

Preparation of silver nanoparticles using microwave plasma jet and a study of their physicochemical properties and applications in photocatalysis

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ABSTRACT

Microwave plasma is considered a crucial engineering technology and an effective method for preparing silver nanoparticles (AgNPs) with controllable properties, thereby opening up numerous possibilities for their application in environmental remediation and water treatment technologies. The structural and optical characteristics of the prepared AgNPs, as well as their photocatalytic activity toward methylene blue dye degradation, were investigated. AgNPs were prepared using a microwave plasma system at operating voltages of 150, 160, and 170 V. Plasma diagnostics based on the Boltzmann plot revealed that increasing the applied voltage gradually increased the electron temperature (T_e) from 4.02 to 6.13 eV. UV–Vis analysis confirmed the formation of silver nanoparticles through the appearance of the characteristic absorption band in the 290–300 nm range. X-ray diffraction (XRD) results revealed the formation of pure metallic silver (Ag) phases at 150 and 160 V, while mixed phases of silver and silver oxide (Ag/AgO) were observed at 170 V due to increased plasma activity and partial surface oxidation of the particles. Additionally, the average crystallite sizes of 18.7 nm at 150 V, 12 nm at 160 V, and 11.6 nm at 170 V indicate an improvement in crystallinity and a reduction in crystallite size. The results showed that 160 V achieved the best efficiency in dye degradation, recording the highest kinetic rate constant ($k = 0.0602 \text{ min}^{-1}$) compared to 150 V and 170 V, indicating that this voltage provided a better balance between particle size, activity, and effective surface area, with the highest photodegradation efficiency of 97.5%. These results confirm that the microwave plasma system at atmospheric pressure is an effective and controllable engineering technique for producing highly efficient silver nanoparticles, making it promising for photocatalytic applications and the treatment of organic pollutants in water.

Keywords: microwave plasma jet, photocatalysis, Ag nanoparticles, methylene blue (MB), electron temperature.

INTRODUCTION

Pigment pollutants, especially synthetic dyes such as methylene blue (MB) from the textile and dyeing industries, are considered significant environmental pollutants because of their chemical stability and resistance to biodegradation, which allows them to persist for long periods in water systems. Discharging wastewater containing these dyes without proper treatment can change the physical and chemical properties of water. Even at low concentrations, their presence can

reduce light penetration in water, limiting photosynthesis for aquatic organisms and affecting oxygen production and ecosystem balance. Additionally, some dyes and their breakdown products may have toxic, mutagenic, or carcinogenic properties, and they can accumulate in sediments as well as aquatic organisms, moving through the food chain, posing a threat to biodiversity and human health. Therefore, removing these pollutants from wastewater before discharging it into the environment has become an environmental necessity, which has driven the development of

effective and sustainable treatment technologies to remove dyes and convert them into less hazardous products [1,2].

MB is a cationic organic dye that belongs to the thiazine dye group. It is a colored compound with a conjugated aromatic structure, which gives it a high ability to absorb light in the visible region due to its conjugated π -electron system. The dye has the molecular formula $C_{16}H_{18}ClN_3S$ and consists of an organic structure containing carbon, hydrogen, nitrogen, and sulfur atoms, along with a chloride ion as the counterion. MB is characterized as a cationic phenothiazine dye that is water-soluble and can be reduced and oxidized, and vice versa, due to its connection with a dimethylamino group [3]. It is widely used as a model compound to evaluate the efficiency of photocatalysts in water treatment and the removal of organic pollutants. Its photodegradation process relies on generating reactive species such as hydroxyl radicals (OH) and reactive oxygen species (O_2), which break down the organic bonds in dye molecules and convert them into simpler products such as carbon dioxide and water [4,5].

Industrial effluents frequently contain persistent organic dyes such as MB, which significantly hinder light penetration and disrupt aquatic ecosystems. Due to their catalytic and antimicrobial characteristics, AgNPs have demonstrated strong potential for mitigating such pollutants, offering an effective strategy for wastewater treatment and environmental remediation [6].

Silver nanoparticles have a lot of benefits, including their ability to break down accumulated organic pollutants that originate from non-biodegradable industrial waste. The accumulation and persistence of these pollutants in water negatively affect aquatic life. One of the pollutants used as a dye is MB. Thus, it is important to treat this waste to protect the environment [7]. Nanoparticles possess distinctive chemical, physical, and morphological features that differentiate them from bulk counterparts, primarily due to quantum confinement and enhanced surface effects at the nanoscale. These attributes enable precise tuning of electrical, optical, and magnetic properties through controlled manipulation of particle size and shape [8]. AgNPs have attracted considerable attention owing to their diverse applications in sensing and catalysis, optoelectronic, and surface-enhanced Raman scattering, as well as in environmental and biomedical fields [9]. Studies have shown that silver nanoparticles have unique

functional properties due to their high surface activity, which has made them useful in developing materials with multiple applications. Kowalik and others prepared silver nanoparticles and combined them with a PVC matrix to create materials with antibacterial properties, highlighting the importance of controlling AgNPs' properties and using them in environmental and biological applications [10,11]. Based on these properties, using AgNPs prepared by eco-friendly methods, such as plasma techniques, has become a promising area for developing effective nanocatalysts to remove organic pollutants, including breaking down methylene blue dye through photocatalysis [12].

Among the different techniques available for preparing nanomaterials, such as chemical reduction, microwave plasma has emerged as a promising method [13], hydrothermal methods [14], sol-gel [15], lasers [16], and biosensor [17], each has its own limitations related to long preparation times, the need for high temperatures and pressures, or the use of large amounts of reducing and stabilizing chemicals, which can affect the purity of the final product and increase the environmental impact of the process [18]. On the other hand, this technology is notable for combining engineering efficiency with environmental sustainability, providing stable, high-density plasma at atmospheric pressure, with the ability to precisely control operating parameters like discharge voltage, gas type, and flow rate [19-22]. This technology stands out for its ease of integration into continuous production lines, as it can be developed for industrial applications, making it more suitable for producing nanomaterials used in photocatalysis, water treatment, and functional coatings [23,24]. Therefore, this technology is considered one of the advanced engineering techniques in the field of nanomaterial manufacturing and surface treatment. The engineering importance of the microwave plasma system lies in its design, which achieves efficient energy distribution and high plasma stability, providing a controllable reaction environment for producing nanomaterials with improved properties. This design also opens the door for developing integrated and scalable manufacturing systems, in line with the needs of modern industrial and environmental applications [25-28].

Microwave plasma at atmospheric pressure has several advantages over traditional methods for preparing nanomaterials, mainly ease of use,

low cost, quick generation, and safety. Additionally, it offers a non-thermal environment full of high-energy electrons and reactive species like ions, free radicals, and reactive oxygen and nitrogen species (ROS/RNS), which help in reduction and chemical conversion processes without the need for added reducing agents or external stabilizers [29,30]. Mokrzycka et al. showed that concluded that plasma parameters are a key factor in controlling the microstructure and surface properties of treated materials, highlighting the importance of adjusting plasma conditions to get the best performance from the material [31].

