoparticle technology is witnessing increasing interest due to its broad applications in medicine, pharmacology, the environment, and energy. Gold nanoparticles (AuNPs) are among the most studied nanomaterials due to their unique properties, such as nanoscale size, distinctive optical properties, high chemical reactivity, and good stability. This study aims to synthesize gold nanoparticles using a chemical method and compare the results in terms of physical and chemical properties.

In the chemical method, gold chloride ($HAuCl_4$) was used as a source of gold ions, while a strong reducing agent, sodium borohydride ($NaBH_4$), was used to induce rapid reduction and form the nanoparticles.

The formation of the nanoparticles was observed by a color change to maroon, indicating the surface plasmon resonance (SPR) phenomenon characteristic of gold nanoparticles. Multiple techniques were used to characterize the particles, such as X-ray diffraction (XRD) to determine the crystalline phase, scanning electron microscopy (FESEM) to study shape and size, energy dispersive spectroscopy (EDX) to identify elements, and four-transform infrared spectroscopy (FTIR) to analyze the stabilizing groups. The results demonstrated the successful preparation of stable nanoparticles.

Keywords: Gundelia, phenolic compounds, surface plasmon phenomenon, crystalline phase, sodium borohydride.

Introduction

1. Introduction

The rapid development of science and technology in the world has greatly contributed to making life easier and more comfortable. However, this has not provided adequate protection and care for humans, as they are increasingly exposed to numerous fungal and bacterial infections, as well as chronic and serious diseases such as cancer, high blood pressure, diabetes, typhoid, malaria, avian influenza, swine flu, dengue fever, and others. Therefore, it has become imperative to work to provide antibacterial and anticancer treatments that are accessible to all by making them less expensive and less risky to use [1].

2. Basic Concepts of Nanomaterials

Nanotechnology was previously defined as the process of designing and manufacturing materials, devices, and systems that are controlled at the nanoscale (1-100 nanometers). However, this definition has been modified to now include the observation and study of material phenomena at the nanoscale, as well as the methods used to manipulate materials at this scale. Many material properties at the nanoscale differ from those at the macroscopic scale [2]. For example, metals at this scale have lower melting points and different lattice constants. Crystal structures also become stable at lower temperatures in the nanoscale. Interestingly, three-dimensional (macroscopic) gold does not exhibit catalytic properties, while gold nanocrystals exhibit remarkable catalytic activity at low temperatures. In general, nanoparticles, whether in the form of crystals or rods, are considered nanomaterials if one of their dimensions (usually diameter) is between 1–100 nm [2]. At the nanoscale, the surface area-to-volume ratio is very large, resulting in unique properties that differ from those seen at the microscale. This enables nanomaterials to be used in a wide range of applications [3]. This technology is sometimes known as "convergent technology" because it involves the fabrication of devices and systems using nanometer-scale materials and their application in diverse fields such as chemistry, physics, biology, materials science, engineering, and medicine. It is also called "enabling technology" because of its role in achieving scientific and technological advancements in multiple fields [3].

Recently, the importance of this technology has increased due to the novel properties exhibited by nanomaterials, such as structural, optical, and mechanical properties, which differ significantly from their properties at the larger scale [4]. In three-dimensional metals, the conduction and valence bands overlap, while a gap between these bands appears in metallic nanoparticles. The energy gap in metallic nanoparticles can be similar to that observed in semiconductors or even insulators [5].

3. Method:

Nanoparticles can be manufactured in a variety of shapes and structures, with their size, shape, and structural composition being key factors in determining their broad applications in fields such as catalysis, medicine, gas sensing, electronics, and others. The properties of manufactured nanomaterials depend on several factors, including the preparation method, the starting materials used, the pressure, temperature, and annealing time [6, 7]. Many methods have been developed for manufacturing nanomaterials, which are generally classified into two main approaches: top-down and bottom-up. The top-down approach involves breaking bulk materials into nanoparticles of a few nanometers in size using mechanical methods such as milling, while the bottom-up approach relies on assembling atoms or molecules to form nanoparticles via chemical methods such as sol-gel processing, hydrothermal treatment, reverse micelle, and co-precipitation. As for gold nanoparticles, they can be manufactured using the bottom-up approach by reducing gold ions to their nanoscale state. This process involves reducing gold salts in the presence of stabilizing agents that prevent particle agglomeration. Gold nanoparticles can be synthesized using three main methods: physical, chemical, and biological [7, 8].

