

Electrochemical Detection of Silver Ions Using Green-Synthesized Silver Nanoparticles: A Novel Approach for Environmental and Industrial Applications

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Abstract:

A modified carbon electrode with silver nanoparticles was manufactured to detect and estimate silver ions in X-ray film residues, industrial water and well water using green chemistry in a new and straightforward way. The work involved the preparation of silver nanoparticles from arugula (*Eruca sativa*) leaf extract as a primary material. The average size of the crystal measured by XRD was 15.2733 nm, and particles were identified using the SEM scanner electron microscope as semi-spherical. In UV-VIS spectrometry, the peak was at the wavelength 433 nm; the result proves that nanoparticles were manufactured from silver, and the functional clusters were identified using FTIR spectrometry. This Electrode showed excellent selectivity and sensitivity with a linear response from 10^{-1} – 10^{-6} molar. Correlation coefficient 0.9701, with a lifespan of 90 days. Optimal temperatures are 15-30 °C, ideal pH range 5-7, with a slope at 25 °C 58.3 mV/decade, detection limit 10^{-6} , and recovery percentage 100.21 %. This method has been applied to estimate silver ions in X-ray film residues, industrial water, and well water, and it has proved significantly effective.

Keywords: Green Synthesis, Silver Nanoparticles, Arugula, Heavy Metal.

الكشف الكهروكيميائي عن أيونات الفضة باستخدام جسيمات الفضة النانوية المصنعة بالطريقة الخضراء: نهج جديد للتطبيقات البيئية والصناعية

الباحث مصطفى فاضل نوري ، أ.د. شذى يونس يحيى ، م.د. مصطفى قتيبة جبار

مستخلص:

تم تصنيع قطب كربون معدل باستخدام جسيمات الفضة النانوية لاكتشاف وتقدير أيونات الفضة في بقايا أفلام الأشعة السينية، والمياه الصناعية، ومياه الآبار، وذلك باستخدام الكيمياء الخضراء بطريقة جديدة ومباشرة. تتضمن العملية تحضير جسيمات الفضة النانوية من مستخلص أوراق الجرجير كمادة أولية. كان متوسط حجم البلورة المقاس بواسطة حيود الأشعة السينية (XRD) حوالي 15.2733 نانومتر، وتم التعرف على الجسيمات باستخدام المجهر الإلكتروني الماسح (SEM) على أنها شبه كروية. أما في قياس طيف الأشعة فوق البنفسجية والمرئية (UV-Vis)، فقد ظهرت الذروة عند طول موجي قدره 433 نانومتر، مما يؤكد تكوين جسيمات الفضة النانوية. تم تحديد المجموعات الوظيفية باستخدام قياس طيف الأشعة تحت الحمراء (FTIR). أظهر القطب أداءً ممتازًا من حيث الانتقائية والحساسية، مع استجابة خطية في مدى تركيز من 10^{-1} إلى 10^{-6} مولاري، ومعامل ارتباط بلغ 0.9701. عمر القطب بلغ 90 يومًا، وكانت درجات الحرارة المثالية لتشغيله تتراوح من 15 إلى 30 درجة مئوية، أما نطاق الرقم الهيدروجيني الأمثل فكان من 5 إلى 7، أظهر القطب ميلًا كهربائيًا بمقدار 58.3 ملي فولت/عقدة عند درجة حرارة 25 درجة مئوية. وكان حد الكشف 10^{-6} مولاري، بنسبة ارتداد بلغت 100.21. لقد تم تطبيق هذه الطريقة بنجاح لتقدير أيونات الفضة في بقايا أفلام الأشعة السينية، والمياه الصناعية، ومياه الآبار، وأثبتت فعاليتها بشكل كبير. الكلمات المفتاحية: التركيب الأخضر، الجسيمات النانوية الفضية، الجرجير، المعادن الثقيلة.

