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The Use of Silver Nanoparticles Prepared by The Green Method to Reduce The Concentration of Nickel in Water

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Abstract

The present work aims to study a convenient and environmentally friendly method for the green synthesis of silver nanoparticles (AgNPs based on lemon juice extract. The biomolecules contained in lemon extract act as self-reducing and stabilizing agents. The optimal solution for the synthesis of AgNPs was determined by varying silver nitrate concentrations. A surface plasmon resonance system identified the synthesized silver nanoparticles by their color change from colorless to dark brown. Silver nanoparticles synthesized were characterized by Uv-visible (UV-Vis) spectroscopy, high-resolution transmission electron microscopy (HR-TEM), and FTIR spectroscopy. As a result of UV-visible spectroscopy, surface plasmon resonance (SPR) can be determined. There are peaks in the samples at (432, 452, and 448 nm). HR-TEM analysis revealed spherical, oval, hexagonal, and elliptical particle morphology, and particle sizes were 31 nm, 15 nm, and 28 nm for spherical shapes. As a result of FTIR analysis, biomolecules were found in the lemon extracts and on the surface of the AgNPs. The current study results may assist in developing an environmental application. This is a means of minimizing the presence of heavy metals in water, such as nickel. Nickel concentration was measured before and after the use of nanoparticles, and the nickel concentration decreased after passing through the filter containing silver nanoparticles.

Keywords: Biosynthesis; silver nanoparticles; lemon extract; UV-vis; HR-TEM; FTIR

Introduction

Throughout the past few years, there has been an increase in interest in nanotechnology, which involves the development of new materials and tools at the nanoscale (1-100 nm) [1, 2]. Nanomaterials have attracted considerable attention due to their unique physical and chemical properties, which do not found in bulk materials [3]. There are several physical, chemical, and biological properties of this material, including optical, thermal, electrical conductivity, and antimicrobial [4]. Nanostructured materials, such as silver nanoparticles, are widely used in many applications because of their unique optical properties, relatively high stability, and ability to couple with biomolecules [3, 5]. The optical properties of metallic nanoparticles (MNPs) can be changed in order to produce surface plasmonic bands (SPR) from the visible to the infrared region [6]. It is important to note that SPR occurs when metal nanoparticles interact strongly with light, which is caused by the collective oscillation of free electrons on the metal surface when excited by a particular wavelength of light [7]. Noble MNPs exhibit different optical properties depending on their geometry, dielectric materials, size, composition, ambient medium, and separation distance between particles. A difference in the ambient medium, a different size distribution, and a different shape caused the color of the MNPs to change [6]. Several methods for preparing nanoparticles have been developed; these include physical and chemical methods, microemulsion, electrochemical, polyol processes, hydrothermal hydrolysis, pyrolysis, electrothermal reduction, chemical reduction, microwave-assisted process, etc. [3, 8-10]. In spite of chemical techniques having some advantages and also being effective in the manufacture of MNPs, they each have their own downsides, including the use of toxic or dangerous reducing agents, high cost, and exhausting energy and time requirements [3, 7, 11]. Alternatively, green synthesis has more positive aspects, such as a lower, lower temperature, a shorter reaction time, and manufacturing methods that are non-hazardous and environmentally friendly manufacturing. In recent research, it has been shown that the use of plant extracts can act as both a reducing and stabilizing agent; as a result, it has been extensively investigated [7, 8, 11-13]. Silver nanoparticles (AgNPs) are used in a variety of applications and are prepared from a variety of plants, including roots, seeds, leaves, stems, and flowers. For clarification, Annona muricata peel extract [8], Melon (Citrullus lanatus) [14], Malva parviflora [15], Petroselinum crispum leaf extract [16], black tea leaf [17], Chinese citrus peel (Navel orange) [3], citrus sinus and citrus limitta [7] were used in the preparation of (AgNPs). It has been recently reported (Luu, Cao, et al. 2020, Biv and Nolan 2021, Eassa and Emam 2021, Jahan and IŞILDAK 2021, Niluxsshun, Masilamani, et al. 2021) that lemon was used in the synthesis of silver nanoparticles AgNPs [5, 10, 18-20]. Despite the fact that many studies have indicated that lemon extract has great potential in other fields, little attention has been given to the role of silver nanoparticles synthesized by lemon extract in reducing heavy metals from water. Previous studies have successfully described the characterization of silver nanoparticles using different techniques, including HR-TEM [21], UV-Vis [22], SEM [23], XRD [24], FTIR [25], DLS [26], Zeta sizer [27]. Numerous studies have demonstrated that nanoparticles can reduce heavy metal concentration in soil, water, and food. Khane et al. synthesized silver nanoparticles using citrus lemon zest extract [28]. Hawar et al. prepared silver nanoparticles using Alhagi graecorum leaf extract [29]. In a study by Barnawi et al., nickel elements were reduced in water by using a green synthesis of gold nanoparticles [30]. Using chitosan as the functional monomer, Chen, Ma, and Peng synthesized nano-sized magnetic ion polymers (MIIPs) to selectively adsorb nickel from the solutions [31]. Hong, Xie, Mirshahghassemi, and Lead have synthesized magnetic nanoparticles coated with polyvinylpyrrolidone (PVP - Fe₃O₄ nanoparticles) for the removal of metals (cadmium, chromium, nickel, and lead) from seawater and purified water manufactured with and without fulvic acid [32]. Several studies have examined how heavy metals can be removed from water but, our biosynthesis method is different and is regarded as a reliable, cost-effective, convenient, and environmentally friendly method.

