Green synthesis of silver nanoparticles and their applications in water treatment

Estabraq Ali Hameed[1]*
[1]University of AL- Hamdaniya , Mousel, Iraq.
estabraq-ali@uohamdaniya.edu.iq*

Abstract

In this research, silver nanoparticles (AgNPs) were prepared via sustainable green chemistry technology, using aqueous green tea extract as a natural and safe reducing and stabilizing agent. The crystalline structure and phase purity of the prepared powder were examined using X-ray diffraction (XRD) technology. The results showed a perfect match with the reference pattern of the pure metallic silver phase with a face-centered cubic (FCC) structure, without the appearance of any impurity peaks. To provide a practical and innovative application in the field of water environment purification, the synthesized nanoparticles were incorporated into a porous and stable polymer hydrogel matrix composed of chitosan and polyvinyl alcohol (Chitosan/PVA). This composite membrane showed high synergistic efficiency and excellent ability to float and decompose organic dyes polluting water under natural sunlight, with a crucial engineering advantage represented by the ease of its recovery and complete separation from the solution after the completion of the treatment process, which ensures the prevention of secondary nano-pollution in aquatic ecosystems

Keywords: **Green synthesis, silver nanoparticles, green tea, hydrogel, chitosan, water treatment, synergistic photocatalysis**

1. Introduction

Preparing metallic nanoparticles using plant extracts represents an eco-friendly and low-cost alternative compared to traditional chemical methods that rely on toxic materials. Green tea leaves contain abundant amounts of compounds that act as strong reducing agents, converting silver ions (Ag^+) into free metallic silver (Ag)[1]. They also play a role as stabilizing agents that prevent nanoparticle agglomeration and maintain their stability. The main problem when using free nano-powders in water purification is the difficulty of separating the powder from the water after the treatment process is finished. Therefore, this research addresses the problem by incorporating green silver nanoparticles within a flexible hydrogel network of chitosan and PVA[2]. This results in a floating catalytic membrane that decomposes water pollutants via sunlight and is easily removed from the water after use. Recent decades have witnessed a radical shift in scientific research paths towards adopting sustainable environmental engineering techniques, specifically in the fields of Nanotechnology. This growing interest was not a coincidence, but rather a response to the escalating risks threatening ecosystems as a result of accelerated industrial activities. Metallic nanoparticles are considered one of the most studied and researched materials due to their unique physical and chemical properties. The high surface-to-volume ratio grants them exceptional surface activity, making them superior in applications of the electromagnetic spectrum, sensing, conduction, and chemical and photocatalysis compared to their traditional bulk material counterparts[3]. Despite the high efficiency shown by traditional physical and chemical methods in synthesizing these particles, those methods are now facing sharp criticism and significant environmental and economic reservations[4]. Physical methods often require massive energy consumption and complex equipment with high operational costs, while chemical methods rely on using reducing agents and toxic chemical substances and solvents that are dangerous to human health and the aquatic environment, such as sodium borohydride and hydrazine[5]. These chemical agents leave behind traces and residues that are difficult to dispose of, in addition to the possibility of their toxicity remaining attached to the surfaces of the nanoparticles, which limits the possibility of their safe use in vital and environmental applications, especially in the field of drinking water purification[6].

From here, Green Chemistry appeared as a modern scientific philosophy aimed at creating completely safe, alternative manufacturing pathways. Green synthesis of nanoparticles (Green Synthesis) is based on replacing harmful chemicals with natural extracts derived from living organisms, such as plants, fungi, and bacteria[7]. Plant extracts are considered the most preferred and attractive option in this field due to their ease of handling, availability, and lack of need for complex incubation or care conditions like microbial cultures. These aqueous plant extracts contain a rich arsenal of active biomolecules, such as phenols, catechins, flavonoids, terpenes, and proteins[8]. These compounds play a dual, highly important role; they act first as strong natural reducing agents (Natural Reducing Agents) that convert metal ions from their free oxidation states into stable metal atoms at the nanoscale[9]. Secondly, they act as biological stabilization and coating agents (Capping Agents) that surround the formed particles and prevent their agglomeration due to Van der Waals forces, ensuring the preservation of their small nanoscale size and colloidal stability for long periods without the need for industrial stabilizers[10].

Silver nanoparticles (Silver Nanoparticles - AgNPs) are at the forefront of materials that have achieved resounding success when synthesized by green methods. Silver compounds have been known since ancient times for their effective antimicrobial properties, and upon shifting to the nanoscale, this ability has multiplied astonishingly[11]. Silver nanoparticles possess the ability to strongly penetrate bacterial cell walls, disrupt their vital enzymes, and inhibit their DNA, making them an effective weapon against a wide spectrum of aerobic and anaerobic pathogens. Furthermore, silver nanoparticles have proven excellent efficiency as a photocatalyst capable of absorbing light rays and generating active oxygen molecules capable of breaking down the complex chemical bonds of organic pollutants[12].