1. Introduction

Nanoscience is defined by studying the initial linkages between the intrinsic characteristics and different production phenomena and determining the nanometer's material dimensions (1 to 100 nm). Nanoscience has a typical dimension range between a sub-nanometer and a few hundred nanometers. Nanoscale materials may possess unique properties that differ from large-mass materials.^(1, 2) Nanotechnology is the exploitation of the unique properties of materials in nanotechnology. The classification of nanomaterials is based on their dimensions. The word "nanotechnology" consists of two parts. The first "nano" is a Greek word which refers to "dwarf", that is, everything that is very small. Nanotechnology is a term used to describe the study and use of small objects in all fields of science, such as physics, materials science, chemistry, biology, and engineering.⁽³⁾

⁴⁾ Nanoparticles are a tremendous and versatile class of materials that have gained significant importance in various scientific and technological fields due to their unique properties, such as the ratio of surface area to large size

and optical, magnetic, adjustable catalytic and biocompatibility properties.⁽⁵⁾

⁶⁾ These characteristics make it suitable for applications in medicine, industry, agriculture and environmental management.^(7, 8)

In recent years, the manufacture of nanoparticles has undergone a marked shift towards green, environmentally friendly methods. Traditional nanoparticle manufacturing methods often include hazardous chemicals, which negatively affect the environment and produce toxic secondary substances. Green chemistry methods, therefore, provide a solution to this problem and are easy to use and apply through biomaterials such as microorganisms, plants and agricultural residues as environmentally friendly sources for forming nanoparticles.^(9, 10) In chemistry, nanoparticles have a significant role, especially in electrochemistry, the electrode industry, and so-called ion-sensitive or ion-selective electrodes. Nanoparticles have become an integral part of advances in electrochemistry due to their unique chemical and physical properties, which enhance the performance of different electrochemical applications. Its small size and high surface area allow for improved

catalytic activity, sensitivity and selectivity in electrochemical processes. This overview explores the diverse uses of nanoparticles in electrical chemistry, focusing on their roles in sensing, stimulation and energy storage.⁽¹¹⁾ Silver is one of the heavy metals; by nanotechnology and the green method, silver ions found in silver nitrate have been reduced to nanoscale silver particles with an increase in the surface area of silver.⁽¹²⁾ Silver nanoparticles greatly enhance the sensitivity and selectivity of electrochemical sensors. They facilitate electrical stimulation, charge transmission, and signal amplification, making them ideal for detecting gases, ions, environmental pollutants, and biomolecules.⁽¹³⁾

A modified carbon electrode was manufactured with silver nanoparticles extracted from arugula (*Eruca sativa*) leaves. Carbon or graphene is considered a single layer of hexagonal molecules.^(14, 15) Carbon was used to manufacture the electrode paste and blended with prepared silver nanoparticles. Carbon-based and graphene-based electrodes for gas sensors or heavy metal ions provide a good and innovative technical style, are easy to operate, have fast performance, and provide

accurate measurements.^(16, 17) Modified carbon paste electrodes (MCPs) are versatile tools in electrochemical sensing, providing improved sensitivity and selectivity for various analyses. These modifications often involve merging materials such as nanoparticles, polymers, or molecularly printed polymers to improve electrode performance. The changes address traditional constraints, such as low selectivity and sensitivity, by adapting the electrode surface to interact more effectively with specific target molecules.^(18, 20) This Electrode detected and estimated silver ions in the X-ray film because silver ions hurt humans, plants, and animals. Humans, plants, and animals absorb nanoscale silver ions through inhalation and oral methods, with slight skin absorption, resulting in deposition in various organs.⁽²¹⁾ Occupational Safety and Health Administration (OSHA) and Mine Safety and Health Administration (MSHA) proposed that the permissible exposure limit (PEL) for both metallic and most soluble Ag compounds should be 0.01 mg/m³.^(22, 24) Despite the benefits, improvements and uses of many silver nanoparticles, rapid progress in this area raises con-

cerns about potential risks to human health and the environment.

This study aims to develop a green, simple, and efficient method for detecting and estimating silver ions in environmental samples such as X-ray film residues, industrial water, and well water by fabricating a modified carbon electrode using silver nanoparticles synthesized from *Eruca sativa* (arugula) leaf extract.

2. Experimental

2.1. Chemical Substances and Solutions:

Silver nitrate solution: A solution of 0.1 M is formed by dissolving 1.6987 grams of the product in a 100 mL volumetric flask and diluted to the mark with deionized water. Sodium hydroxide solution 0.1 M is created by dissolving 0.400 g of the product in a 100 ml volumetric flask and adjusting the volume to the mark with deionized water. Hydrochloric acid solution 0.1 M hydrochloric acid solution was produced by adding 0.82 ml of hydrochloric acid (12.06 M, specific density 1.19 g/cm³, 37% solution) into a 100 ml volumetric flask and diluting to the mark using deionized water.