In this study, silver nanoparticles (AgNPs) were synthesized by a green logical route using extracts of lemon citrus fruits and different amounts of silver nitrate. Various varying conditions were then studied and characterized via UV-visible spectroscopy, High-Resolution transmission electron microscopy (HR-TEM), and Fourier transform infrared spectroscopy (FT-IR). The ability of the synthesized AgNPs to remove nickel ions from aqueous solutions was investigated. As a result, the prepared samples can be used to purify water and remove heavy metals.

Materials and Methodology

Materials

The silver nitrate (AgNO₃) (99%) was purchased from BDH (England). Sodium dodecyl sulfate (SDS) 99% was obtained from Sigma-Aldrich (USA). Fresh fruit of citrus lemon was purchased at the local market in Mecca (Saudi Arabia). The experiments were conducted using double-distilled water. To remove traces of metal contaminants glassware was washed with aqua regia.

Preparation of Lemon Extract

The fresh citrus lemon extract was used as a reducing and capping agent. Lemon juice was extracted by washing, cutting, and squeezing the juice. The extract was filtered using Whatman filter paper to remove unwanted impurities. Afterward, the mixture was boiled for two hours, and the juice was used for further experiments.

Green synthesis of Silver Nanoparticles

The synthesis of AgNPs was based on previously published works with some modifications [33]. The sample S_1 of silver nanoparticles was prepared by dissolving 0.1019 g of $AgNO_3$ (3 mM) in 200 ml distilled water. This solution is added dropwise to 40 mL of lemon extract at 80 \boxtimes while stirring continuously under dark conditions for reduction of Ag⁺ ions. In the presence of lemon extracts, a reduction process produced AgNPs. In continuous heating and stirring, Ag⁺ ions are converted to Ag⁰ particles, which results in the creation of silver nuclei. Additionally, the solution's color was altered as a result of this process. After the nucleation process had begun, additional heating and stirring helped the nucleation and growth processes, resulting in synthesizing the required AgNPs. A reduction process was observed here; as a consequence, the observed color change could be attributed to a reduction of Ag⁺ ions to Ag⁰ particles. Several studies describe a similar process [34, 35]. As a result, the yellowish color appeared simultaneously with the addition of the silver solution, then turned to yellow after two minutes before turning into a dark brown. 12 mL of 8 % wv Sodium dodecyl sulfate (SDS) was added, after three hours of a mixture to prevent the aggregation of silver nanoparticles, and the hot plate was stopped after 25 minutes. The colloid is stored at room temperature. Samples S_1 and S_3 were prepared in the same manner as before, with the concentrations of $AgNO_3$ changing to 4 mM and 5 mM, respectively. For these samples, UV-vis spectra and TEM images were measured. For infrared measurement, each sample was centrifuged at (15,000 rpm) for 30 minutes, washed with distilled water, and then dried at 80 °C for 18 hours. Figure 1 illustrates the steps involved in the preparation of silver nanoparticles. Figure 2 shows the images of the three samples.

