

RESEARCH ARTICLE

Toxic Dye Degradation Employing *Phoenix dactylifera* Seed Extract for the Green Synthesis of Silver Nanoparticles: Characterization and Application

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Received: 31 August 2024 | **Revised:** 4 November 2024 | **Accepted:** 12 November 2024

Review Editor: Mingying Yang

Funding: This work was supported by Taif University, TU-DSPP-2024-65.

Keywords: AgNPs | dyes | green synthesis | *Phoenix dactylifera* | photocatalytic degradation

ABSTRACT

This research highlights the facile green synthesis of silver nanoparticles (AgNPs) using *Phoenix dactylifera* seed extracts and its photocatalytic application for the degradation of toxic dyes. The AgNPs synthesis was confirmed by the appearance of its representative absorption peak at 416 nm in UV–visible absorption spectroscopy. Moreover, the reduction of silver ions to Ag was justified through Fourier transform infrared (FTIR) spectroscopy. X-ray diffraction pattern revealed crystalline AgNPs structure with particle size ranging from 5 to 15 nm calculated using the Debye–Scherrer equation. The rectangular-like structural morphology of synthesized AgNPs was observed in scanning electron micrographs. The as-synthesized AgNPs demonstrated higher photocatalytic activity for the degradation of malachite green (MG) and congo red (CR) followed by methylene blue (MB), and crystal violet (CV) under UV irradiation. In addition, rate constant (k) and percentage degradation were also calculated. The present study presents a facile green synthesis pathway and its potentially successful manipulation in the reduction of toxic dyes under the illumination of UV-light.

1 | Introduction

The date palm, or *Phoenix dactylifera* L., is a member of the Aceraceae family and is among the first known agricultural plants, having been domesticated between 5500 and 3000 BCE. For over 1400 years, *P. dactylifera* products have been a staple in the diets of Islamic countries. The “Ajwa” variety, prevalent

in Arab nations, has been extensively studied and utilized for its medicinal and pharmacological benefits (Liu et al. 2018). Ajwa date seeds are rich in proteins and crude fat, and they also contain significant amounts of total dietary fiber (TDF). They contain not only soluble dietary fiber (SDF) but also insoluble dietary fiber (IDF) content (Liu et al. 2020). Products derived from *P. dactylifera*, including its fruits, seeds, pollen, leaves, and

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Summary

- The facile green construction of nanoparticles of silver (AgNPs) using the extract of *Phoenix dactylifera*.
- XRD revealed crystalline AgNP structure with particle size ranging from 5 to 15 nm calculated using the Scherrer equation.
- The stability of bio-reduced AgNPs was analyzed using UV-spectroscopy.
- Results showed that AgNPs were stable even after 1 month.
- FTIR unfolded the functional groups responsible for reducing Ag^+ to AgNPs.

syrup, offer numerous benefits for both humans and animals (Zeng et al. 2020). The protective properties of *P. dactylifera* are thought to arise not only from its fiber, vitamins, and minerals but also from a wide array of plant secondary metabolites. The Ajwa date is abundant in phytochemicals like polyphenols, flavonoids, isoflavonoids, sterols, and lignin, all of which have effectively decreased blood cholesterol and reduced the chances of cardiovascular problems. Moreover, the polyphenolic proanthocyanidins, in conjunction with other phenolics, may act as free radical scavengers or heavy metal chelators, thereby helping to alleviate oxidative stress and inflammation (Zhang et al. 2018).

Nanotechnology is a diverse scientific domain that includes almost all scientific disciplines that is chemistry, physics, biology, colloidal science, and so forth for investigating materials at the nanoscale level (Shi et al. 2022). Recent advances in nanotechnology have unfolded many new research horizons in materials science. Nanotechnology-aided research in biotechnology (Munir et al. 2020), analytical science (Iqbal and Iqbal 2013), quantum dots (Bahadur et al. 2018), microbiology, (Bahadur et al. 2021) and many other disciplines, have paved the way for solving many critical problems and are vitally important in new technologies such as mechanics, optics (Irfan et al. 2021), biomedical sciences (Chan et al. 2017), chemical industry (Irfan et al. 2020; Hussain et al. 2019), optoelectronic devices (Abubshait et al. 2021), drug delivery (Saeedi et al. 2019), and photoelectrochemical applications (Iravani 2011). Nanoparticles are highly valued because of their exceptionally small size, and they also possess a large surface-to-volume ratio, which results in significant physical and chemical differences from their bulk counterparts of similar chemical configurations. These differences manifest in various properties, such as biological integrity, mechanical strength, steric behavior, melting point catalytic activity, thermal stability, optical absorption, and electrical conductivity (Iravani 2011; Li et al. 2018).

In the past decade, metal nanoparticles (MNPs) have garnered significant attention due to their unique properties particularly size and surface area, optical, electronic, sensing, catalytic, and antibacterial characteristics (Shoaib et al. 2021). Out of all noble metals, silver nanoparticles (AgNPs) stand out as a versatile element, historically used in the creation of coins and jewelry (Hambardzumyan et al. 2020). Silver nanoparticles are

primarily utilized in glassware, electric batteries, and ceramic pigments, as well as in medical devices for treating diseases such as HIV, cancer, diabetes, tuberculosis, and malaria (Ur Rahman et al. 2020). AgNPs also possess catalytic properties and can detect biological molecules. Currently, their applications in biolabeling and optical sensing are being extensively explored (Hamed and Shojaosadati 2019). Various chemical methods have been developed for synthesizing nanoparticles, many of which are commonly used to produce nanostructured materials. These methods include pyrolysis, chemical reduction, sol-gel processing, microemulsion, hydrothermal synthesis, polyol synthesis, physicochemical processes, chemical vapor deposition, electrochemical techniques, sonochemical methods, thermal decomposition, microwave-assisted synthesis, solvothermal approaches, photochemical reduction, photosynthesis, and continuous-flow methods (Hambardzumyan et al. 2020; Muthu and Priya 2017; Patil and Chandrasekaran 2020). Additionally, traditional chemical synthesis of MNPs is often energy-intensive and involves hazardous chemicals and reagents. The byproducts generated during these processes can be harmful to humans and detrimental to the environment. Consequently, the use of these nanoparticles is often restricted in biological applications (Khodadadi, Bordbar, and Nasrollahzadeh 2017).