2.2. Preparation of arugula leaf extract solution and AgNPs formation:

The Arugula (*Eruca sativa*) leaf plants were collected from the local markets of Tikrit city in northern Iraq and classified in the Iraqi National Herbarium according to the approved taxonomic keys. Weigh 10 grams of fresh arugula leaves in 150 mL of deionized water. Heat 50-60 degrees Celsius for 50 minutes, cool the extract to room temperature. Filter the extract using filtration paper, then use a 0.45 μm syringe filter to filter out pollutants; keep the extract in the refrigerator. Add 100 ml of the pre-prepared arugula extract into a 500 ml beaker. Add 100 mL of the 0.1 molar silver nitrate solution into the burette. Gradually add small quantities of the silver nitrate solution to the arugula extract while stirring constantly and heating to 40-50 °C. Observe the color change of the mixture from light yellow to dark brown, indicating the reduction and formation of silver nanoparticles, which will be later identified. Leave the mixture to cool at room temperature. Filter the mixture using filter paper and let it dry for 24 hours or put it in an electric oven set to 70 °C for 1 hour. Figure1.

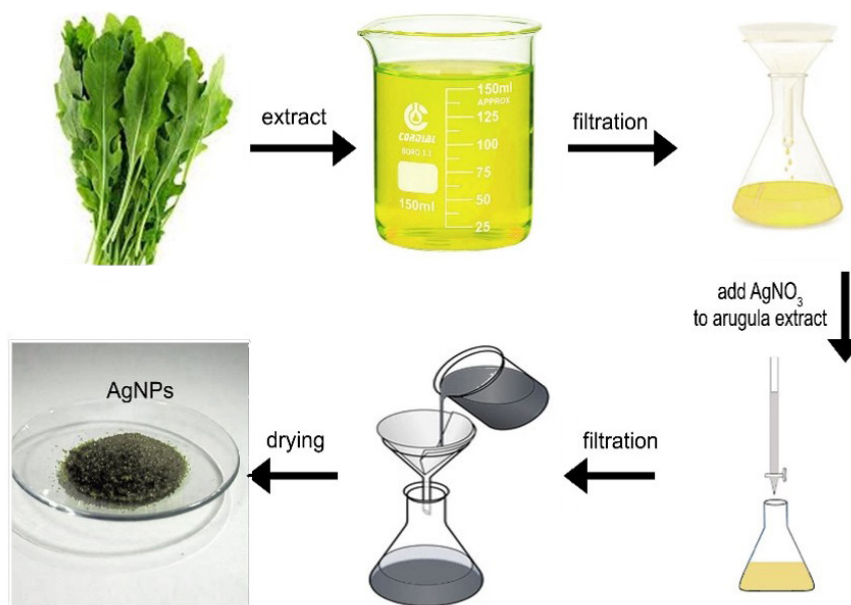


Figure 1. Steps to prepare arugula extract and prepare silver nanoparticles

2.3. Prepare Carbon-AgNPs Paste:

Carefully crush the carbon powder using an agate mortar, then mix a tiny quantity of AgNPs into the powdered Carbon in a 50:50% silver to carbon ratio. Mix the final composition for 30

minutes until it becomes uniform, then gradually add tiny drops of paraffin oil until a uniformly sticky texture is reached. Let the paste sit for 24 hours to remove excess paraffin oil. Figure 2

**Figure 2.
Carbon-silver
electrode
preparation**



2.4. Preparation of modified carbon-silver electrode.

A PVC plastic tube is created using a 5cc syringe tube with a copper wire placed on the tip. Transfer a small amount of prepared paste into the first end, ensuring the copper wire reach-

es the paste. Repeat this step until the tube's tip is full, then compress the second end to secure the paste. Allow the Electrode to rest for 24 hours, then immerse it in a 0.1 M silver nitrate solution for 4 hours. Figure 3.



Figure 3. Carbon-silver electrode model.