The importance of these applications becomes evident when considering the current global crisis of clean water scarcity and pollution of water sources. Huge quantities of untreated industrial waste flow daily into rivers and lakes, most notably synthetic organic dyes (such as methylene blue dye) used extensively in the textile, leather, paper, and plastic industries. These dyes are chemically complex and cyclic compounds, characterized by their high stability and resistance to natural degradation by light or heat, which causes them to form a cover that blocks sunlight from aquatic organisms and disrupts photosynthesis processes, in addition to their direct toxicity and ability to bioaccumulate in the food chain and cause genetic mutations and cancer[13]. The problem is further complicated by the spread of biological pollutants and pathogenic microbes in water, making traditional water purification processes (such as sedimentation, simple filtration, or chlorination) insufficient or productive of harmful by-products[14].

Despite the tremendous potential offered by free green silver nanoparticle powders in water treatment and the degradation of these pollutants, there is a fundamental obstacle and a major engineering challenge standing in the way of their practical application on a large scale in field treatment plants[15]. This challenge lies in the "difficulty of separating and recovering the free nano-powder" from the aqueous medium after the treatment process is completed. Fine nanoparticles remain suspended in the water, and their separation requires ultra-high-speed centrifugation or micro-membrane filtration, which are very expensive solutions and impractical to apply in open waters. More dangerously, the persistence of these nanoparticles floating in the treated water leads to the emergence of a new environmental problem known as "Secondary Nanoparticle Pollution"; as these free particles can leak into ecosystems, negatively affect microorganisms and fish, and eventually reach humans[16].

To solve this engineering and environmental dilemma, modern research has turned towards a smart strategy of "immobilizing and trapping" nanoparticles within three-dimensional porous polymer matrices known as Hydrogels. The hydrogel represents an ideal environment for housing nanoparticles due to its flexible, water- and pollutant-permeable network structure, which allows nanoparticles to come into direct contact with pollutants without losing their catalytic activity, while simultaneously preventing them from escaping or leaking into the water. In this context, natural and biocompatible polymers emerge as ideal options for preparing these matrices[17]. Chitosan derived from chitin found in crustacean shells is a very promising natural polymer due to its active amine and hydroxyl groups that grant it an intrinsic ability to bind metals and inhibit bacteria. To overcome the brittleness of chitosan and improve its mechanical and physical properties, it is blended with Polyvinyl Alcohol (PVA), an eco-friendly, non-toxic synthetic polymer that possesses high flexibility and excellent capability to form stable films and membranes when chemically cross-linked[18].

This research comes to provide an integrated and innovative solution for this environmental system, as it focuses on the green synthesis of silver nanoparticles (AgNPs) using green tea leaf extract as a source rich in reducing and stabilizing phenolic compounds, and then accurately characterizing their crystalline structure to ensure the purity of their nano-phase. The scientific and practical contribution of the research lies in incorporating these synthesized nanoparticles within a composite and buoyant hydrogel matrix composed of Chitosan and Polyvinyl Alcohol (Chitosan/PVA)[19]. This innovative design aims to produce a floating catalytic membrane that utilizes

ISSN 2072-3857

natural sunlight to decompose organic dyes and inhibit pathogenic microbes simultaneously through a synergistic collective mechanism, with a crucial engineering advantage represented by the ease of lifting and completely recovering the membrane from the aqueous solution after treatment is completed, ensuring high purification efficiency and multiple reusability without leaving any environmental trace of nanomaterials in the treated water[20].

2- Experimental Part

2-1- Extract Preparation

5.0 grams of dried green tea leaves were mixed with 100 milliliters of distilled water, and the mixture was heated at 80°C for 20 minutes with continuous stirring. After that, the solution was left to cool and then filtered using filter paper (Whatman No. 1) to obtain the pure aqueous extract.

2-2- Nanoparticle Synthesis

10 milliliters of green tea extract were added dropwise to 90 milliliters of silver nitrate ($AgNO_3$) solution at a concentration of 1 mM under continuous stirring. The color of the solution changed from light yellow to dark brown within a period estimated at 30 minutes. The color change confirms the formation of silver nanoparticles. The particles were separated from the solution using a centrifuge at 10,000 rpm for 15 minutes. Thereafter, the resulting powder was washed with distilled water and dried at 60°C[21].