Result and Discussion

Ultraviolet-Visible Spectra

As silver samples changed in color, the colloidal solution was analyzed by UV-Vis spectroscopy, and changes in silver color were monitored. Figure 3 illustrates the absorption bands of the synthesized samples at different wavelengths. According to Figure 3, the absorption peaks appear at 432 nm, 452 nm, and 448 nm of (S1), (S2), and (S3), respectively. Those bands are due to the formation of silver nanoparticles and are distinguished by the excitation of surface plasmon vibration in AgNPs [36, 37]. In silver nanoparticles, the conduction and valence bands are close to each other, which permits electrons to move freely. A specific wavelength of light excites these conduction-free electrons on the surface of AgNPs, causing them to oscillate. Since the electrons oscillate around the silver nanoparticle's surface, the charge separation with respect to the ionic lattice forms a dipole oscillation that travels in the same direction as the light's electric field. At a particular frequency, the maximum oscillation amplitude is known as SPR [37]. Moreover, these peaks are sensitive to nanoparticle size, shape, the concentration of lemon, and silver nitrate ratio [38-40].



Figure 1: Representation diagram of experimental steps for preparing AgNPs sample S₁



Figure 2: Silver nanoparticle prepared by adding 200 ml of silver nitrate in three different molar ratios; (S1) (3 mM), (S2) (4 mM), and (S3) (5 mM) to 40 mL lemon extract.

The SPR band in (S1) and (S2) have wavelengths of 452 nm and 452 nm, respectively, which increase with increasing in AgNO₃ concentration and then decrease again in (S3) to 448 nm. It has been suggested that silver nanoparticles form through the aggregation of Ag atoms by reducing $AgNO_3$, Ag⁺ ions into silver at the nanoscale. In the present study, the size of silver nanoparticle was increased by increasing the concentration of $AgNO_3$ from (3 mM) to (4 mM) [37]. As the size of the AgNPs increased, the SPR shifted toward to the red absorption band (high wavelength), resulting from electric dipole interaction and coupling between plasmons oscillation of neighboring particles. Sample (S3) contains 5 mM of AgNO₃, which is the highest amount, showing particular results at a lower wavelength. As the precursor was added step by step to reducing agents, with some precursor added at the beginning of synthesis, the silver ions completed their nucleation phase as the precursor was continually added. In this case, the particles would be larger. The extra amount of precursor, however, may lead to the formation of new nucleation centers, which would appear as tiny metal nanoparticles and could result in a blue shift in the spectrum.



Figure 3: Ultraviolet-visible spectra of silver nanoparticles prepared by adding 200 ml of silver salt in three different molar ratios; (S1) (3 mM), (S2) (4 mM), and (S3) (5 mM) to 40 mL lemon extract.

HR-Transmission Electron Microscopy (TEM)

A high-resolution transmission electron microscope (HR-TEM) image of silver nanoparticles is shown in Figure 4. According to the graph, sample (S1) exhibited spherical AgNPs with a diameter of about 23 nm ~ 43 nm as plotted in the Gaussian distribution. Furthermore, most particles appear as spherical shapes with smooth edges and an average size of approximately 31 nm. There are also a few ellipsoids present. Furthermore, HR-TEM of the sample (S2) reveals a random distribution of silver nanoparticles. A spherical shape is also present, as well as some oval shapes with smooth edges. In this sample, particle size fluctuation is about 8 nm ~ 20 nm with an average size of 15 nm, as shown in the form of a Gaussian distribution opposite HR-TEM image of (S2). The HR-TEM image of the sample (S3) also exhibited spherical, hexahedral, and ellipsoidal silver nanoparticles. As shown in the Figure corresponding to the Gaussian distribution of the HR-TEM image of (S3), the majority of AgNPs were in the range between 13 nm ~ 37 nm as a spheroid with an average size of 28 nm. According to HR-TEM analysis of silver nanoparticles, most anisotropic nanostructures have a spherical shape compared with other geometric shapes. There have been several publications that have documented that silver nanoparticles prepared by plants have a variety of geometrical shapes [41]. The average particle size was calculated from the relationship between repeated sphere particles in the same size sample. Moreover, the HR-TEM result is also inconsistent with the UV-Vis data about the position of the surface plasmon band, which may be due to the aggregation of AgNPs during the TEM measurement.