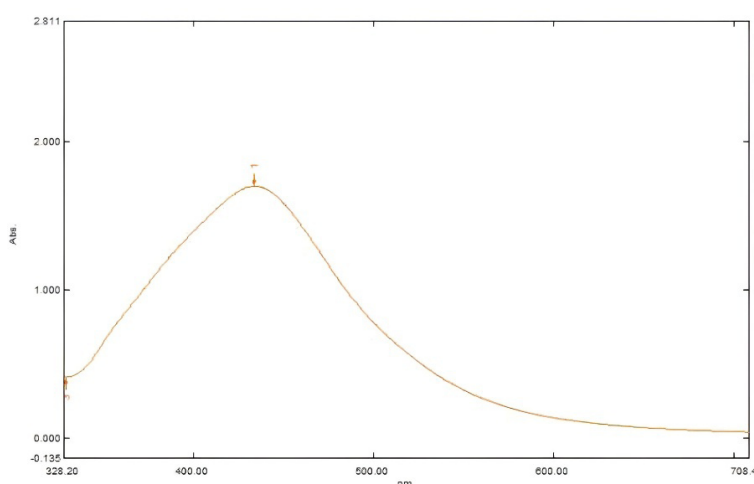
3. Results and Discussion:

3.1. Characterisation of AgNPs

3.1.1. UV-Visible Spectroscopy for AgNPs

The UV-visible spectra of silver nanoparticles have been measured as a function of 5mL wavelength using a UV-visible spectrophotometer (SHIMADZU, Japan) with a resolution of

0.5 nm. 5 arugula leaf extract solutions, including nano silver particles, were studied using UV-Vis spectroscopy. The ultraviolet spectrum reached its maximum at a wavelength of 433 nm (Figure 4). This corresponds to the stable Ag NPs structure that absorbs light within the 400 to 460 nm²³ range.



**Figure 4.
UV-VIS
spectroscopy
for Ag NPs**

3.1.2. SEM Microscopy of Ag NPs prepared by plant extracts.

The scanning electron microscope (SEM) was applied to study the surface morphology of the created samples. Figure 5 presents scanning electron microscopy images of Ag NPs fabricated at 1 μm and 200 nm scales. The synthesized AgNPs exhibited uniform

semi-spherical silver nanoparticles with an average particle size of 32.94 nm, consisting of agglomerated ultra-fine particles, as shown in Figure 5. Images from the scanning electron microscope revealed that applying arugula leaf extract reduced the dimensions of silver nanoparticles, each exhibiting varying sizes.

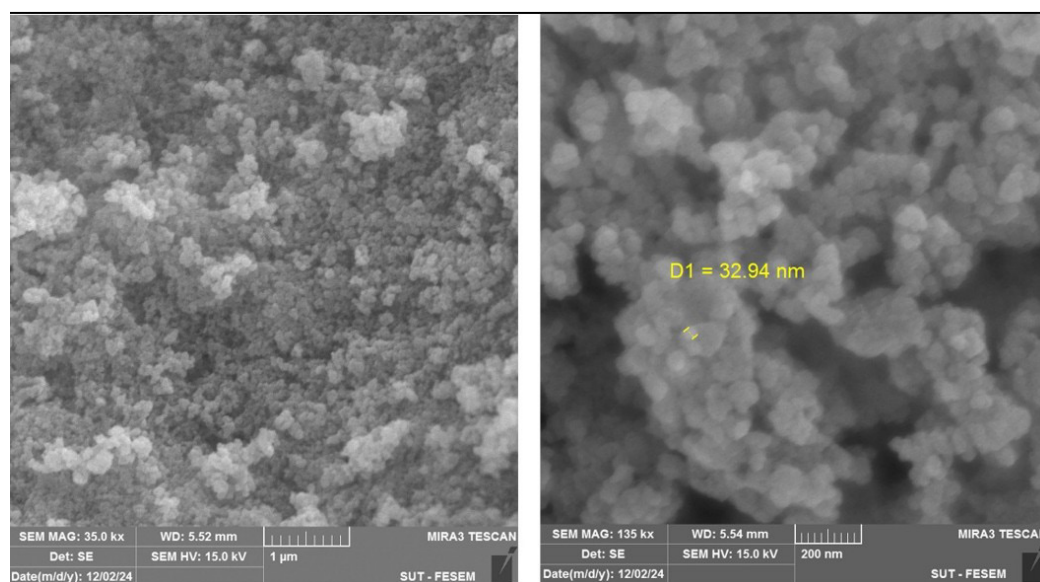


Figure 5. SEM image of prepared AgNPs

3.1.3. XRD Spectrum of AgNPs prepared by plant extracts.

As shown in Figure 6. XRD patterns indicated other unspecified diffraction peaks attributable to impurities. The size of silver nanoparticles was calcu-

lated using the Shearer equation; the results are shown in the table below.