The microwave plasma jet technique was adopted in the current study for preparing AgNPs because most other techniques rely on reducing agents and chemical stabilizers, which can affect the purity of the molecules, increase environmental impact, and complicate preparation steps. This is the main difference from previous studies. On the other hand, microwave plasma jets at atmospheric pressure provide a reactive environment rich in high-energy electrons and reactive species like oxygen and nitrogen (ROS and RNS), which help activate the surface of the material and enhance conversion and reaction processes without the need for additional chemicals. Moreover, this technique produces stable plasma with high electron density, allowing the rapid and efficient preparation of silver nanoparticles, with the ability to control their properties by changing the plasma voltage. All of this has been confirmed through various tests, such as plasma spectrum, x-ray diffraction (XRD), field emission scanning electron microscopy FESEM, energy-dispersive x-ray spectroscopy (EDS), UV-visible, and by applying it to methylene blue dye to confirm the reactivity of particles in breaking down the dye.

MATERIALS AND METHODS

Experimental setup

The microwave plasma system consists of several main components that work together to produce a stable and efficient plasma jet, as shown in Figure 1. The system starts with the magnetron, which is the primary source of microwave waves at a frequency of 2.45 GHz, converting electrical energy into high-frequency electromagnetic waves. These waves travel through the

waveguide, while the splitter protects the magnetron from reflected energy caused by impedance mismatch. The three-pin tuner is used to adjust the impedance and ensure maximum energy transfer from the magnetron to the discharge area through a copper electrode, which guarantees the formation of a very stable and effective plasma. When the microwaves reach the quartz tube containing argon gas, which was set to flow at 20 L/min throughout the experiment, the oscillating electromagnetic fields interact with the free electrons in the gas, giving them high kinetic energy and causing them to collide with gas atoms. A quartz tube of 30 cm in length, with an inner diameter of 0.5 cm and an outer diameter of 0.8 cm, was used. The distance between the quartz tube outlet and the sample surface was set at 2mm to ensure the maximum amount of active species reaches the sample. These collisions lead to ionization and excitation processes, resulting in the formation of plasma that contains electrons, ions, excited particles, and various reactive species. Microwave plasma is characterized by having electrons with high energy compared to the gas temperature, which makes it effective in driving physical and chemical reactions without needing to significantly raise the temperature of the medium. When the plasma jet hits the water surface, containing micron-sized silver particles, high-energy electrons and reactive species generated from the plasma transfer into the liquid, leading to the formation of free radicals and reactive oxygen and hydrogen species according to [32]. These species attack the surface of the silver microparticles and gradually break them down, while also releasing silver atoms and ions into the solution. At the same time, the high-energy electrons and solvated electrons formed in the water help reduce the silver ions into neutral silver atoms, which then cluster together to form very small nuclei. As the exposure to the plasma continues, these nuclei grow to form stable silver nanoparticles. Because the electron temperature is one of the most important plasma parameters in ionization and excitation processes, it was calculated using optical emission spectroscopy (OES) at different voltages. Increasing the electron temperature leads to a higher density of active electrons and more effective collisions, which in turn increases the rate of nanocluster formation and improves the efficiency of the preparation process, agreeing with [33].

Diagnostics plasma and nanoparticle characterization

Optical emission spectroscopy was chosen as a diagnostic tool to understand the plasma environment and identify the nature of reactive species generated during the discharge process. Measurements were carried out using an Ocean Optics Flame-SXR1 model with an optical resolution of 1.69 nm, manufactured by ocean optics spectrometer (USA), with the fiber optic probe placed about 1 cm from the plasma jet to obtain stable and representative emission signals. The spectrometer was connected to the fiber near the sample, with the other end attached to a computer to display the plasma spectrum on the screen and to calculate the electron temperature using NIST software [34]. The optical response of the resulting silver particles was checked using UV-visible spectroscopy to evaluate their light absorption behavior as well as the electronic transitions caused by argon gas.

Synthesis of silver nanoparticles

Initially, 0.1 g of micro silver powder was placed in a beaker containing 10 mL of double-distilled water. The suspension was exposed to a microwave plasma jet at three different applied voltages (150, 160, and 170 V) for just one minute and a flow rate 20L/min, to convert the micro silver particles into silver nanoparticles in a quick, simple, and low-cost way. After obtaining the

colloidal solution containing the silver Nano and micro particles, the colloidal solution was centrifuged at 5000 rpm for 15 minutes to separate the larger particles. This caused the micro and denser particles to settle at the bottom of the tube, while the nanoparticles stayed suspended at the top of the solution (supernatant). Then, the top layer was carefully collected and used for subsequent tests and measurements. Then divided into two parts. The first part was dedicated to characterization and testing, where about 0.025 mL of the solution was deposited onto a glass slide measuring $1 \times 1 \text{ cm}^2$, and then left to dry in preparation for structural, morphological, and optical tests such as XRD, FESEM, EDS, and UV-Vis. These tests were carried out to verify the formation of the nanoparticles and to study their size, shape, and physical properties. The second part of the colloidal solution was used in photocatalysis applications to check how effective the prepared silver nanoparticles are at catalyzing chemical reactions under light (sun-light), and to see if they could be used for pollutant treatment and boosting catalytic performance.

Preparing the methylation dye and the mixture

A MB dye solution was prepared by dissolving 0.01 g of the dye powder in 1000 mL of distilled water. The solution was stirred using a magnetic stirrer for 30 minutes to ensure the dye fully dissolved and to obtain a homogeneous solution. After that, the solution was stored in

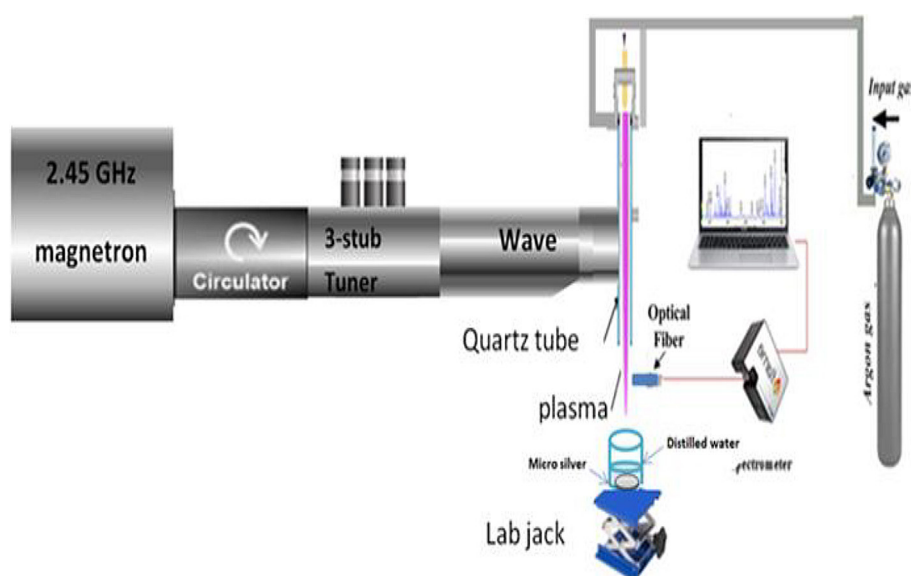


Figure 1. Schematic of atmospheric microwave-plasma jet system for preparation AgNPs at 150, 160, 170 V