Green synthesis of AgNPs offers several advantages such as it is a cost-effective, convenient single-step process and eco-friendly. This method does not require high pressure, harsh temperature conditions, or lethal chemicals, making it a more sustainable alternative (Robinson et al. 2020). Many scientists have stated the plant-mediated green synthesis of silver nanoparticles using various plants and their extracts from different parts of the plant, including roots (Behravan et al. 2019), stem (Uddin et al. 2021), bark (Burlacu et al. 2019), leaf (Goutam et al. 2018), fruit (Jayaprakash et al. 2017), bud (Lakhan et al. 2020), and latex (Kalaiselvi et al. 2019) as natural resources. The extracts from these plant parts contain various biomolecules, that help to reduce metal ions and stabilize nanoparticles, helping to achieve the desired shapes and sizes (Modi 2018; Jyoti and Singh 2016). In various studies, a huge number of medicinal plants such as *Ocimum tenuiflorum* (Singh et al. 2018), *Cassia auriculata* (Muthu and Priya 2017), *Pulicaria glutinosa* (Tahir et al. 2013), *Diospyros lotus* (Hamed and Shojaosadati 2019), *Ananas comosus* (Ahmad and Sharma 2012), *P. dactylifera* (Farhadi, Ajerloo, and Mohammadi 2017), *Capsicum annuum* (Li et al. 2007), *Argemone mexicana* (Singh et al. 2010), *Olea europaea* (Khalil et al. 2014), and many more have already been used to synthesize and stabilize metallic NPs, particularly biogenic AgNPs.

Dyes are synthetic organic compounds used for versatile applications (Habibi and Askari 2011). The textile industry uses a substantial number of synthetic dyes, accounting for about 60% of total dye production, to enhance the appearance and texture of fabrics. Additionally, approximately 15% of these dyes are wasted after their intended use (Gonawala and Mehta 2014). These dye compounds dissolve in water bodies at concentrations ranging from 10 to 200 mg/L, leading to substantial water pollution on a global scale (Jyoti and Singh 2016). Therefore, treating dye effluents from textile industries is essential for wastewater management. Since the hue of these effluents lowers sunlight penetration and dissolved oxygen in water bodies, their discharge into aquatic systems presents serious

environmental problems. Additionally, it can release toxic compounds through chemical or biological reactions, adversely affecting aquatic flora and fauna. Traditional physical–chemical and biological methods for reducing dye compounds are often ineffective, time-consuming, and challenging, especially at high effluent concentrations. In contrast, reductive degradation of dyes using nanomaterials offers a more promising solution due to their unique physicochemical properties (Sharma et al. 2018).

Hence, in this paper, AgNPs have been synthesized by the green and environmentally benign method using *P. dactylifera* seed extract as reducing-cum-stabilizing agent and investigated their photocatalytic activity for degradation of hazardous dyes (Figure 1) that is malachite green (MG), methylene blue (MB), congo red (CR), and crystal violet (CV) under the exposure of UV light.

2 | Materials and Methods

2.1 | Reagents

Silver nitrite (AgNO_3) was procured from Sigma-Aldrich Germany. MB, CV, and MG were purchased from Merck Germany, whereas CR was purchased from Riedel-de Haën. Furthermore, deionized water (DI) was used for the synthesis of AgNPs.

2.2 | Procedure

2.2.1 | Preparation of Ajwa Seed Powder

Ajwa date seed extract (from *P. dactylifera*) was utilized as both a reducing and stabilizing agent. The dates were purchased from Lahore, Pakistan, where they are commercially available. The seeds were carefully separated and washed multiple times with DI. After washing, the seeds were weighed and dried overnight in an oven at 60°C . Once dried, the seeds of the Ajwa were

ground to a fine powder using industrial-grade steel mortar and pestle.

2.2.2 | Preparation of Ajwa Aqueous Extract

Aqueous extract of *P. dactylifera* was prepared by taking 2g powdered Ajwa seeds in 100 mL distilled water and kept soaked for 24 h. The Ajwa seeds extract was filtered and the filtrate was stored at 4°C for the synthesis of AgNPs.

2.2.3 | Green Synthesis of Silver Nanoparticles

AgNPs were synthesized by combining 5 mL of 5 mM AgNO_3 with 0.5 mL of Ajwa aqueous extract in a 25 mL beaker and it is diluted to achieve a total volume of 10 mL. The solution was stirred for 2 h to ensure it became homogeneous. After this period, the yellow color of the solution was changed to brown, indicating the successful formation of colloidal AgNPs.