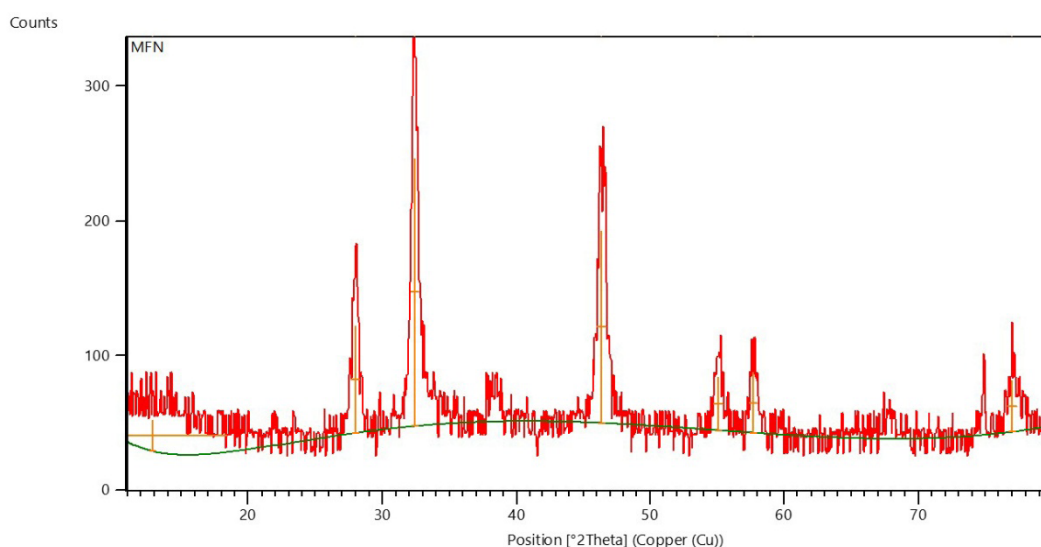


Figure 6. XRD patterns

Table 1. Summary of X-ray Diffraction Results of Nanoparticles.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip Width	Average D nm
13(3)	24(15)	11(3)	6.87693	11.81	13.4798	15.2733
27.95(1)	79(4)	0.65(3)	3.18936	39.80	0.7851	
32.385(6)	199(6)	0.55(3)	2.76223	100.00	0.6561	
46.375(8)	143(5)	0.65(3)	1.95637	71.95	0.7845	
55.05(2)	39(3)	0.67(6)	1.66671	19.82	0.8074	
57.68(2)	45(5)	0.56(4)	1.59689	22.43	0.6687	
77.00(3)	38(4)	0.7(2)	1.23740	19.08	0.8849	

3.2. Study the ideal conditions for manufacturing the Electrode.

The specifications and properties of the AgNPs nano-electrochemical sensor were confirmed after it was manufactured to determine the electrode response, linear concentration range,

slope value, coefficient of correlation, electrode lifespan, detection limit, accuracy, compatibility, selectivity, applications, pH function, and temperature effect.