a dark glass container or kept away from direct light to prevent any potential photo degradation before conducting the experiments. To test the photocatalytic efficiency, 10 ml of the colloidal solution containing plasma-prepared silver nanoparticles was added to 90 ml of methylene blue solution, and then the mixture was stirred magnetically for 45 minutes to ensure even distribution of the nanoparticles throughout the solution. After that, the mixture was left in the dark for one hour to reach equilibrium between adsorption and desorption on the surface of the nanoparticles. Then, the solution was exposed to sunlight, with 100 ml of the mixture prepared for each voltage used in the microwave plasma system (150, 160, and 170 V). The samples were placed in separate tubes and exposed to sunlight for 15, 30, 45, and 60 minutes, using the initial absorbance of the MB–AgNPs mixture measured before sunlight exposure as the reference value for calculating the degradation efficiency. The photocatalytic degradation of the methylene blue dye was monitored using UV–Vis spectroscopy at designated times, and the results of the treated samples were compared with the reference value to evaluate photocatalytic efficiency.

The degradation efficiency was calculated using Equation 1 [35],

$$D\% = \frac{A_0 - A_t}{A_0} \times 100\% \quad (1)$$

The kinetic parameters of MB dye photodegradation were determined using the pseudo-first-order kinetic equation:

$$\ln \frac{A_t}{A_0} = -kt \quad (2)$$

where: A_0 is the initial absorbance, A_t is the absorbance at time t , and k is the rate constant.

RESULTS AND DISCUSSION

Optical emission spectrum characterization

The electron temperature is considered one of the most important physical parameters that control the properties of plasma and its efficiency in preparing nanomaterials, as it directly affects the energy of electrons and their ability to cause ionization, excitation, and the formation of active

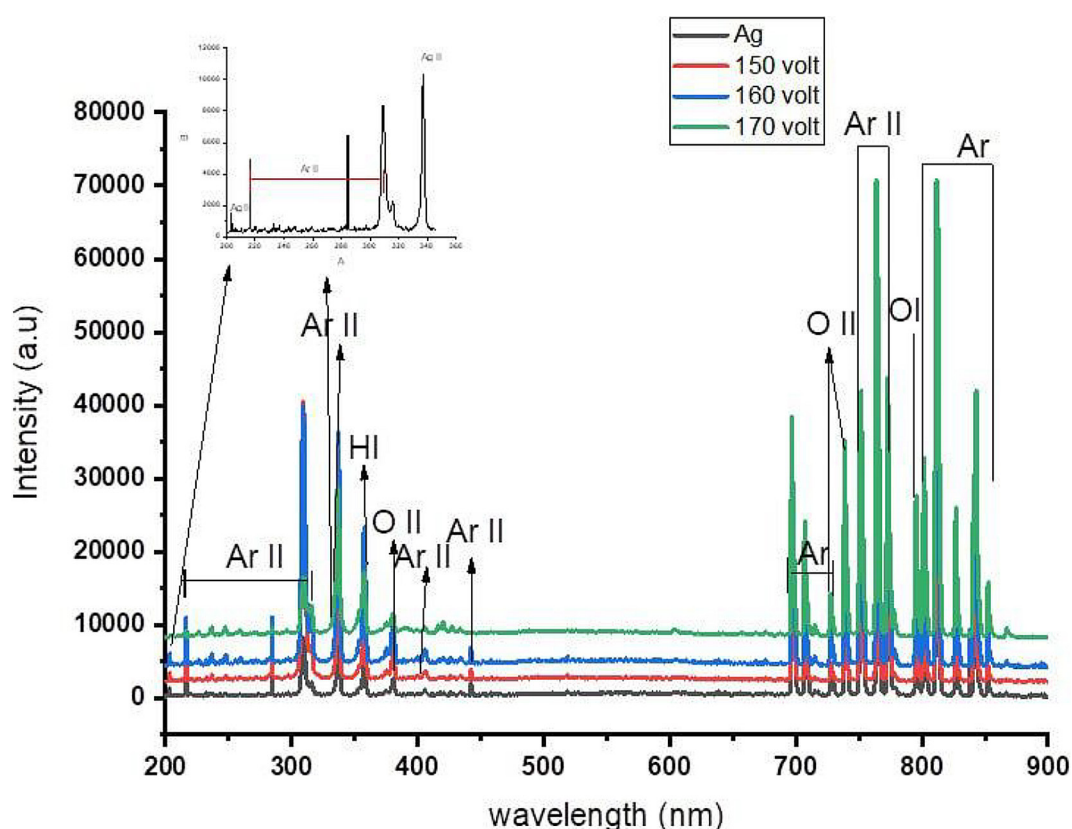
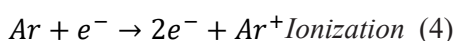
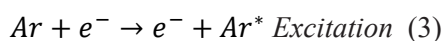


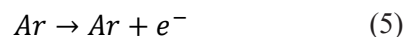
Figure 2. Emission spectra of atmospheric pressure microwave-plasma at 150, 160, and 170 V

species within the medium [36]. Therefore, the electron temperature was calculated at different voltages using OES to study its impact on the process of converting micro-sized silver particles into nanoparticles. Increasing the temperature of electrons leads to a higher number of high-energy electrons, free radicals, and reactive species generated from the plasma, which promotes the corrosion and peeling of the surface of micro-sized silver particles and the release of silver atoms and ions into the liquid medium. These electrons also help reduce silver ions and redeposit them as small nanonuclei, which later grow into stable silver nanoparticles. Therefore, studying the electron temperature at each voltage gives a deeper understanding of the mechanism behind nanoparticle formation and helps identify the optimal operating conditions to obtain smaller particles with a more uniform size distribution [37,38].

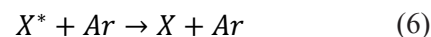
To determine the reactive excited species that were created during the discharge of plasma, with and without an Ag sample present, the OES data were obtained. The OES spectra are displayed in Figure 2. The plasma was produced at three applied voltages (150, 160, and 170 volts), and the tip of the optical fiber was placed 1 cm away from the plasma. Because oxygen was present in the air when the plasma was created at atmospheric pressure, the argon and oxygen species predominated in the spectrum. The primary species of argon Ar lines were detected between 216 and 312 nm from 696 to 900 nm, and the highest peak of Ar can be seen in 763.82 nm ($3s^23p^4(^3P)3d$) from all cases. Moreover, the highest peak of oxygen emission lines $2s^22p^2(^3P)3d$ is present in 309.13nm. In addition, the H β peak is seen at wavelengths of 379 nm in each instance, signifying the presence of hydrogen. The Ag emission lines can be found at 203.33 and 337.2 nm. When plasma is generated and interacts with matter, several processes may occur simultaneously or sequentially, such as excitation, ionization, recombination, and others. These reactions can be estimated when relying on argon gas in plasma generation by using (OES) [39],



Under very special conditions (such as the presence of very low energies), an argon atom can capture an electron, but it is often unstable. This interaction is rare because argon does not favor the formation of negative ions:



The appearance of light in plasma results from the interaction of argon with neutral atoms, which excites them to higher energy levels, as in X, where X is the neutral atom.