Fourier Transform Infrared Spectroscopy (FTIR)

For IR measurement, 0.002 grams of prepared sample (powder) were mixed with 0.198 grams of KBr in an agate mortar, and the powder was then compacted into a disc. Then, let IR radiation pass through a disc. The FTIR absorption spectrum of lemon juice extract and the biosynthesized silver nanoparticles at different concentrations of silver nitrate is shown in Figure 5. In silver nanoparticles, the absorption band that appears at 3432 cm^{-1} is associated with the O–H stretching of hydroxyl groups, was moved to a lower wavenumber at 3403 cm^{-1} (S1) and 3415 cm^{-1} in (S2), and 3402 cm^{-1} in (S3). There is a change in frequency due to the hydroxyl group in the citrate component and the moisture in the water in the samples or the change in electrostatic negativity around OH. In addition, the sample (S1) has a sharper peak with a more intense band, which is separated into two peaks at 3470 cm^{-1} and 3403 cm^{-1} . This may be due to the lower concentration of silver nitrate in the sample (S1). It has been observed that the

characteristic band of C – H asymmetric stretching observed in lemon FTIR at 2924 cm⁻¹ has not shifted in the samples (S2) and (S3), but has changed to a higher wavenumber at 2931 cm⁻¹ in the sample (S1). As compared to the symmetric C – H stretch band recorded at 2853 cm⁻¹, which is 2854 cm⁻¹ for (S1), 2857 cm⁻¹ for (S2), and 2856 cm⁻¹ for (S3), there is a slight shift in the symmetric C - H stretch band in the whole silver nanoparticle samples.



Figure 4: High-Resolution transmission electron microscopy images of silver nanoparticles samples S1-S3 and their Gaussian distribution particle size.



Figure 5: FTIR of synthesized silver nanoparticles samples (S1-S3).

Symmetric and asymmetric confirmed the presence of a C – H stretching vibrational bond in the sample. All these changes in the O – H group and C – H bands might be due to the changes in the ratio of AgNO₃ that affected the electrostatic negativity in the samples. A stretching vibration related to the carbonyl group C = O is observed at 1730 cm⁻¹ in lemon [30]. The band was shifted to 1642 cm⁻¹, 1633 cm⁻¹, and 1648 cm⁻¹ in samples (S1), (S2), and (S3), respectively. This change is due to silver nanoparticles chemically coordinated with oxygen in the carbonyl group C = O [30]. For samples (S1), (S2), and (S3), the C-H bend vibration band appeared at about 1387 cm⁻¹, 1391 cm⁻¹, and 1392 cm^{-1,} respectively. In the silver nanoparticles surrounded by the citric extract, there are absorption bands around 500 cm⁻¹, likely caused by Ag - O vibration peaks. Assigning absorption bands to

samples (S1-S3) is summarized in Table 1. Based on $\frac{1}{2\pi c}\sqrt{\frac{k}{\mu}}$, the stretching vibration of Ag – O can be calculated at 778.86

cm⁻¹, where \bar{v} represents the absorption of infrared radiation, k represents the force constant of spring, μ refers to a reduction in mass of atoms, m_1 and m_2 represent the mass of atom 1 and 2, respectively [42, 43]. The slight difference in theoretical and experimental results differ slightly due to silver nanoparticles being coordinated with other functional groups in the sample.

Wavenumber (cm [°])			Assignments
(\$1)	(\$2)	(\$3)	
3470, 3403	3415	3402	O – H stretching vibration
2931	2923	2923	C – H asymmetrical stretching
2845	2857	2856	C – H symmetrical stretching
1642	1633	1648	C = O carbonyl group
1387	1391	1392	C – H bending vibration
567	592	587	Ag – O stretching vibration

Table 1: FTIR absorption bands and functional assignment groups for silver nanoparticles capped with lemon extract samples (S1 - S3).