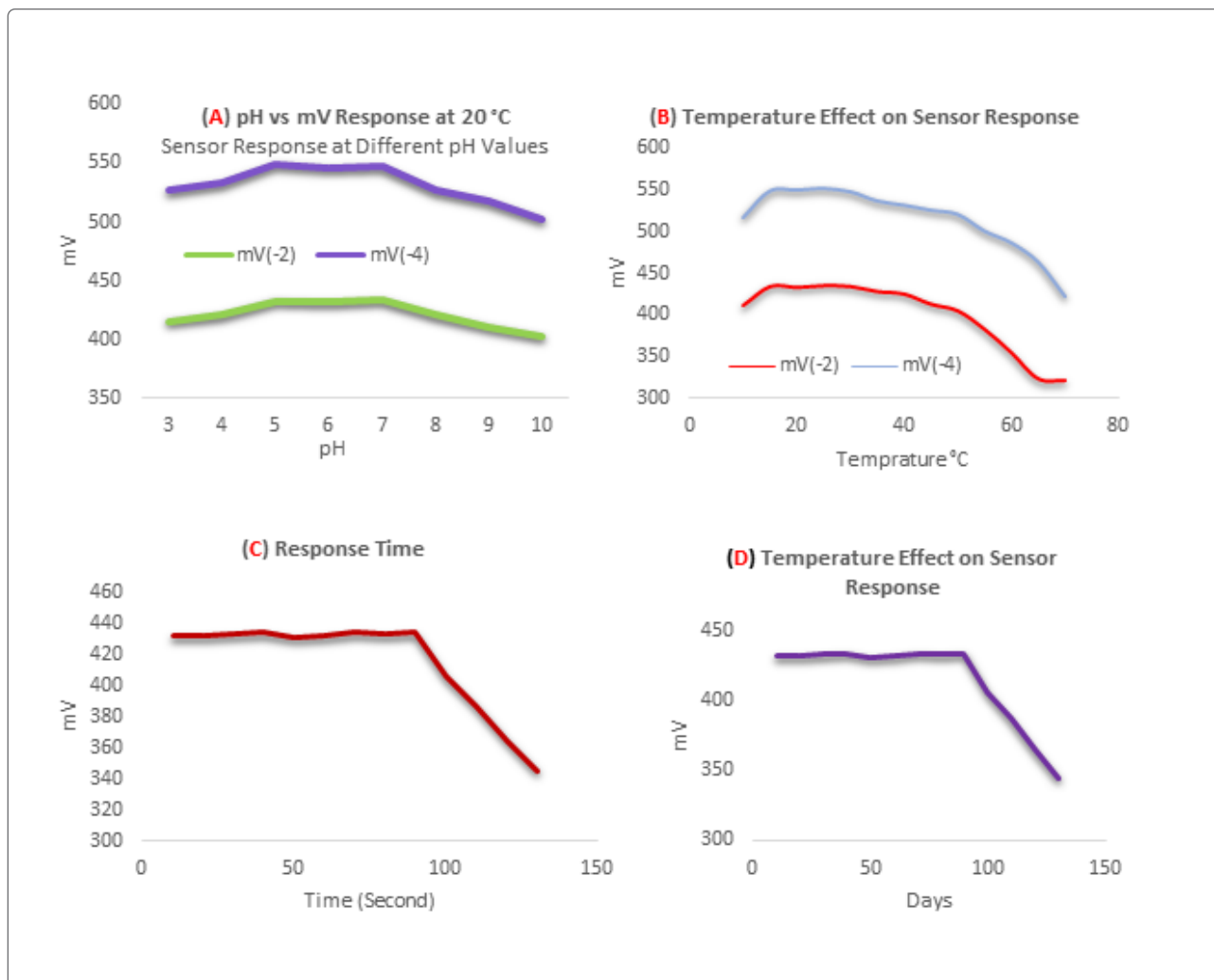


Figure 7. The performance of the modified carbon electrode with AgNPs was evaluated under various conditions. (A) The ideal pH range for stable voltage output was between 5 and 7, with deviations at higher pH levels due to silver hydroxide precipitation and at lower pH levels due to silver chloride formation. (B) The electrode functioned optimally at temperatures between 15–30°C, with a significant voltage drop beyond 30°C due to electrode paste

degradation. (C) The response time of the electrode varied between 2–20 seconds, with faster responses observed at higher ion concentrations. (D) The electrode lifespan was approximately 90 days, and maintaining stable voltage output before degradation led to a decline in performance, likely due to electrode paste deterioration or copper wire conduction failure.

The ideal pH value for modified carbon electrodes used with AgNPs,

without significantly impacting voltage, was between 5 and 7. The electrode measures remained stable and consistent within this range, with pH adjustments made using diluted sodium hydroxide and hydrochloric acid solutions. High pH levels (exceeding 8) have been kept away due to solution turbidity, as the addition of excess base resulted in the formation of a brown precipitate of silver hydroxide, while reduced pH values led to a hazy solution caused by the precipitation of insoluble silver chloride.

The effect of temperature has been investigated within the range of 15 to 75 °C. The best temperature range for the electrode sensor, ensuring proper functionality without significantly impacting voltage or electrode paste, is between 15-30 °C; a substantial decrease in voltage differential values occurs at temperatures over 30 °C. The reduction is attributable to elevated temperatures impacting the electrode paste, diminishing the Electrode's sensitivity to silver ions in the solution.