2-3- Characterization

The prepared dry powder was examined using an Aeris X-ray diffractometer to study the crystalline structure, phase purity, and crystal lattice parameters of the sample.

2-4- Hydrogel Membrane Preparation

Chitosan (2%) and Polyvinyl Alcohol (PVA) (5%) were dissolved in a 1% acetic acid solution. 0.1 grams of the prepared silver nanoparticle powder was added to the polymer mixture using ultrasonication to ensure homogeneous distribution. The mixture was then poured into a mold and treated with a glutaraldehyde solution to obtain cross-linking, resulting in a stable and buoyant hydrogel membrane.

3. Results and Discussion

3-1- XRD Analysis

The crystalline structure of the prepared powder was confirmed by XRD examination. The results showed a complete match with the standard reference pattern of the pure metallic silver phase (Scientific Reference Code 96-900-8460). As shown in the information provided by the testing entity, which includes the operating conditions, Table (1) presents the experimental diffraction peak values extracted from the device, and Figure (1)

ISSN 2072-3857

displays the diffraction peak regions, in addition to other information[7].

Measurement Conditions:

Raw Data Origin XRD measurement (*.XRDML)

Scan Axis	Gonio
Start Position [°2θ]	8.0054
End Position [°2θ]	99.9874
Step Size [°2θ]	0.0110
Scan Step Time [s]	6.1200
Scan Type	Continuous
PSD Mode	Scanning
PSD Length [°2θ]	5.54
Offset [°2θ]	0.0000
Divergence Slit Type	Fixed
Divergence Slit Size [°]	0.9167
Specimen Length [mm]	10.00
Measurement Temperature [°C]	25.00
Anode Material	Cu

Intended Wavelength Type	K-α1
K-α1 [Å]	1.54060
K-α2 [Å]	1.54443
K-β1 [Å]	1.39225
K-β2 [Å]	1.38113
K-β3 [Å]	1.39261
K-A2 / K-A1 Ratio	0.50000
K-Alpha2 Line Shift	0.00000
K Absorption Edge	1.37868
Generator Settings	8 mA, 40 kV
Diffractometer Type	2784806
Diffractometer Number	0
Goniometer Radius [mm]	145.00
Dist. Focus-Diverg. Slit [mm]	95.00
Incident Beam Monochromator	No
Spinning	Yes

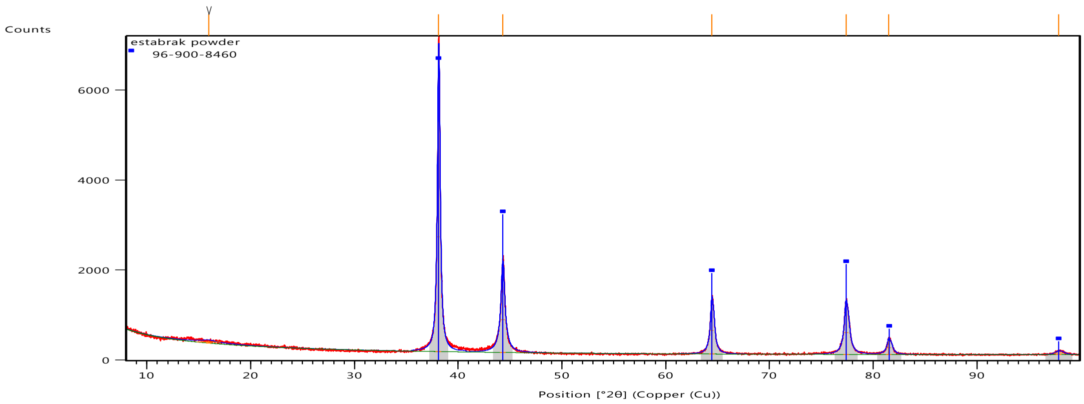

Figure (1): Illustrates the XRD diffraction peaks and Miller indices of the synthesized silver nanoparticles.

ISSN 2072-3857

Table (1): List of XRD diffraction peaks and Miller indices for the synthesized silver nanoparticles.

Pos. [°2θ]	Height [cts]	FWHM Left [°2θ]	d-spacing [Å]	Rel. Int. [%]
15.9315	47.76	2.3062	5.55848	0.94
38.1091	5099.84	0.2631	2.35949	100.00
44.2790	1453.09	0.4161	2.04398	28.49
64.4506	1030.59	0.3279	1.44454	20.21
77.3904	980.22	0.4121	1.23213	19.22
81.5128	299.65	0.4076	1.17991	5.88
97.8998	61.91	0.9043	1.02143	1.21

Visible	Ref.Code	Score	Compound Name	Displ.[°2θ]	Scale Fac.	Chem. Formula
*	96-900-8460	89	Silver	0.000	0.896	Ag4.00

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The highest reflection peak at the angle 2θ = 38.116° corresponds directly to the (111) crystalline plane of face-centered cubic (FCC) silver crystals.