Optical spectroscopy is very important for diagnosing the plasma spectrum and calculating its parameters. The electron temperature can be determined using the Two-Line Boltzmann Method [40]. to find the electron temperature (T_e) for three voltages applied to the plasma (150, 160, and 170 volts) under normal atmospheric pressure and at a room temperature of 25 °C. This method relies directly on the Boltzmann distribution, so it is used when the plasma is close to a state of local thermal equilibrium (LTE or at least partial LTE) [40],

$$\frac{I_1}{I_2} = \frac{g_1 A_1 \lambda_2}{g_2 A_2 \lambda_1} e^{\left(\frac{E_1 - E_2}{KT}\right)} \quad (7)$$

where: I_1, I_2 represent the intensity of the first and second peaks, the statistical weight of the first and second peaks g_1, g_2 , the possibilities of transformation are A_1, A_2 , the wavelength of the first and second lines are λ_1, λ_2 , K is Boltzmann's constant (1.38×10^{-23} J/K), and the excited state energy between the two levels are E_1, E_2 .

The electron temperatures (T_e) were determined to be 4.02 eV, 5.17 eV, and 6.13 eV at 150 V, 160 V, and 170 V, respectively.

Increasing the operating voltage of the microwave plasma system leads to a higher electric field density in the discharge area, which raises the average electron energy and electron temperature (T_e). As a result, the gas ionization efficiency and water molecule dissociation increase, producing larger amounts of reactive species, such as hydrated electrons (eaq^-), hydrogen atoms ($H^\cdot$), and hydroxyl radicals (OH). Hydrated electrons and hydrogen atoms act as strong reducing agents, reducing silver ions (Ag^+) to metallic silver atoms (AgO), which then aggregate to form silver nanoparticles. Additionally, the higher concentration of reactive species along with the increased electron temperature enhances the nucleation rate and speeds up the formation process, directly affecting particle size and distribution, crystallinity, and consequently impacting their optical properties and photocatalytic activity [41–43].

Structural characterization

Figure 3 shows that the x-ray diffraction (XRD) patterns of silver nanoparticles prepared at different plasma voltages (150, 160, and 170 V) are clearly affected by increasing the applied voltage. The samples prepared at 150 and 160 V showed sharp and high-intensity peaks at the crystal planes (111), (200), (220), and (311), agreement with the standard data card JCPDS Card No. 04-0783, which correspond to the face-centered cubic (FCC) structure of metallic silver, indicating that the reduction process was successful and that high-crystallinity silver nanoparticles were formed [44]. The slight increase in peak intensity at 160 V suggests that raising the microwave plasma voltage within this range increased electron temperature and the density of active species in the plasma, providing better conditions for crystal growth and improved regularity. On

the other hand, the pattern of diffraction for the sample prepared at 170 V changed noticeably, with the intensity of the metallic silver peaks dropping clearly and new peaks appearing that correspond to silver oxides. This indicates a partial transformation from the metallic phase to the oxidized phase. This behavior can be explained by the fact that the significant increase in plasma voltage generates larger amounts of reactive oxygen species, which accelerate the oxidation of the silver particle surfaces, reducing the proportion of metallic silver and leading to a mixture of silver and silver oxide. On the other hand, the X-ray diffraction pattern of the sample prepared at 170 V noticeably differed, as the intensity of the metallic silver peaks decreased compared to the samples prepared at 150 and 160 V, with new diffraction peaks appearing at the crystal planes (101), (012), (110), and (301), corresponding to the silver oxide (AgO) phase according to JCPDS

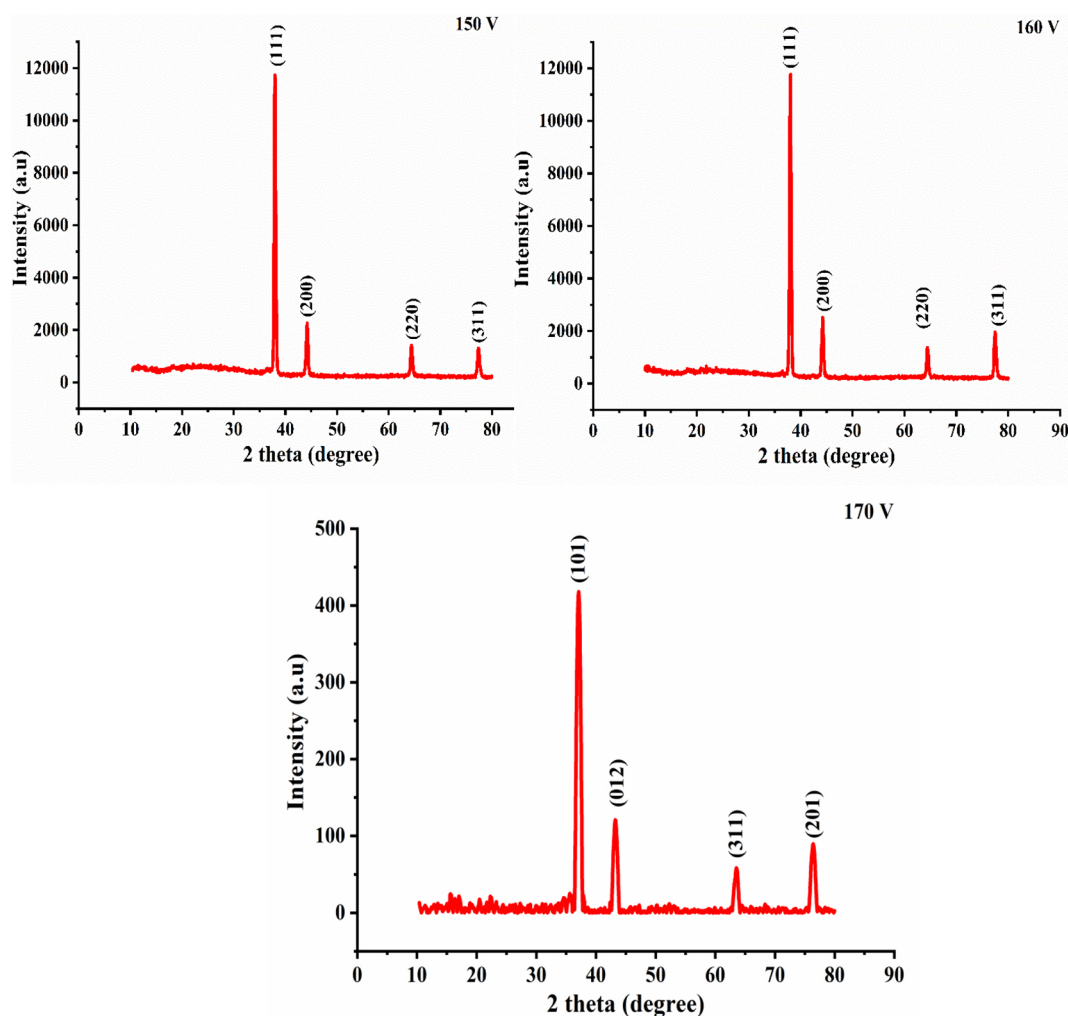


Figure 3. XRD spectra of Ag nanoparticles at different applied voltages (150, 160, and 170 V) using a microwave plasma jet

