UV-light Mediated Green Synthesis of Silver Nanowires and Their Catalytic Degradation Potential Against Methyl Orange

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Abstract

In the present study, a simple and eco-friendly method for the synthesis of silver nanowires (Ag-NWs) has been reported. *Psidium guajava* seed extract was used as a reducing agent for silver nitrate solution at 70 °C temperature under continues UV-irradiation, for the production of Ag-NWs. Silver nanowires were initially characterized by using UV-visible and FTIR spectrophotometer. Synthesis of nanowires and involvement of functional groups was confirmed by FT-IR spectra. The morphology and particle size of synthesized Ag-NWs was determined using Field Emission Scanning Electron Microscopy (FESEM) and X-ray diffraction (XRD). XRD results revealed cubic phase morphology of Ag-NWs. Nanowires were found having 12-8 µm length and 300-500 nm diameter. In addition, catalytic potential of the sample for degradation of methyl orange dye (MO) was tracked spectrophotometrically. The results exposed that; bio-synthesized silver nanowires were found having excellent morphological features as well as impressive catalytic potential.

1. Introduction

Heavy metals, organic and un-natural dyes cause countless serious complications to human health and eco-system, however, manipulation of such chemicals are unescapable due to their widespread applications in diverse meadows of science and technology. With the hasty growth of industries for instance ore mining, textile industries, paper and paint industry, fertilizers and pesticide industries etc., an extensive aggregate of dyes, heavy metals and pigments are predisposed afterward the industrial progressions (Mance, 2012). The effluents commencing from such industries are discharged-off into natural-surfaces and ground-water resources. These toxic-effluents amends the composition of surface-water and consequently sound effects to the healthiness of living-beings. With the passageway of time, pigments and dyes available in water submit yourself to degradation chemically and transformed to supplementary hazardous toxic-chemical entities (Chang & Chen, 2009). Finally, these heavy metals and dyes possibly will indirectly or directly enter the food-web and originate unadorned toxic-impacts on surroundings. Thus, it is lethal to become aware of and reduce such engineering-wastes from water.

There are numerous techniques reported previously for the treatment of unnatural-dyes filthy-water. These comprise that, catalytic reduction (Begum et al., 2018), photo-catalytic degradation (Saeed, Ahmad, Boddula, ul Haq, & Azhar, 2018), advance oxidation routes (Qiu et al., 2017), membrane matrices (Fersi, Gzara, & Dhahbi, 2005), bio-remediation (Mani & Kumar, 2014), adsorption (Chongmin Liu, Wu, Tran, Zhu, & Dang, 2018). Amongst them, reductive squalor by employing nanocatalyst partake been pragmatic on broad-spectrum for the treatment of dyes in aqueous solutions prior to its proficiency, clean dispensation, and cost effectiveness (Hassan et al., 2011; Hu et al., 2015). Reduction of such dyes by nanocatalyst transfigure them to gamely biodegradable out-puts, that be able to further processed on requirement (Hassan et al., 2011). Metal based nanocatalyst for such reduction feedback mainly count on noble metals, like Ag and Au (P. Wang et al., 2008; Yasin, Liu, & Yao, 2013) in line for their extraordinary stability and high specific area. Thus, starved of appropriate surface stabilizers, these nanocatalysts endures aggregation, which can primarily domino effect of degradation of their catalytic-activities and lifetime

(Lin, Tsai, Chen, Lin, & Chen, 2004). Therefore, this is vitally important to stabilize nanocatalyst with accurate-ligands to enhance its efficiency and life-span. Several routes for preparing metal nanoparticles have been developed i.e., co-precipitation (Kim, Kim, & Lee, 2003), hydrothermal synthesis (Daou et al., 2006), sol-gel method (Chao Liu, Zou, Rondinone, & Zhang, 2001), inert gas condensation (Pérez-Tijerina et al., 2008), laser ablation (Amendola & Meneghetti, 2009), sputtering (Rane, Kanny, Abitha, & Thomas, 2018), template synthesis (Sreeram, Nidhin, & Nair, 2008), and biological synthesis (Devi et al., 2013). However, biological synthesis is the hot choice with advantages over physical and chemical methods as it is quick, eco-friendly, highly stable, and cost-effective. Biological synthesis does not require any culture growth and does not produce toxic residues to contaminate the atmosphere (Kulkarni & Muddapur, 2014).