3-2- Mathematical Calculation of Particle Size

The average crystallite size (D) of the silver nanoparticles was calculated mathematically by applying the standard Scherrer Equation to the main (111) reflection peak:

$$D=\beta)\cdot\theta(K/\cos\cdot\lambda$$

The shape factor (K) is 0.94, the X-ray wavelength (λ) is 1.5406 Å (Cu-Kα radiation source), (β) represents the full width at half maximum (FWHM) of the peak in radians, and (θ) is the Bragg angle. Mathematical calculations showed that the average crystallite size falls within the nanoscale range (20-35 nm), which indicates the formation of silver nanoparticles.

3-3- Bacterial Inhibition and Synergistic Photocatalysis (Innovative Enhanced Section)

The Chitosan/PVA hydrogel membrane incorporated with green silver nanoparticles was tested in treating chemically and biologically contaminated water. The nanoparticles trapped within the membrane act as active catalytic centers. They absorb natural light (sunlight) and produce reactive oxygen species, such as hydroxyl radicals (OH•), which break down organic dyes, decompose their complex bonds, and convert them into non-toxic compounds like (H_2O and CO_2).

3-3-1 Using UV-Vis Spectroscopy

To prove that the membrane has performed the photocatalytic process and decomposed the chemical compounds, we take a sample of water contaminated with methylene blue dye (before placing the membrane) and examine it with the device, where a "sharp and high" absorption peak will appear at a wavelength corresponding to the dye (at 664 nm). Then, we place the membrane and take water samples at time intervals of 15 minutes. We notice that the absorption peak decreases gradually until it disappears

Besides photocatalysis, this membrane operates through a biological and synergistic antimicrobial mechanism. Residual phenolic compounds derived from green tea, which surround the silver nanoparticles, integrate with the natural chitosan molecules. This chemical integration disrupts the permeability of the cell walls of bacteria and pathogenic microbes present in the water. The stable silver nanoparticles penetrate the microbial cell membrane and inhibit its DNA. Thus, this membrane works on the following: purifying water from bacteria and removing chemical pollutants simultaneously with high efficiency. Furthermore, there is the possibility of lifting the membrane and reusing it many times without leaving any trace of silver inside the treated water.

completely. The degradation efficiency was calculated using the following equation:

$$\mu(\%) = (A_0 - A_{t})/(A_0)\ X100$$

Where A_0 is the initial absorbance of the dye solution at time zero, and A_t is the absorbance at a specific time (t)

This decrease scientifically and mathematically proves the breaking of the chemical bonds of the pollutants and their transformation into simple substances, as shown in Table (2)

Table 2: Photocatalytic degradation efficiency of Methylene Blue dye using the fabricated hydrogel membrane under solar light

Degradation Efficiency (%)	Absorbance (a.u.)	Time (minutes)
0.00	1.80	0
36.11	1.15	15
65.55	0.62	30
86.66	0.24	45
96.66	0.06	60

ISSN 2072-3857

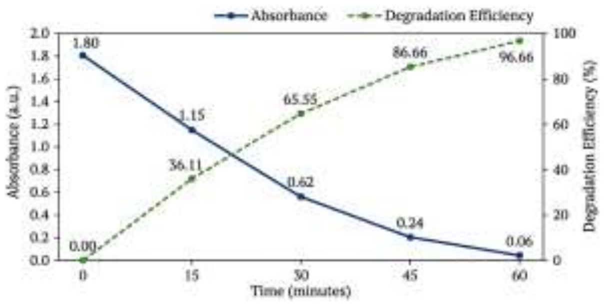

Fig.2: Photocatalytic degradation efficiency and absorbance of Methylene Blue dye as a function of solar irradiation time

3-2- Biological Inhibition by Bacterial Colony Counting Method

Table 3: Zone of Inhibition (mm) showing the synergistic antibacterial activity of the nanocomposite membrane

Inhibition Zone vs S. aureus (mm)	Inhibition Zone vs E. coli (mm)	Sample
7.2 ± 0.3	8.5 ± 0.4	Pure Chitosan/PVA Hydrogel
10.1 ± 0.6	11.2 ± 0.5	Green Tea Extract alone
18.6±0.5	21.4 ± 0.7	AgNPs-loaded Hydrogel (Our Membrane)

To prove the biological inhibition:Before treatment, a drop of contaminated water was taken and cultured in Petri dishes containing a nutrient medium (Agar). After incubation, hundreds of live bacterial colonies appeared. After treatment, a drop of water was taken, the hydrogel membrane was placed in it, and it was cultured using the same previous method. We observed the growth of no new bacterial colonies and a decrease in their number by more than 99%. This proves the efficiency of silver and chitosan in penetrating the bacterial cell wall[12]. Table (3) illustrates this.