No. 41-1402. This indicates a partial transformation from the metallic silver phase to the oxidized phase due to the increased microwave plasma voltage. This behavior can be explained by the fact that increasing the voltage raises the electron density and energy transferred within the plasma, which boosts the generation of reactive oxygen species (ROS), such as oxygen atoms and free radicals, that react with the surface of the silver nanoparticles, leading to partial oxidation and the formation of the AgO phase. These results indicate that 160 volts is the optimal voltage for forming metallic silver nanoparticles, while increasing the voltage to 170 volts exceeded the optimal conditions and promoted oxidation processes, leading to a decrease in the intensity of the metallic silver peaks and the appearance of silver oxide peaks. This method stands out from others because it allows control over the crystalline structure of silver nanoparticles simply by changing the microwave plasma voltage, without using extra chemical reducing or stabilizing agents, making the method simpler and more environmentally friendly [45]. Scherrer Equation is used to estimate the average crystallite size of nanomaterials from X-ray diffraction (XRD) data [46].

$$\text{Crystallite size } D = \left(\frac{0.9\lambda}{\beta \cos \theta} \right) \quad (8)$$

Calculating the average crystal size (D) of silver nanoparticles using the Scherrer equation based on XRD data, where β represents the full width at half maximum (FWHM) of the peak, and λ is the wavelength of the X-rays used in the measurement. The results showed that the average crystal size ranged between 19.46–25.76 nm, 20.17–23.10 nm, and 9.71–12.17 nm for the samples prepared at operating voltages of 150, 160, and 170 V, respectively. It was observed that the crystal size significantly decreased at 170 V, which is attributed to the fact that increasing the microwave plasma voltage raised the density and energy of the reactive species in the plasma, leading to a higher nucleation rate compared to the crystal growth rate. As a result, a large number of nuclei formed in a short time, limiting crystal growth and resulting in smaller crystals. Also, the appearance of the AgO phase at this voltage, as shown by the XRD results, indicates that oxidation processes became more significant at 170 V, which could also contribute to limiting the growth of metallic crystals and reducing their size.[47]. In case additional weak

peaks appear, they can be attributed to silver monoxide AgO₂ or silver oxide AgO. The formation of these phases is due to the partial reaction of silver atoms with the oxygen present in the environment during the preparation or plasma treatment process.

Morphological characterization

Figure 4 shows FESEM images and the size distribution of the silver nanoparticles prepared using microwave plasma at different operating voltages (150, 160, and 170 V). All samples indicated that most particles were spherical, with no large aggregates observed on the glass surface, suggesting that the plasma preparation process provided suitable conditions for particle formation. This is attributed to the fact that the active species generated within the plasma contribute to forming a large number of crystal nuclei in a short time, which limits particle coalescence during growth. As shown by the ImageJ analysis results, the average particle size gradually decreased from 18.7 nm at 150 V to 12 nm at 160 V, and then to 11.6 nm at 170 V agree with [48].

This decrease can be explained by the fact that increasing the microwave plasma voltage raises the temperature of electrons and the density of active species, which increases the nucleation rate more than the crystal growth rate. As a result, more nuclei form in a short time, dispersing silver atoms over many growth centers, which limits the growth of each particle individually and leads to smaller and more uniform nanoparticles.

These findings are consistent with the X-ray diffraction (XRD) results, where 170 V showed a decrease in crystal size along with the appearance of AgO phase. This indicates that increasing plasma energy not only affects particle size but also alters their crystal structure due to enhanced oxidation processes. This shows that controlling the microwave plasma voltage is a key factor in determining both particle size and crystal structure compared to other methods.

Although the FESEM images showed similar average particle size between the two samples prepared at 160 and 170 volts, the XRD results showed a greater decrease in the crystal size at 170 volts. This is because FESEM measures the physical size of the particles, while the Scherrer equation reflects the size of the crystallite domains, and a single particle can consist of more than one crystallite domain, especially with the