2.2.4 | Photocatalytic Degradation and Kinetic Studies

The photocatalytic activity of the newly synthesized AgNPs has been investigated by exposing 50 mL of suspension containing 15 ppm MG dye and 0.05 g AgNPs to UV-light. Prior to the light exposure, the mixture was stirred magnetically and kept in the dark for 30 min. The sample was then collected with syringe filter after a regular interval of 10 min. The photodegradation of the MG dye was monitored by recording absorption spectra using UV–visible spectrophotometer. Moreover, the percentage degradation of the MG dye was determined using the given equation (Raj et al. 2020):

$$\% \text{ Degradation} = \left(\frac{A_0 - A_t}{A_0} \right) \times 100 \quad (1)$$

where A_0 and A_t are the absorption of the sample at time zero and any time “ t ”. Furthermore, kinetic study was investigated

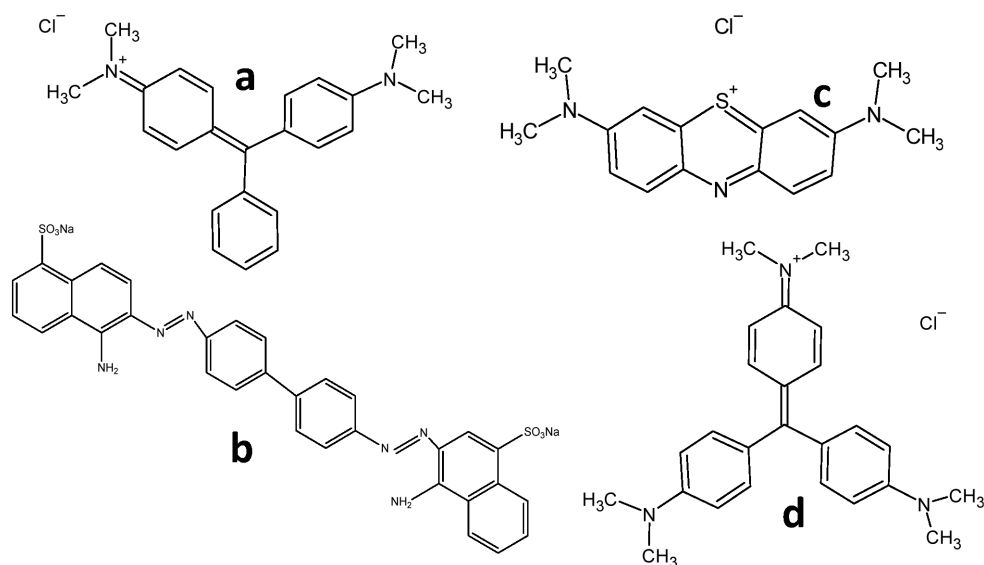


FIGURE 1 | Structure of (a) malachite green, (b) congo red, (c) methylene blue, and (d) crystal violet dyes.

by assessing the validity of pseudo first and second order kinetic models, and subsequent rate constants were estimated from the slope of the following linear equations (Raj et al. 2020):

$$\ln(A_0/A_t) = kt \quad (2)$$

$$\frac{1}{A_t} = kt + \frac{1}{A_0} \quad (3)$$

Moreover, similar procedure was adopted to study the % degradation and kinetics of MB, CR, and CV dyes by the as synthesized AgNPs in the illumination of UV light.

3 | Results and Discussion

3.1 | Phytoreduction of Silver

In this investigation, phytosynthesis of AgNPs by seed extract of date palm (aqueous medium) was studied. It was noticed that the mixture of silver nitrate and the date seed extract exhibited a major color change from yellow to brown within the first 30 min while stirring for 2 h. Subsequently, no further change was observed. This can be attributed to the formation of AgNPs due to the reduction of Ag^+ into Ag by the phytochemicals present in the extract. Whereas, no any color change was observed in the AgNO_3 solution without date seed extract. Brown color appearance can also be attributed to surface plasmon resonance (SPR) vibrations in AgNPs (Ahmed et al. 2016).

3.2 | UV-Visible Analysis

UV-Vis spectroscopy, is an important technique for investigating the synthesis of AgNPs (Huang et al. 2008). It was used to characterize the formation of AgNPs from the silver salt and date seed extract solution. Various physiochemical parameters, including concentration of date seed extract and the salt, temperature conditions, and time of reaction, were adjusted to reduce Ag^+ ions to AgNPs using date seed extract. To optimize the various concentrations of silver nitrate, its different solutions (2–5 mM AgNO_3) were reacted with 1 mL date extract. Figure 2 shows the UV-Visible absorption spectra of AgNPs acquired at different concentrations (2, 3, 4, and 5 mM) AgNO_3 . At 2 mM, a clear SPR band figured, indicating a low yield of AgNPs formed, but with the increase in the concentration of NPs to 5 mM, the SPR of AgNPs shifted to 426 nm as represented in Figure 2. This happening may be attributed to the relatively fast growth of the particles at higher concentrations. At higher concentrations of AgNO_3 , the red-shifted band indicates the formation of larger particles (Ahmed et al. 2016). The AgNPs yield was observed increased with increasing concentration of AgNO_3 (2–5 mM) and the major yield was procured with 5 mM, which was hence used for further investigations.

Furthermore, the effect of varying the volume of date seed extract from 0.2 to 2 mL on the synthesis of AgNPs was also investigated. A steady increase in the intensity of the typical surface plasmon resonance absorption band for AgNPs up to 1.2 mL was observed as shown in Figure 3. Beyond this point, a broad SPR absorption band was observed and the maximum absorption occurred with

a 2 mL volume of date seed extract. The comparison of absorption spectra shown in Figure 3 indicates a change in wavelength from 423 to 448 nm, signifying a redshift as extract concentration rose from 0.2 to 2 mL. This indicates an increase in AgNPs size while increasing the amount of date seed extract.