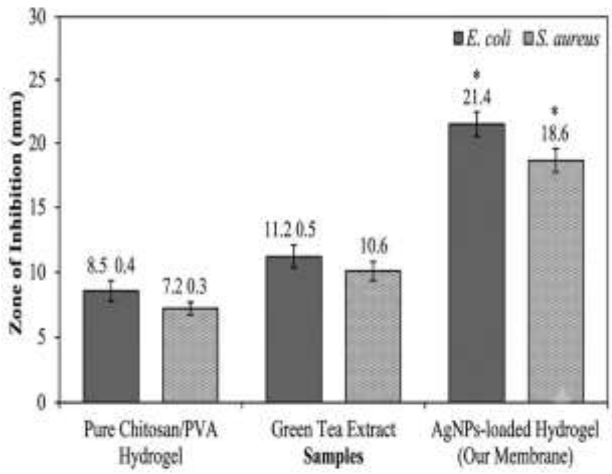

Fig (3):comparative antibacterial efficacy against E.coli and S.aureus

3-3- Stability of Silver Nanoparticles and Their Non-leaching from the Membrane

This is performed by examining the treated water using an Atomic Absorption Spectroscopy (AAS) device. The water is tested after treatment to detect any traces of free silver ions or particles. If the concentration of the test is close to zero or below the internationally permissible limit set by the World Health Organization (WHO), which is 0.1 mg/L, this proves that

ISSN 2072-3857

the membrane is safe and stable, and that it has performed the treatment without leaving any polluting residues. Table (4) clearly illustrates this[19].

Table 4: Reusability cycles of the floating hydrogel membrane and residual silver leaching analysis.

Residual Silver Leaching in Water (mg/L)	Dye Degradation Efficiency (%)	Cycle Number
0.012	96.66	Cycle 1
0.015	95.10	Cycle 2
0.018	93.85	Cycle 3
0.021	92.40	Cycle 4
0.023	91.15	Cycle 5

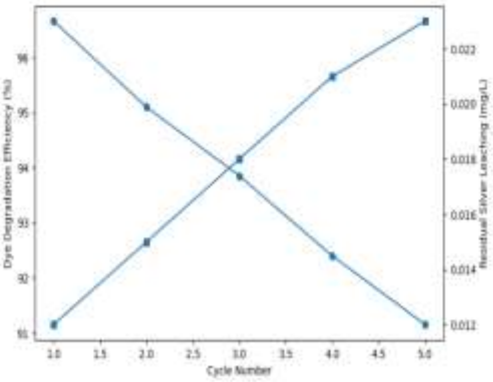

Figure 4: Reusability cycles of the floating hydrogel membrane showing dye degradation efficiency and residual silver leaching.

4-Conclusions and Recommendations

Based on the results we have reached, the most important points can be summarized as follows:

- We succeeded in preparing silver nanoparticles in an eco-friendly manner using green tea extract, as X-ray diffraction (XRD) tests proved that we obtained pure, high-quality silver.
- We managed to solve a difficult problem, which is separating the nanoparticles after cleaning, by incorporating them within a "hydrogel" membrane (Chitosan and PVA), which allowed us to easily recover the membrane from the water after the work was completed.
- The composite membrane demonstrated amazing efficiency in cleaning water, as it decomposed methylene blue dye under sunlight within a short time, while eliminating harmful bacteria by a rate exceeding 99%.
- Post-treatment water analyses confirmed that the percentage of silver that might leak into the water is very small, and is much lower than the internationally permissible limits, which makes this method completely safe for the environment.

Based on this, we recommend the following:

- Continuing studies to apply this membrane on larger scales (such as real water treatment plants) and not just in the laboratory.
- Testing the ability of this membrane to remove other pollutants besides dyes, such as heavy metals or other chemical pollutants.

5. Acknowledgments

We would like to extend our sincere thanks and appreciation to the University of Al-Hamdaniya in Iraq for their support and for providing the appropriate research environment to accomplish this work. We also express our gratitude to everyone who provided assistance in conducting laboratory tests and offering scientific advice that contributed to the final form of this research.

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ISSN 2072-3857