3.1 Chemical Methods

In this method, nanoparticles are produced in an aqueous medium using a reducing agent. The most common reducing agents used in this process are citrate and sodium borohydride. Among the traditional chemical methods for synthesizing colloidal gold, the Turkevitch method is the most widely used, as it is simple and easy to achieve the desired size and stability of colloidal nanoparticles. In 1951, Turkevitch modified the Hauser method for synthesizing nanoparticles, using sodium citrate as the reducing agent. This results in the production of spherical gold nanoparticles with a narrow size distribution. In 1994, the Brust-Schiffrin method was developed, which relies on thiol-gold interactions to protect the nanoparticles with thiol ligands. In this method, AuCl4- is reduced by sodium borohydride in the presence of an alkanethiol. The reaction proceeds in two stages (water-toluene), producing nanoparticles with diameters ranging from 1 to 3 nm.[6] Gold nanoparticles were synthesized chemically using sodium borohydride (NaBH4) as a stabilizing agent. 0.5 mL of gold chloride was added to a conical flask containing 100 mL of double-distilled water, and the resulting solution was stirred for 1 hour at room temperature. Next, 40 mL of NaBH4 was dissolved in 30 mL of ice-cold double-distilled water, and the resulting solution was gradually added (drop by drop) to the gold chloride solution, while continuing to stir for 30 minutes.We then observed that the solution turned red, indicating the formation of gold nanoparticles [6].

4. Results and Discussion

Gold nanoparticles were synthesized chemically using sodium borohydride (NaBH4) as a stabilizing agent. 0.5 ml of gold chloride was added to a conical flask containing 100 ml of double-distilled water, and the resulting solution was stirred for 1 hour at room temperature. Next, 40 ml of NaBH4 was dissolved in 30 ml of ice-cold double-distilled water, and the resulting solution was gradually added (drop by drop) to the gold chloride solution, while continuing to stir for 30 minutes. We then observed that the solution turned violet, indicating the formation of gold nanoparticles [7].

The successful preparation of gold nanoparticles (Au NPs) was confirmed primarily by the observed color change from yellow to violet and dark red by the chemical method. The composition of the gold nanoparticles was also verified using UV-Vis spectroscopy. Figure 1 shows the characteristic surface plasmon resonance band of Au NPs, which appears at a wavelength of 527 nm (λ max).

The diameters of the gold nanoparticles range from 26 to 44 nm, which is much smaller than the incident wavelength. Therefore, the scatterings of these gold nanoparticles are known as Rayleigh scattering, because elastic scattering in which the incident wavelength is equal to the scattering wavelength is called Rayleigh scattering [7].

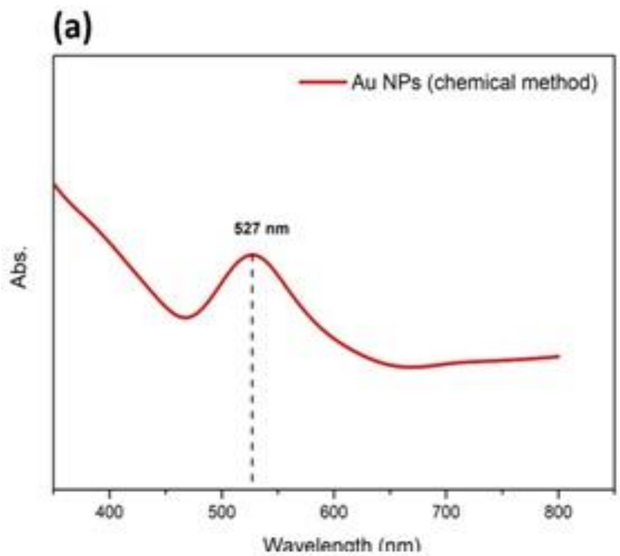

Figure (1) UV-visible absorption spectrum of gold nanoparticles.

The band gap energy was successfully determined by plotting $(\alpha h\nu)^2$ versus $h\nu$, where α represents the optical absorption coefficient and $h\nu$ represents the photon energy. Absorption spectra of gold nanoparticle (Au NPs) samples grown during growth. This sample exhibits absorption bands at wavelengths around 527 nm, reflecting the characteristic surface plasmon resonance of gold nanoparticles [8]. This absorption results from the collective oscillations of free electrons within the particles when exposed to light, resulting in distinct optical properties in the visible spectrum and the color change from yellow to red and violet for the chemical method. According to previous studies, the location of the absorption peak depends on the particle size and the surrounding environment.