Figure 4 presents the effect of reaction temperature 303.15, 313.15, and 323.15 K on the synthesis of AgNPs. The comparison of spectra shows that initially absorption peak was shifted to higher wavelength with increasing temperature from 303.15 to 313.15 K. But at higher temperatures (323.15 K), a lower λ_{max} value was noticed which can be attributed to the formation of smaller AgNPs (Zia et al. 2016). The optimized reaction conditions were determined to be 0.5 mL of date seed extract, 5 mM AgNO_3 solution and 323.15 K reaction temperature. The observed broadening and redshift in the absorption peak are attributed to agglomeration or an increase in particle size. The similar shifts were also noticed in a previous study (Ndikau et al. 2017). After 30 min of reaction time, 323.15 K was found to be the ideal temperature for finishing the reaction. According to published research, raising the temperature to 80°C accelerates the pace at which silver ions are reduced (Amin et al. 2012). Hence, the NPs absorption has lot of information related to the particle size and aggregation (Philip 2010). Moreover, the inset of Figure 4 presents the band-gap energy (~2.4 eV) of Ag NPs estimated from the tauc plot.

3.3 | XRD Analysis

The XRD pattern of AgNPs after a 10-min incubation period tells a face-centered cubic structure. As shown in Figure 5, the XRD configuration of the dried NPs suggests that they are crystalline. According to the JCPDS card number 04–0783, the broad diffraction peaks detected in the 2θ range of 20°–80° at 38.73°, 46.69°, 64.93°, and 77.81° relate to the (111), (200), (220), and (311) planes, correspondingly. These XRD patterns confirm the synthesis of pure silver nanoparticles and are consistent with previously reported data (Kumar, Palanichamy, and Roopan 2014; Kumar et al. 2012). The average crystallite size was calculated using following Debye–Scherrer equation:

$$D = \frac{k \lambda}{\beta \cos \theta} \quad (4)$$

where D represents the average size of the nanoparticle crystals, k denotes the geometric constant (0.9), λ indicates the wavelength of the x-ray radiation source, and β represents full width at half maximum (FWHM) of the XRD preferred peak at 2θ diffraction angle (Balaji, Senthilkumaran, and Thangadurai 2014). The average crystallite size of the AgNPs calculated using Equation (4) was ~15 nm.

3.4 | SEM Analysis

Figure 6 presents the SEM images of the Ag NPs which were recorded at 10,000× and 20,000× magnifications with a working distance of 5.1 mm to consider the various aspects of the morphology of prepared nanoparticles such as size and texture. The prepared nanoparticles are observed rectangular in micrographs, which are distributed with smooth surfaces and

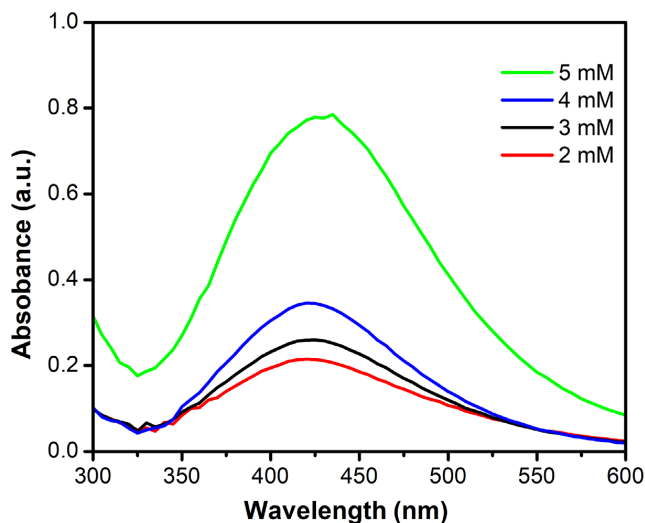


FIGURE 2 | The comparison of UV-visible measurements of Ag NPs for various contents of AgNO_3 .

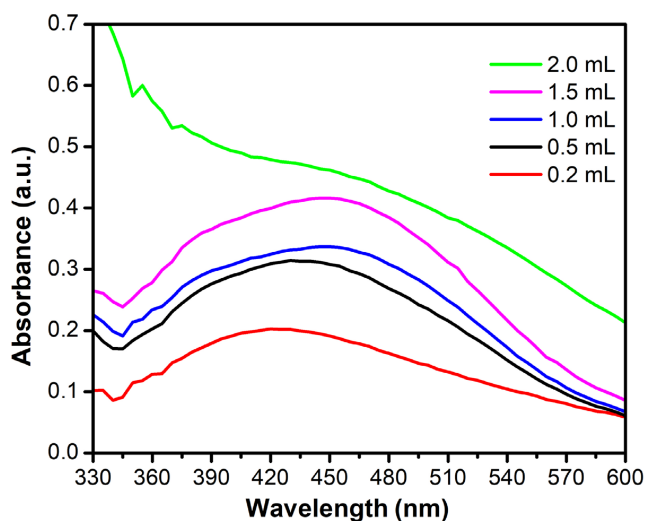


FIGURE 3 | The comparison of UV-visible absorption spectra of Ag NPs prepared from 5 mM AgNO_3 with varying volumes of date seed extract.

compact structures. The nanoparticles tend to form clusters because of their minor size and high surface energy. Moreover, particles with variable diameter can be seen in the SEM images.