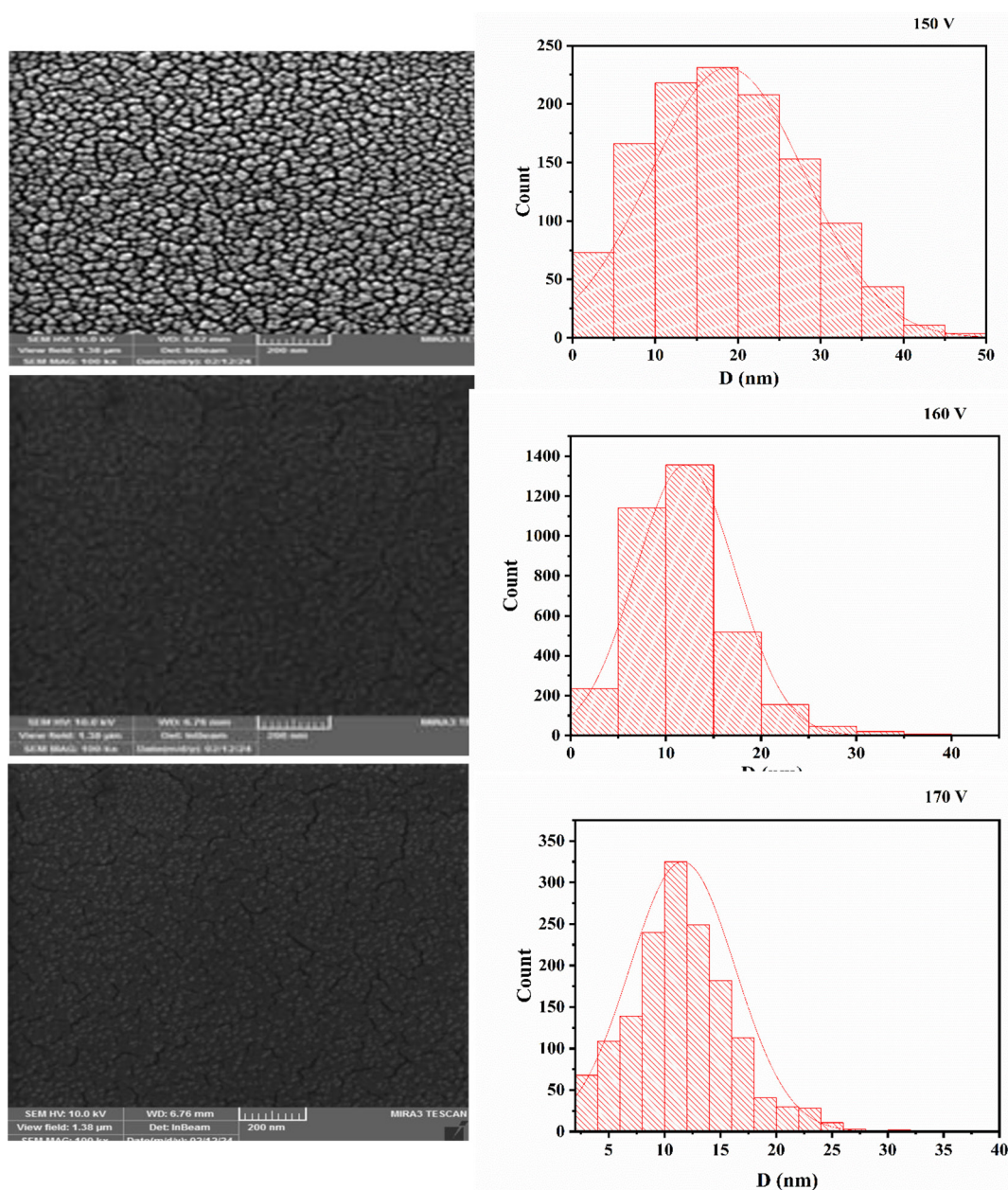


Figure 4. FESEM images and particle size distribution of Ag NPs at different applied voltages

beginning formation of the AgO phase at the higher voltage.

The elemental composition was determined by EDS, as shown in Figure 5. The first and second orbital shells represent any L and K atom. For silver atoms, the first shell represents AgL, where the X-ray peak causes electron transition or emission. Moreover, the lower peak is known as β (beta), and the higher peak is called α (alpha) [49]. EDS analysis shows that the weight percentages of silver are 85.87%, 44.94%, and 63.10% at 150, 160, and 170 volts respectively; carbon is 5.35%, 23.23%, and 19.63%; nitrogen is 4.16%, 7.22%, and 5.69%; and oxygen is 4.61%, 24.61%, and

11.59% at 150, 160, and 170 volts respectively. The binding energy peaks at 0.277, 0.3, and 2.621 keV correspond to $CK\alpha$, $ClK\alpha$, and $OK\alpha$, respectively, while those at 3.01 and 3.2 keV are associated with $AgL\alpha$.

UV-Visible measurements

The UV–visible absorption spectrum of the silver nanoparticles prepared using microwave plasma jets at different voltages (150, 160, and 170 V) shows absorption ranges roughly between 290–300 nm. It was observed that the absorption intensity was highest at 150 V, then gradually decreased

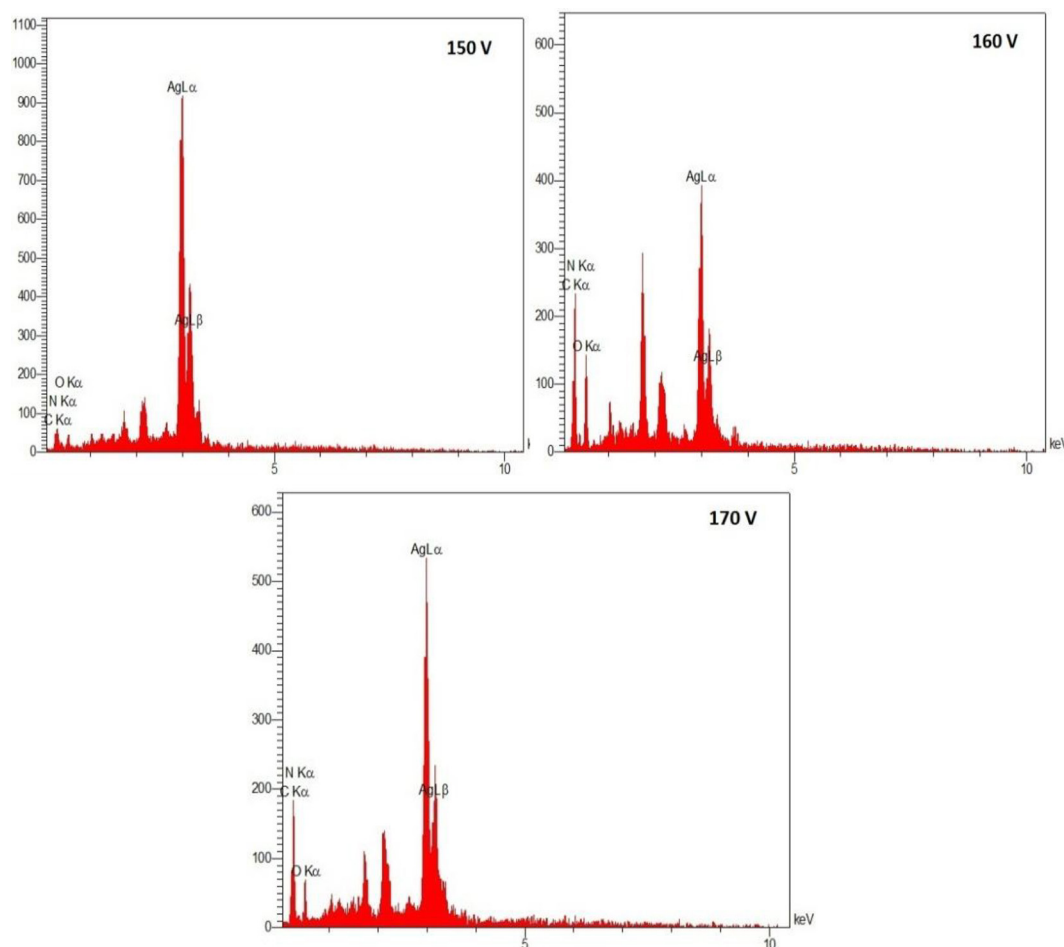


Figure 5. Energy-dispersive X-ray spectroscopy (EDS) analysis of silver nanoparticles

at 160 V, reaching its lowest value at 170 V. This behavior suggests that increasing the plasma voltage directly affects the formation mechanism of silver nanoparticles and their optical properties.

The decrease in absorption intensity at higher voltages can be explained by the increased plasma energy and higher electron temperature, which boosts the activity of reactive species within the plasma, influencing particle formation and surface growth processes. At 170 V, the high plasma energy might cause partial oxidation of metallic silver and the formation of AgO, along with the possibility of surface reorganization or partial aggregation of particles, reducing the concentration of metallic silver particles responsible for absorption [50]. This interpretation aligns with the results of XRD, as the samples prepared at 150 and 160 V showed peaks for metallic Ag, while additional phases related to silver oxide appeared at 170 V, confirming the effect of increased plasma energy on the crystalline structure of the particles. The results from the FESEM also support the change in particle size and distribution with

increasing voltage, due to the different formation and growth rates under varying plasma conditions. The blue shift of the absorption band at 290–300 nm can be attributed to the formation of extremely small silver nanoparticles/nanoclusters with a high surface-to-volume ratio, along with surface and surrounding medium effects, which lead to the optical response shifting towards shorter wavelengths.