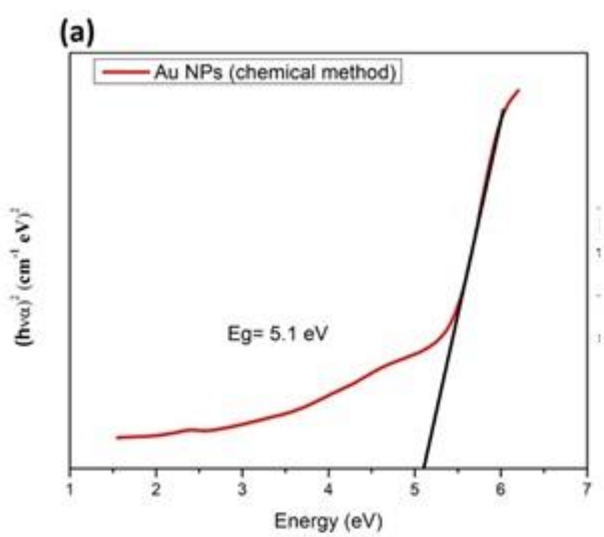

Figure (2) Bandgap of Gold Nanoparticles

The Ttau'c diagram for gold nanoparticles (Au NPs) was calculated by plotting $(\alpha h\nu)^2$ versus photon energy ($h\nu$). As shown in Figure 2, the band gap value was determined to be 5.1 eV for gold particles prepared by the chemical method [9]. A slight decrease in the band gap value was observed, as mentioned in the section above.

The band gap energy was successfully determined by plotting $(\alpha h\nu)^2$ versus $h\nu$, where α represents the optical absorption coefficient and $h\nu$ represents the photon energy. Absorption spectra of the gold nanoparticle (Au NPs) samples grown during growth. This sample exhibits absorption bands at wavelengths around 527 nm, reflecting the characteristic surface plasmon resonance of gold nanoparticles. This absorption results from the collective oscillations of free electrons within the particles when exposed to light, resulting in distinct optical properties in the visible spectrum and causing the color change from yellow to red and violet due to the chemical process [9]. According to previous studies, the location of the absorption peak depends on the particle size and the surrounding environment.

The morphology of the prepared material was analyzed using a high-resolution scanning electron microscope (FE-SEM). Figure 3 illustrates the general shape and size of the particles, which are assumed to have a geometrically variable texture due to the chemical process.

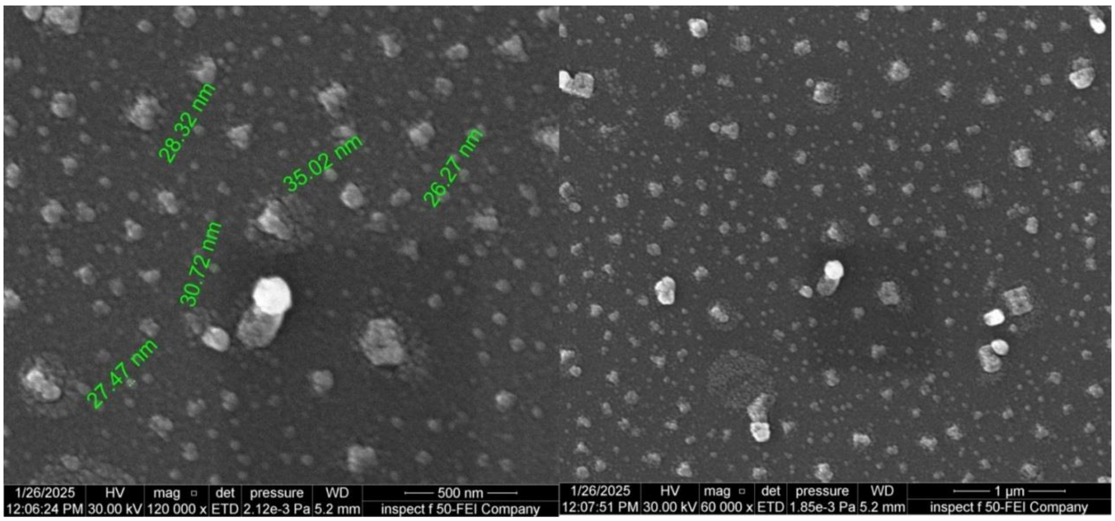

Figure (3) Scanning electron microscope (SEM) image of gold nanoparticles synthesized by chemical methods.