3.4.1 | FTIR Analysis

FTIR spectroscopy was used to identify the potential biomolecules from *P. dactylifera* involved in the synthesis of AgNPs and it also plays a substantial function in the stabilization of NPs. Typically, pure silver nanoparticles exhibit weak IR absorption due to silver's minimal interaction with IR radiation. The FTIR spectrum (Figure 7) displayed peaks at 3271 and 1642 cm^{-1} , which may correspond to O—H, aliphatic C—H, and carbonyl stretching vibrations of flavonoids or phenolic groups. Specifically, OH stretching of phenolic groups is depicted at 3271 cm^{-1} , while at 1734 cm^{-1} is likely associated with the C=O stretching of proteins, consistent with literature findings

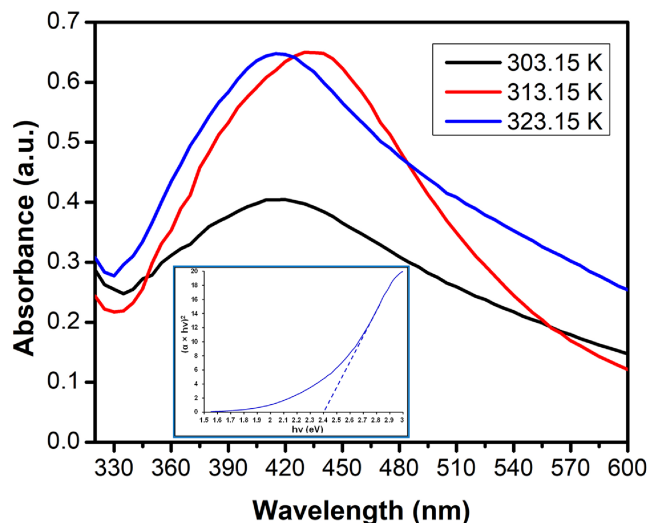


FIGURE 4 | The comparison of UV-visible absorption spectra of Ag NPs prepared at different temperatures (K) from 5 mM AgNO_3 and 0.5 mL date seed extract whereas inset is the direct bandgap energy evaluation of Ag NPs prepared at 323.15 K.

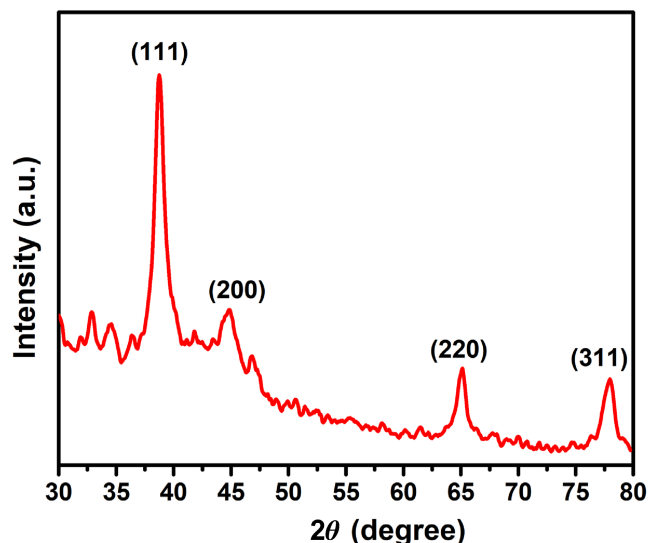


FIGURE 5 | The x-ray diffraction pattern of Ag NPs prepared from date seed extract.

(Bobbu et al. 2016). FTIR analysis identified proteins, terpenoids (Mashwani et al. 2016), and flavonoids (Jain and Mehata 2017) in the aqueous seed extract of *P. dactylifera*. Flavonoids and terpenoids are likely involved in the synthesis of AgNPs, while proteins may contribute to their stabilization by coating the nanoparticles (Kumar et al. 2017). A shift in the peaks for AgNPs to 3275 and 1634 cm^{-1} indicates that O—H and CO groups have adsorbed onto the surface of the nanoparticles, suggesting their involvement in the reduction process (Kumar et al. 2018).

3.5 | Photocatalytic Degradation of Dyes by the Ag NPs

Figure 8 presents a decrease in absorption of dyes in the presence of Ag NPs while increasing the UV light exposure duration. This