Therefore, the variation in absorption intensity and peak position within the 290–300 nm range can be attributed to changes in particle size, distribution, and crystallinity, as well as changes in the ratio of metallic silver-to-silver oxide caused by the increased microwave plasma energy [51].

Photocatalytic activity measurement

Silver nanoparticles made using the microwave plasma method were studied as photocatalysts for breaking down methylene blue dye under natural sunlight. Methylene blue is a typical cationic organic dye used to assess the efficiency of

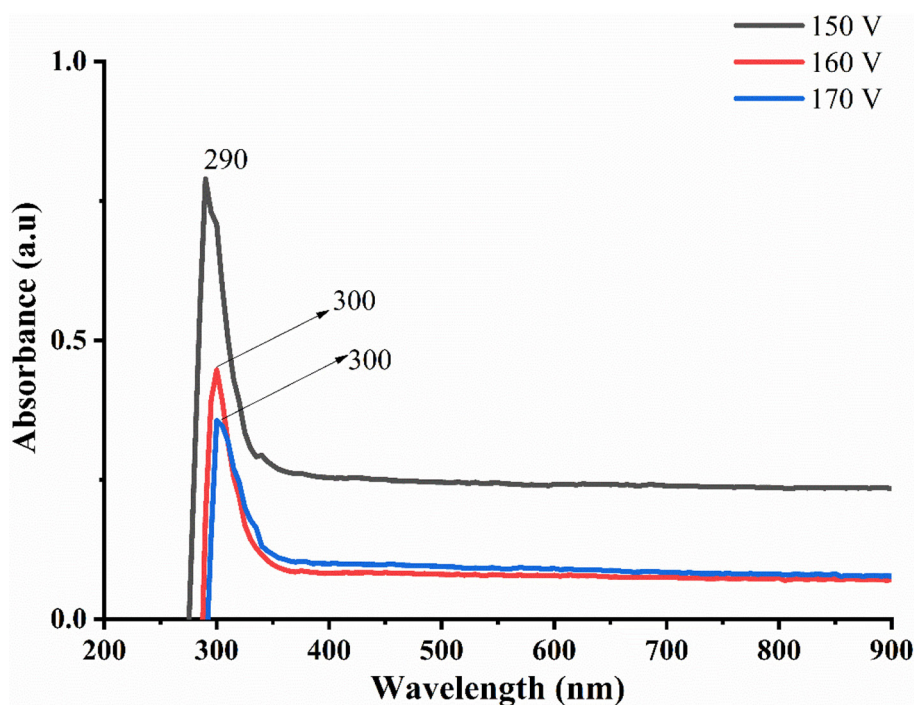
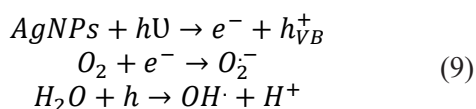
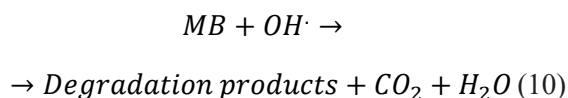


Figure 6. UV–Visible spectra of Ag NPs at different applied voltages

photocatalysts, which is why it was chosen in the research on silver nanoparticles. In photocatalysis using Ag nanoparticles, the decomposition of methylene blue occurs through the reactive species generated by the catalyst [52,53]. Figure 7 shows the stage of dye degradation after adding the silver nanoparticles and exposing it to sunlight.



Free radicals break down the methylene blue molecule:



During sun exposure, a gradual change in the color of the methylene blue solution from dark blue to a pale color was observed as the irradiation time increased, as shown in Figure 8. UV–Vis measurements confirmed this behavior through the continuous decrease in the intensity of the main absorption band of the dye at 661.5 nm, indicating the breakdown of MB molecules due to the photocatalytic reaction in the presence of silver nanoparticles. The gradual decrease in absorption intensity with increasing exposure time reflects the reduction in dye concentration in the solution, demonstrating high efficiency

of the photocatalytic degradation process. The MB degradation efficiencies were about 96.9%, 97.5%, and 94.6% for particles prepared at 150, 160, and 170 V, respectively. The sample prepared at 160 V showed the highest photocatalytic activity, which can be attributed to obtaining optimal plasma conditions that led to the formation of silver particles with suitable surface and crystalline properties, providing a greater number of active sites for catalytic reactions. The relative decrease in efficiency at 170 V may be related to increased plasma energy that could lead to the formation of silver oxide or changes in particle size distribution, as supported by the XRD and FESEM results. The degradation rate of methylene blue increased with longer exposure to sunlight due to the ongoing photocatalytic reactions on the surface of the silver nanoparticles. The kinetics study showed that the MB degradation followed a pseudo-first-order kinetic model, as the linear relationship between $\ln(A_0/A_t)$ and irradiation time fit this model well [35,54]. The sample prepared at 160 V showed the highest apparent rate constant ($k = 0.0602 \text{ min}^{-1}$), indicating it had the highest catalytic activity compared to the samples prepared at 150 and 170 V. This can be explained by the fact that 160 volts provided suitable plasma conditions that led to the formation of silver nanoparticles with improved surface and crystalline

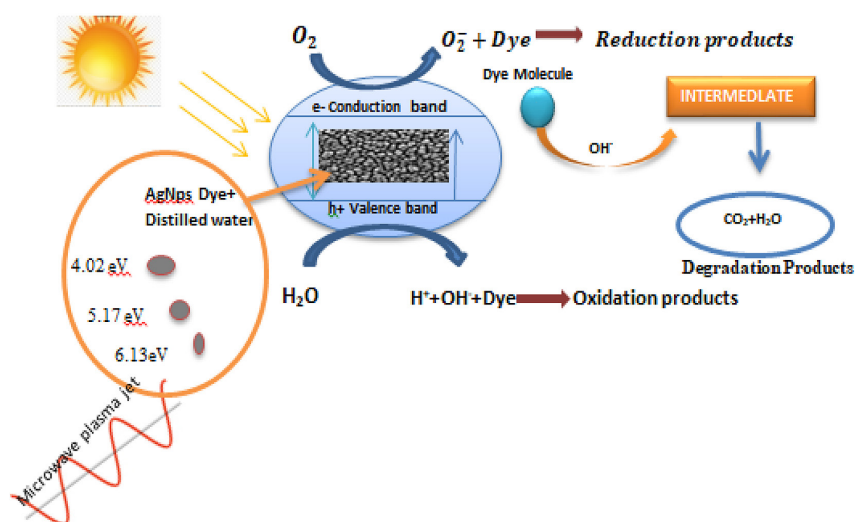


Figure 7. Diagram showing the process of photodegradation of methylene blue dye using silver nanoparticles prepared by microwave plasma at operating voltages of 150, 160, and 170 V