EDX testing revealed clear peaks in the gold band, confirming the successful formation of gold nanoparticles, as shown in Figure(4). Other peaks, such as oxygen, carbon, potassium, sodium, etc., are due to the phytochemical components of the aqueous extract of Gundelia. It is worth noting that the metallic gold nanocrystals exhibited a light absorption peak at approximately 2.3 keV, but at a low intensity. This is due to the dominance of extract components in the low-concentration samples used for analysis [10].

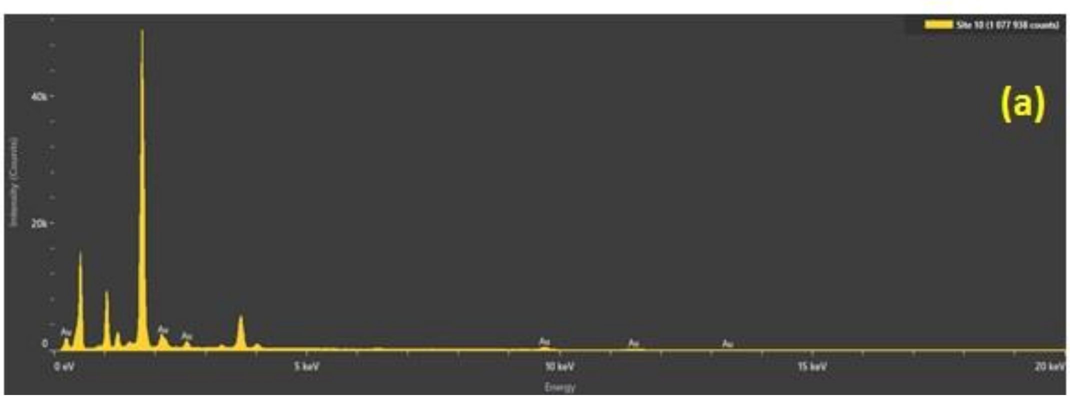

Figure (4) EDX pattern of gold nanoparticles prepared by chemical and green methods.

Fourier transform infrared (FTIR) measurements were performed to identify the primary and secondary groups in the gold nanoparticles and the plant extract, which contributed to the reduction of the gold particles, as well as their effective encapsulation and stabilization. Figure (5) shows the spectra resulting from FTIR analysis, which helps identify the functional groups associated with this process [11].

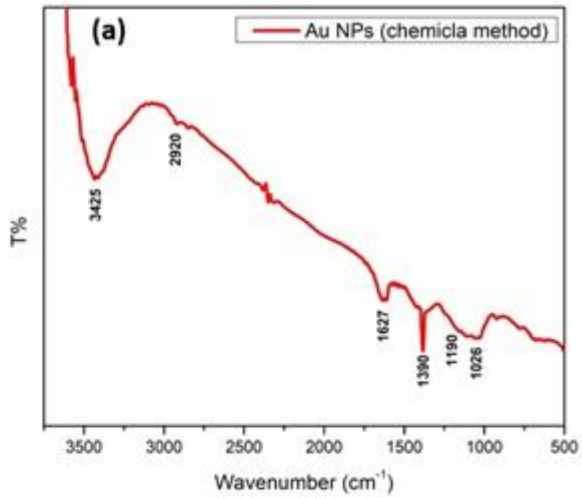

Figure (5) FTIR spectrum of gold nanoparticles prepared by chemical methods.

Figure 5(a) shows the FTIR spectrum of gold nanoparticles prepared by chemical methods. The -OH group stretch was observed at 3425 cm-1, the C-H group at 2920 cm-1, the aromatic C=C group at 1627 cm-1, the aromatic -C-O ring at 1390 cm-1, and the C-O-C group at 1026 cm-1. These results are consistent with previous studies [11-13].

The crystalline nature of the gold nanoparticles was confirmed by X-ray diffraction (XRD) analysis. Figure 6 shows the X-ray diffraction pattern of gold nanoparticles obtained by chemical methods.

The diffraction peaks appearing at 38.2°, 44.8° and 65° correspond to the (1 1 1), (2 0 0) and (2 2 0) [15] faces of the face-centered cubic crystal structure, respectively. The peak corresponding to the (1 1 1) plane is more intense than the other planes. [14,15].

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