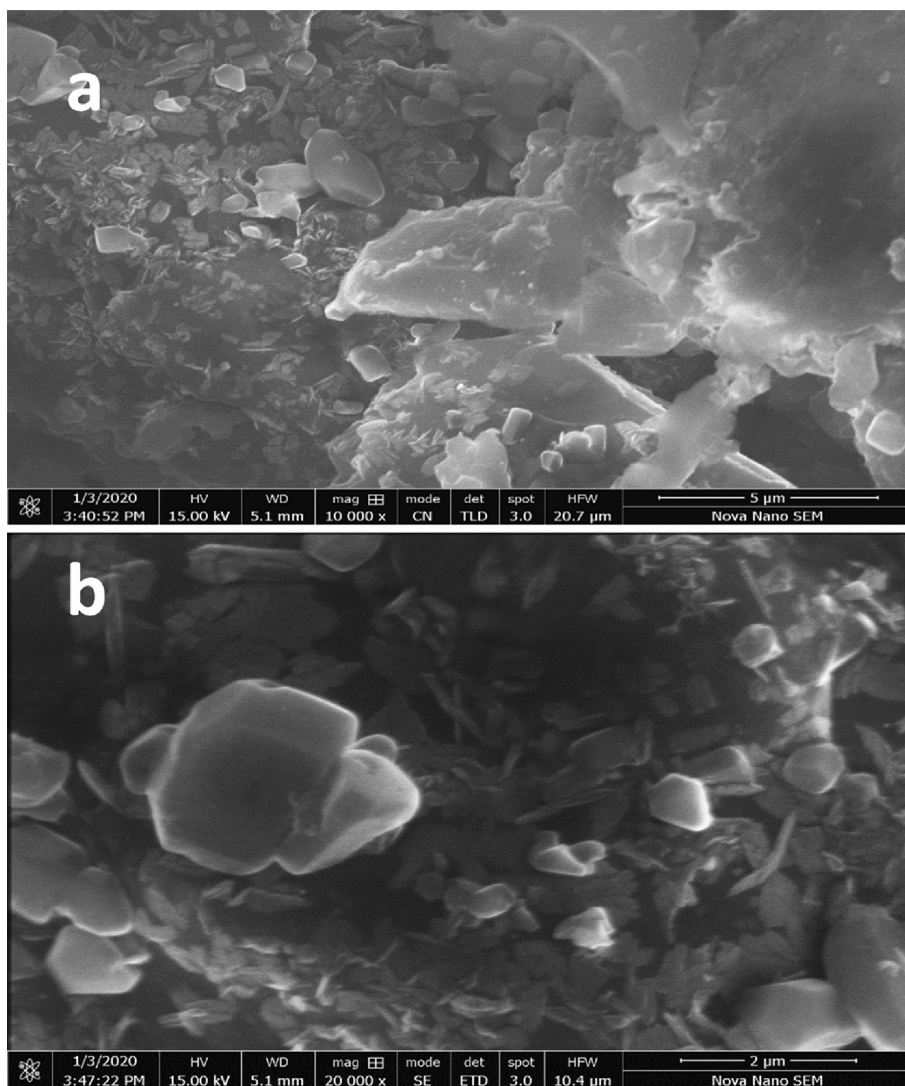


FIGURE 6 | (a, b) SEM images of AgNPs at different magnifications.

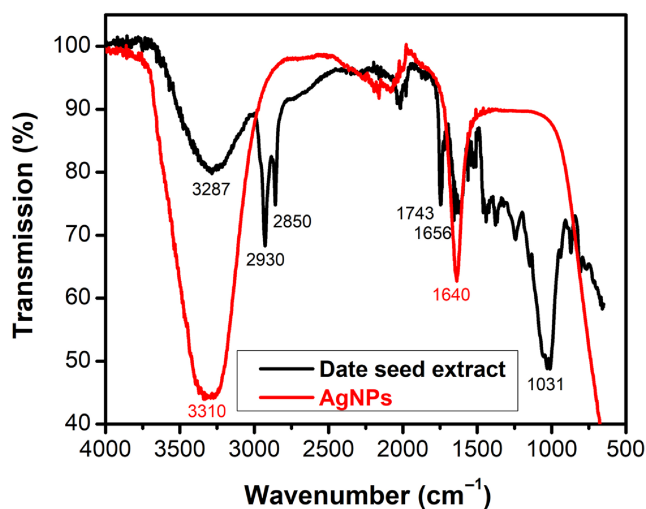


FIGURE 7 | The FT-IR spectra of AgNPs and date seed extract.

continuous decrease in the absorption of dyes also represents the involvement of Ag NPs in removing the dyes from the polluted water under light illumination. MG dye is characterized by a

specific wavelength of 620 nm as shown in Figure 8a wherein a steady drop in the dye solution's absorbance was observed which further suggests ~83% MB dye degradation through MB chromophore's breakage and then dye decolorization. Moreover, Figure 8b–d also present their successive degradation by AgNPs photocatalyst with increasing light exposure duration. The higher photodegradation of MG dye (Figure 9) by the AgNPs is mainly attributed to the dye's greater structural compatibility in terms of adsorption and interaction with the photocatalyst as compared to other dyes. The decreasing order of photodegradation of dyes is as follows:

Malachite green (83%) > Congo red (79%)
> Crystal violet (62%) > Methylene blue (48%)

Kinetic studies revealed the validity of pseudo 1st order kinetic model with greater correlation coefficient than pseudo 2nd order kinetics for the photodegradation of all four dyes as shown in Figure 10. Moreover, the photodegradation through 1st order kinetics shows the breakage of dye structure initiated by its adsorption on the surface of a photocatalyst. In addition, the higher rate constant ($8.2 \times 10^{-3} \text{ min}^{-1}$) for the degradation of