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The response time of the modified Electrode was assessed by immersing it adjacent to the calomel electrode

in a series of standard silver nitrate (AgNO_3) solutions with decreasing concentrations, commencing at 10^{-1} and concluding at 10^{-6} molar. The response time of the modified Electrode varied between 2-20 seconds, indicating that response time is inversely correlated with ion concentration in the solution. As the concentration of ions in the solution rises, the reaction time of the Electrode appears to diminish, resulting in an extended duration to achieve equilibrium at the Electrode. ²⁷

The lifespan of the Electrode was discovered by measuring the potential difference within 90 days using a standard AgNO_3 solution at a concentration of 1×10^{-2} molar. The results proved that the measured voltage remained constant and stable throughout this duration. Consequently, the age of the Electrode was estimated to be 90 days. However, it was noted that after this period, the measured voltages started dropping, perhaps because of damage to the electrode paste or malfunction in the conduction of the copper wire. ²⁸

Table 2. The optimal conditions for R-AgONPs electrode

Parameter	modified carbon paste electrode
pH Effect	5-7
Temperature (C°)	15-30
Response Time (sec)	2-20
Lifetime (day)	95

3.3. Calibration Curve

The modified carbon electrode was immersed beside a calomel electrode (reference electrode) in standard diluted silver nitrate solutions descending concentrations from 10^{-1} to 10^{-6} molar; the voltage difference was recorded five times per concentration at temperature 25 °C and calibration curve

using straight line equation, Figure10, this indicates the standard curve of the Electrode, proving that the linear range of the Nernstian response is between 10^{-1} to 10^{-6} molar. The correlation coefficient was 0.9701, and the Electrode's slope was detected at 58.3 mV/decade, approximating the real slope value of 59 mV/decade.²⁹

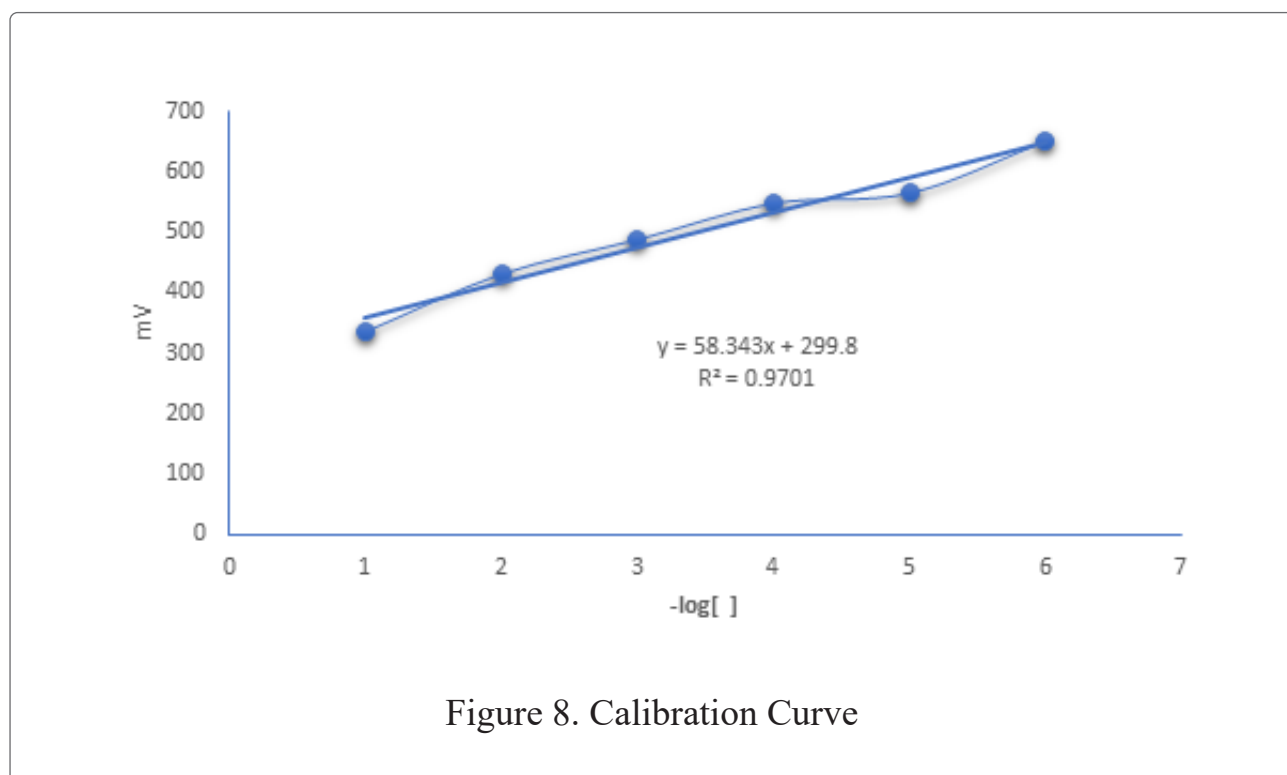
**Figure 8. Calibration Curve**

Table 3. The potentiometric properties of the modified carbon paste electrode

Properties	modified carbon paste electrode values
Linear range	$10^{-1} - 10^{-6}$ molar
Slope	58.3 mv/decade
Intercept	299.8 mv
Detection limit	7×10^{-6} molar
correlation coefficient	0.9701