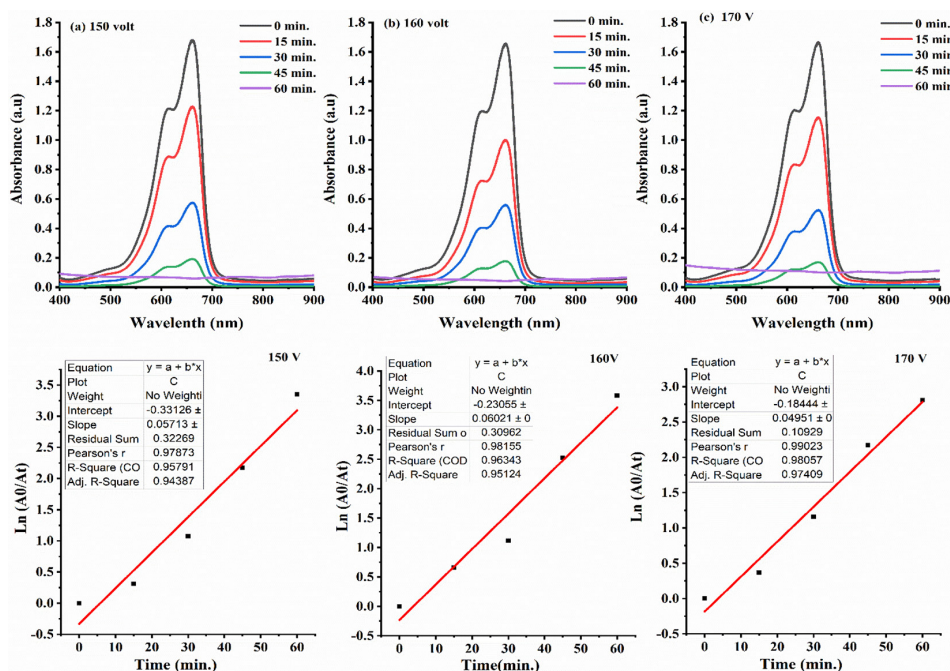


Figure 8. UV-Vis photocatalytic absorption spectrum and first-order kinetic plot of methylene blue (MB) dye using silver nanoparticles synthesized at (a) 150 V, (b) 160 V, (c) 170 V, and showing the color change of the MB solution during the photodegradation process

properties, increasing the number of active sites capable of generating reactive oxidative species responsible for MB degradation. On the other hand, increasing the voltage to 170 V caused a slight decrease in catalytic activity, which might be related to changes in the particle surface composition, such as the formation of AgO or changes in size distribution, as confirmed by XRD and FESEM results.

Table 1. Comparison of the degradation rate constant (k) values and the determination coefficient (R²) for dye degradation using silver nanoparticles prepared at microwave plasma voltages of 150, 160, and 170 V

Microwave plasma voltage	K (min) ⁻¹	R ²
150	0.0571	0.9579
160	0.0602	0.9634
170	0.0495	0.9806

Table 2. Comparison of photocatalytic degradation of MB using AgNPs

Reference	Method of synthesis	Degradation %	Time	Remarks
Vanaja et al.[55]	Chemical/biological reduction	95%	72 h	Long reaction time
Roy et al.[56]	Biosynthesis	93%	6 h	High efficiency but Long reaction time
Sanket et al.[57]	Green synthesis	89%	48–72 h	Long reaction time
I-Zaban et al. [58]	Biosynthesis	92%	72 h	Long reaction time
Mechouche et al.[59]	Biosynthesis	71.3%	6 h	Low degradation efficiency
Quoc-An Trieu et al.[60]	Green synthesis	95%	72 h	Long reaction time
Present work	Microwave plasma	97.5%	60 min.	High efficiency

X-ray diffraction results confirmed the presence of two phases: Ag and AgO, indicating that increasing plasma energy helped form effective interfaces between them. These interfaces act as key centers for separating photo-generated charges and reducing electron-hole recombination, which positively affects the production of free radicals like ($\bullet OH$) and ($\bullet O_2^-$) and improves the photocatalytic efficiency for methylene blue degradation. Therefore, it can be concluded that an electron temperature of 5.17 eV played a major role in enhancing the structural and surface properties of the particles, boosting their photocatalytic effect. To illustrate this kinetic behavior more clearly, the linear relationship between $\ln(A_0/A_t)$ and processing time was plotted, as shown in the following figure.

Here, k represents the apparent reaction rate constant in units of min^{-1} . The value of k was determined from the slope of the straight line obtained by plotting $\ln(A_0/A_t)$ versus processing time. The coefficient of determination R^2 was also used to assess how well the experimental data fit the kinetic model used. The values of the rate constants and determination coefficients at different plasma voltages are shown in Table 1

The results show that the value of the decomposition rate constant k varied depending on the microwave plasma voltage used in preparing the silver nanoparticles. The k value was 0.0571 min^{-1} at 150 V, and increased to 0.0602 min^{-1} at 160 V, indicating that the silver nanoparticles prepared at 160 V had the highest dye degradation rate under the experimental conditions. When the voltage was raised to 170 V, the rate constant decreased to 0.0495 min^{-1} . Despite this variation in decomposition rates, all experiments showed high values for the determination coefficient R^2 , ranging from 0.9579 to 0.9806, indicating good agreement between the experimental data and the pseudo-first-order kinetic model.

To highlight the efficiency of the nanoparticles prepared in this study, the results for the photodegradation of MB dye were compared with those of previous studies using silver nanoparticles (AgNPs) prepared by different methods. The comparison includes the preparation method, operating conditions, dye removal efficiency, and degradation time to show where the current results stand relative to published work and to demonstrate the effectiveness of using microwave plasma in enhancing photocatalytic performance, as shown in Table 2.

CONCLUSIONS

The results of this study showed that the microwave plasma system at atmospheric pressure is an effective and controllable engineering technique for preparing silver nanoparticles at different voltages without the need for chemical reducing or stabilizing agents. The results from UV–Vis spectroscopy confirmed the formation of silver nanoparticles in the range of 290–300 nm. XRD results showed the formation of pure Ag at 150 and 160 V, while at 170 V, mixed phases of silver and silver oxide (Ag/AgO) were observed, indicating partial oxidation of the particle surfaces due to increased plasma activity at the higher voltage. There was also a noticeable variation in crystal sizes, ranging from 11.6 to 18.7 nm. The microwave plasma-prepared silver nanoparticles showed photocatalytic efficiency using MB as a model pollutant. The silver particles demonstrated notable photocatalytic efficiency under sunlight at 160 V, reaching 97.5% within 60 minutes.

The scientific novelty of this study lies in developing a method that uses atmospheric-pressure microwave plasma to convert micro silver particles into nanoparticles without the need for any chemical reducing or stabilizing agents. The

results also showed that controlling the plasma voltage not only affects the structural and optical properties of the nanoparticles, but also directly impacts their efficiency in photocatalytically breaking down methylene blue dye. These results highlight the potential of microwave plasma as an effective and environmentally friendly platform for producing high-performance nanomaterials for environmental pollutant treatment applications.

Acknowledgments

The authors acknowledge they thank the group of the Plasma Physics Lab. Mustansiriyah University, collage of science, Department of physics, Iraq Baghdad.

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