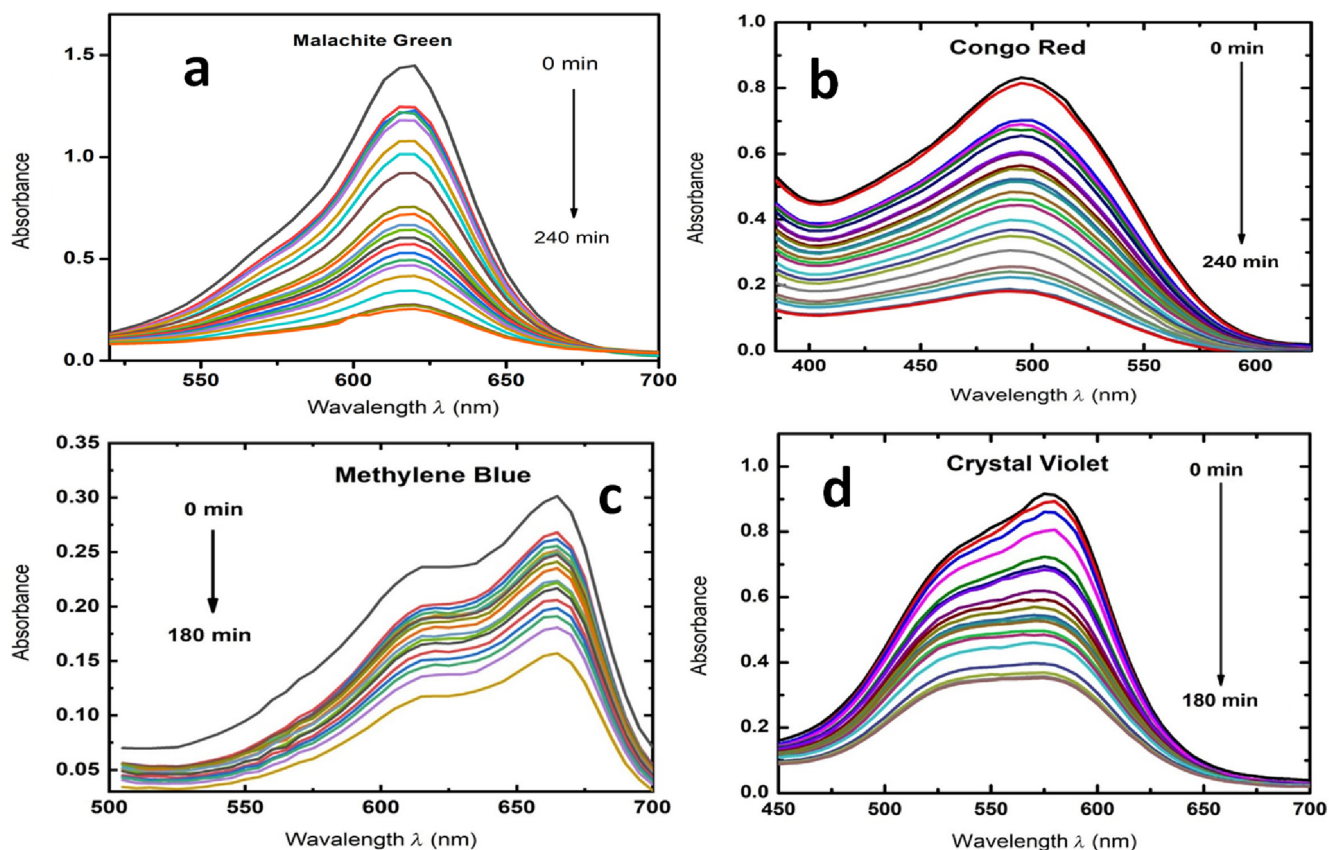


FIGURE 8 | The comparison of absorption spectra of (a) MG, (b) CR, (c) MB, and (d) CV dyes in the presence of Ag NPs at ambient temperature by increasing UV light exposure duration.

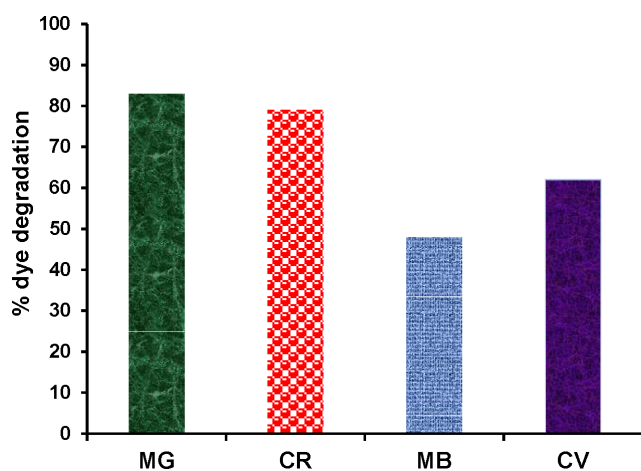


FIGURE 9 | The comparison of % degradation of dyes by Ag NPs under the exposure of UV light.

MG dye indicates the fast MG adsorption and its structure compatibility with the surface of Ag NPs.

The decreasing trend of dye degradation rate over Ag NPs is provided below: MG ($8.2 \times 10^{-3} \text{ min}^{-1}$) > CR ($6.0 \times 10^{-3} \text{ min}^{-1}$) > MB ($5.4 \times 10^{-3} \text{ min}^{-1}$) > CV ($3.4 \times 10^{-3} \text{ min}^{-1}$).

The mechanism of degradation of these dyes on the surface of AgNPs under the illumination of UV light follows both adsorption process initially and then dyes' structure breakage through reactive oxygen species (ROS). It has been reported that the reduction of O_2 and oxidation of H_2O on the AgNPs surface generates highly ROS, which are accountable for dye degradation (Fardood et al. 2019; Sirdeshpande et al. 2018; Zulfiqar, Temerov, and Saarinen 2020). Moreover, the comparison of this study with the few already reported studies is also provided in Table 1.

4 | Conclusion

The present work demonstrates the potentially viable route to synthesize AgNPs using *P. dactylifera* seed extract and their utilization in the photodegradation of MG, CR, MB, and CV dyes. The synthesis and stability of synthesized AgNPs were confirmed by UV-visible spectroscopy. The seed extract of *P. dactylifera* gave small rectangular-shaped nanoparticles, which were confirmed through XRD pattern. FTIR unfolded the functional groups responsible for reducing Ag^+ to AgNPs. Furthermore, the synthesized AgNPs showed excellent activity for MG dye degradation (~82%) with $8.2 \times 10^{-3} \text{ min}^{-1}$ rate among other dyes. The validity of pseudo 1st order kinetics confirms the degradation of dyes through adsorption process