Application of Silver Nanoparticles in Water Purification

UV-Visible Spectroscopy with Different Concentrations of Nickel Nitrate

Figure 6 shows the UV-visible spectra of nickel nitrate hexahydrate dissolved in distilled water at different concentrations $(1 \times 10^{-1} \text{ M})$, $(2 \times 10^{-1} \text{ M})$, $(3 \times 10^{-1} \text{ M})$ and $(4 \times 10^{-1} \text{ M})$. It is clear from this Figure that two bands with high absorption values were seen at 300 nm and 396 nm and two bands with low absorption values at 656 nm and 720 nm, respectively. It has been discussed previously that the electronic transition occurs when molecular ions absorb radiation and cause absorption bands in a range of wavelengths [44]. In nickel nitrate hexahydrate dissolved in water, the two sharp bands were observed at 300 nm and 396 nm as a result of the electron transfer between nickel ions. Based on these results, it appears that nitrate is uncomplexed. There are two low bands at 656 nm and 720 nm produced by d-d^{-d} electronic transitions [45]. The relationship between nickel nitrate hexahydrate concentration was increased, the two bands were increased. In general, the change in intensity follows Beer-Lambert law $A = \log_{10} \left(\frac{I_0}{I}\right) =$ ecl which demonstrates increased absorbance with increasing concentration.

Study the Changes in the Concentration of Nickel Dissolved in Water After Passing Through A Filter Impregnated by Silver Nanoparticles

The effect of AgNPs on nickel concentrations in water was examined by imbibing 5 ml of AgNPs with an average particle size of 28 nm into a filter element (WILKERSON). The filter was then dried in an oven at 40 degrees. A dissolved nickel nitrate solution of different concentrations was then passed through the filter, and the filtered solution was immediately measured by UV-Vis. Figure 8 shows that there are four absorption bands observed in this Figure around 300 nm, 396 nm, 656 nm, and 720 nm. As nickel concentrations changed, intensities changed. Figures 9 and 10 demonstrate the change in absorbance intensity with nickel concentrations for the 300 and 396 nm bands studied for the filtered solution with and without silver nanoparticles. In filtered solutions impregnated with silver nanoparticles, the absorbance intensity decreased at concentrations higher than (2x10⁻¹), which indicates that these filters can reduce nickel concentrations beyond this level. Since silver has a high electronegativity, it is preferred to utilize AgNPs membranes at high concentrations of nickel.



Figure 6: UV-Vis absorbance spectra of nickel salt solutions at different concentrations.



Figure 7: Relationship between the absorbance intensities of nickel electronic transition bands with concentrations at 300 nm and 396 nm.



Figure 8: UV–Vis absorbance spectra of Ni solution with different concentrations after passing through a filter impregnated with silver nanoparticle of size 28 nm.



Figure 9: Relationship between the absorbance intensity values at 300 nm with change in nickel concentration before and after passing through an imbibed filter with AgNPs 28 nm.



Figure 10: Relationship between the absorbance intensity values at 396 nm with change in nickel concentration before and after passing through an imbibed filter with AgNPs 28 nm.

Conclusion

Greenly synthesized silver nanoparticles with different particle sizes were achieved using lemon extract and silver nitrate concentrations. A color change in UV-Vis spectroscopy confirmed the formation of AgNPs. A HR-TEM was used to define particle morphology and distribution, and average particle sizes were calculated. Fourier transform infrared measurement revealed the presence of different functional groups in silver nanoparticles. AgNPs prepared for desalination were used to reduce nickel concentration in water. The study recommends the use of different nanoparticles for groundwater treatments.

Conflict of interest

We wish to confirm that there are no known conflicts of interest associated with this publication and there has been no significant financial support for this work that could have influenced its outcome. We confirm that the manuscript has been read and approved by all named authors and that there are no other persons who satisfied the criteria for authorship but are not listed.

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