3.4. Accuracy and Precision.

Accuracy and precision were studied using several concentrations within the standard electrode calibration curve range. After stabilising the optimal conditions, the voltage was measured individually and sequentially for

each concentration. Temperature: 25 °C; pH: 7. The accuracy and precision results show that the method works well in estimating silver ions, has a low standard deviation, and is highly retrievable. The results are shown in Table 4.³⁰

Table 4. Accuracy and Precision.

Conc. Taken (mole\L)	Conc. Found (mole\L)	Rec%	RSD%
1×10^{-2}	0.9923×10^{-2}	99.246	0.7112
1×10^{-4}	0.9237×10^{-4}	98.032	0.3280
1×10^{-6}	0.9883×10^{-6}	99.336	0.4904

3.5. Selectivity

The selectivity coefficient was determined utilizing Equation 1. The selectivity coefficient denotes an electrode's capacity to identify a specific ion amidst other ions. The impact of interferences on the Electrode's response

was examined by the mixed solutions approach, in which varying amounts of different salts were included in a 10^{-2} M silver nitrate solution.⁽³¹⁾

$$\log K_{A,B}^{\text{pot}} = \frac{E_2 - E_1}{S} \dots \dots \dots (1)$$

Table 5. Selectivity coefficient values

Selectivity Coefficient	Interference Ion Concentration	Type of interference ion
0.039576632	10^{-2}M	CO_3^{-2}
0.318839765	10^{-4}M	
0.069585443	10^{-2}M	Fe^{+3}
0.075674419	10^{-4}M	
0.206813982	10^{-2}M	Co^{+3}
0.089865412	10^{-4}M	
0.138734983	10^{-2}M	Cd^{+2}
0.057612982	10^{-4}M	
0.289862762	10^{-2}M	SO_4^{-2}
0.046728763	10^{-4}M	

3.6. Applications

Silver ions were estimated by sampling water from different regions, such as water and industrial Water from various places in the Tikrit governorate and X-ray film residues. The samples were measured immediately after sta-

bilizing the optimal electrode pH 6 and temperature 25 °C. The voltage was offset in the silver electrode curve equation, and the concentration was measured for each sample, as shown in the table below.

Table 6. Direct Method Result

Sample area	Taken Conc	Found Conc	Rec %	RSD %
Well water from Tikrit	1×10^{-2}	0.9954×10^{-2}	99.54	0.7179
Industrial water from Tikrit	1×10^{-4}	98.032×10^{-4}	98.032	3.2804
Industrial water from Tikrit	1×10^{-6}	99.336×10^{-6}	99.336	0.4904

The accuracy and tuning findings demonstrate that the used approach is highly retrievable, has a low standard deviation, and effectively estimates sil-

ver ions in industrial wells and waters. It is straightforward, cost-effective, and ecologically sustainable.

4. Conclusions.

This study involved the synthesis of silver nanoparticles with arugula leaf extract through a green chemistry approach. The improved carbon electrode was synthesized by directly combining silver nanoparticles with carbon powder, which enhanced direct sensitization and produced an electrically conductive composite. Scanning electron microscopy images demonstrate that the dimensions and morphology of silver nanoparticles may be regulated by modifying temperature and medium, yielding semi-spherical silver nanoparticles with an average particle size of 15.2733 nm. The XRD pattern indicated that applying arugula leaf extract diminished the intensity of the peaks corresponding to silver and silver nanoparticles within the identical crystal structure. Silver nanoparticles (AgNPs) combined with carbon powder were utilized to fabricate a selective electrode for silver ions. The incorporation of silver nanoparticles with carbon powder resulted in distinctive characteristics of the selective Electrode, including a low detection limit of 10^{-6} M, a rapid response time of 2-20 seconds, a well-defined linear range of

10^{-1} - 10^{-6} M, an extended electrode lifespan 90 days, and stability and resistance of the electrode-forming paste. The Electrode was effectively utilized on several contaminated water samples, yielding excellent findings.

5. Conflict of Interest

The authors declare that there is no conflict of interest regarding the publication of this manuscript.

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