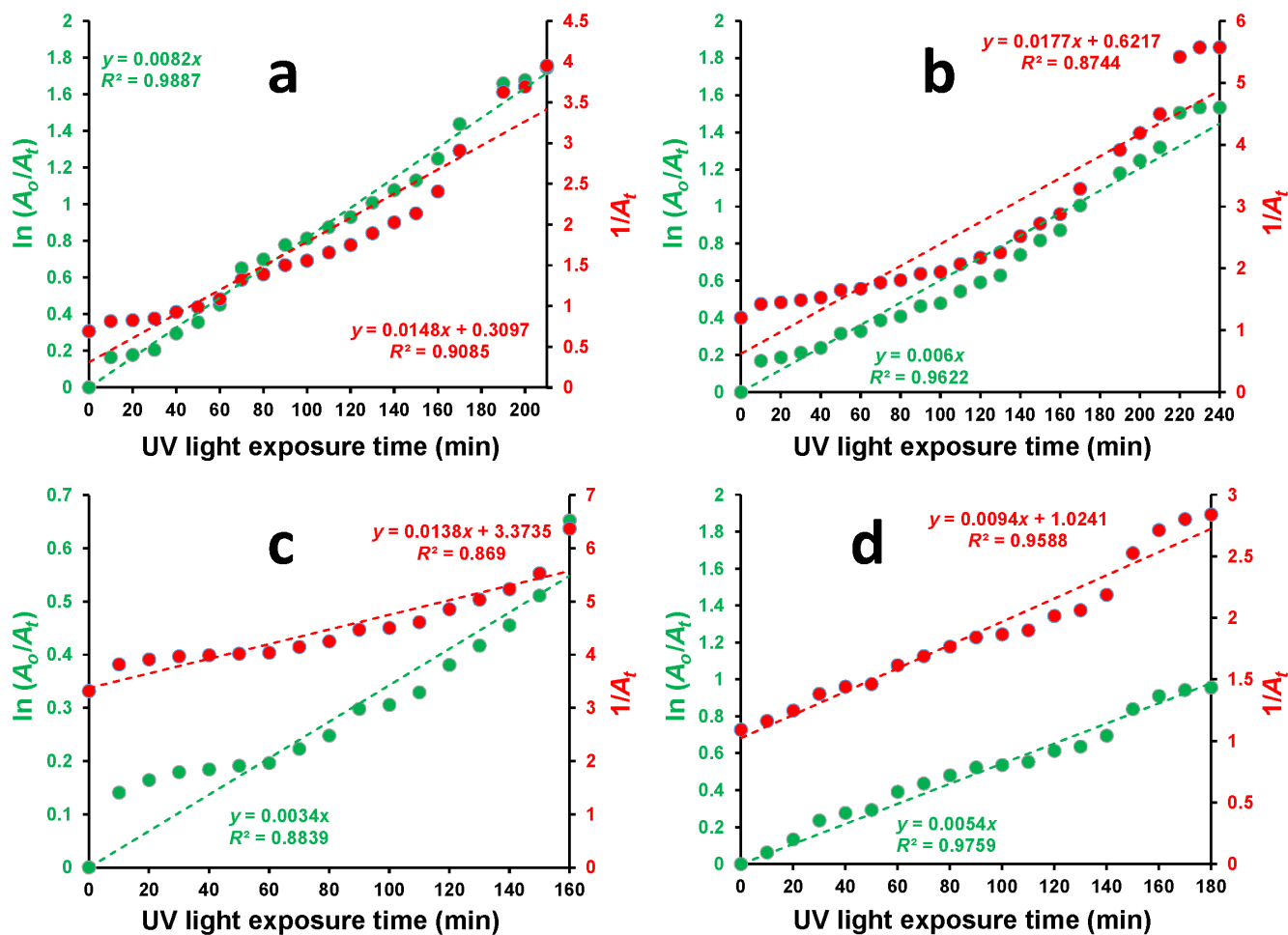


FIGURE 10 | Kinetic studies plots for the photodegradation of (a) MG, (b) CR, (c) MB, and (d) CV by Ag NPs under the illumination of UV light exposure.

TABLE 1 | The comparison of bandgap energy, particle size, and photocatalytic activity of Ag NPs prepared from seed extract of various plants with the date seed extract used in this study.

Plant name	Bandgap energy (eV)	Particle size (nm)	Photocatalytic system	Photocatalytic degradation activity (%)	References
<i>Mesua ferrea</i>	2.7	15.0	Congo red under solar light	92	(Thirumagal and Jeyakumari 2020)
<i>Annona squamosa</i> L.	—	22.0	Coomassie brilliant blue under sunlight	100	(Jose, Raphel, and Aiswariya 2021)
<i>Nigella sativa</i>	—	11.7	Congo red under solar light	96	(Chand et al. 2021)
<i>Moringa oleifera</i>	—	4.0	Methylene blue under solar light	91	(Mehwish et al. 2021)
<i>Phoenix dactylifera</i>	~2.4	5–15	Malachite green under UV light	~83	This study
			Congo red under UV light	~79	
			Methylene blue under UV light	~48	
			Crystal violet under UV light	~62	

initially and then dye structure's breakage by the generated ROS from the AgNPs.

Author Contributions

Areesha Maryam: writing – original draft, validation, formal analysis. **Saqib Rabbani:** methodology, formal analysis, investigation. **Athar Yaseen Khan:** methodology, formal analysis, visualization, writing – original draft. **Hina Abid:** writing – original draft, investigation, resources, visualization. **Ammar Zidan:** writing – review and editing, visualization, software. **Ali Bahadur:** conceptualization, writing – original draft, software, resources. **Muhammad Tariq Qamar:** project administration, conceptualization, writing – original draft, visualization. **Shahid Iqbal:** writing – original draft, supervision, validation. **Sajid Mahmood:** investigation, writing – review and editing, project administration, data curation. **Abd-ElAzim Farouk:** funding acquisition, data curation, formal analysis. **Ibrahim Jafri:** funding acquisition, data curation.

Acknowledgments

The authors extend their appreciation to Taif University, Saudi Arabia, for supporting this work through project number (TU-DSPP-2024-65).

Conflicts of Interest

The authors declare no conflicts of interest.

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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