Catalytic reduction mainly depends upon the morphology (Sun, 2010) and surface area of the nano-structures (Das & Soni, 2017) i.e., nanoparticles (Yousaf, Mehmood, Ahmad, & Raffi, 2020), nano-rods (Sawant & Sawant, 2020), nano-spheres (Ramya, Jyothi, Vardhan, Gopal, & Desai, 2020), nanotubes (Nadagouda, Speth, & Varma, 2011) and nanowires (Goh et al., 2012). Among them nanowires have grabbed the prime focus of the material scientists due to their excellent features like surface area, micro-porous structural features, highest contact area with adsorbate surfaces (Z. Wang, Liu, Chen, Wan, & Qian, 2005). Lin Bao *et al.* has reported the syntheses of nanowires via green approach by using poly-vinyl acetate (PVA) polymer back-bone as a stabilizing agent due to the lower stability of Ag-nanowires (Ag-NWs) (Luo, Yu, Qian, & Gong, 2006). Many serious attempts have been made to enhance the stability of Ag-NW by using stabilizers like polymer matrices (PVA, PPy EG, and glucose) (Zhu et al., 2016), ITO glass electrode base (Sim et al., 2016) and electrostatic charge stabilizers (Yang et al., 2011). The synthesis of Ag-NW *via* a facile, green and eco-friendly routes without any external stabilizers and prolonged shelf-life is still a great challenge for the scientists (Guo, Chen, Wang, Jiang, & Wang, 2020).

Herein, we are reporting a facile and green approach to synthesize the Ag-Nanowires (Ag-NW) by using *Psidium guajava* seed extract to stabilize the nanowires, without any external stabilizers like ITO-glass electrode, electrostatic charge which was the classical approach to synthesize the nanowires. The plant seed extract by dissolving 0.1 g of the dry and finely divided form in 100 mL water at 60°C for 50 minutes followed by the vacuum filtration and used as a precursor for the reduction of silver ions. Then freshly prepared 0.1 mM solution of AgNO₃ was taken in conical flask. The addition of 5 mL of *Psidium guajava* seed extract was done to AgNO₃ under continues stirring and UV-irradiation of 265 nm wavelength white light, and stirred the reaction mixture for 4 hours. This is first effort to synthesize Ag-NW *via* a green, eco-friendly and template free pathway, with greater stability of Ag-NW.

2. Materials And Methods

2.1. Materials

Silver nitrate was acquired from Merck, Germany, sodium hydroxide (NaOH) and sodium borohydride (NaBH₄) was obtained from Sigma Aldrich, UK. The methyl orange dye was purchased from Fisher,

Pakistan Ltd. All chemicals were in analytical grade and used without further purification. Double deionized water was used throughout the analysis.

2.2. Preparation of *Psidium Guajava* Seeds Extract

Psidium guajava seeds were collected washed and dried in oven around 60°C till 48 hours. Ground the *Psidium guajava* seeds in grinder and filtered with mesh sieved of 0.40 micron and stored in Eppendorf tube. The 0.1 g of the *Psidium guajava* seeds in 100 mL distilled water were heated in a conical flask with constant stirring at 60°C temperature for 50 minutes (Sharma et al., 2021). The filtration was carried out to separate the plant extract with vacuum filtration apparatus. Freshly prepared plant extract was used for the further experiments (Sathiyavimal et al., 2021).

2.3. Synthesis of Silver Nanowires

A solution of AgNO₃ of 0.1 mM concentration was prepared by dissolving AgNO₃ in deionized water. In a classic green-synthesis practice, *Psidium guajava* seeds extract was used to reduce and cap the Ag ions. 5 mL of *Psidium guajava* seeds extract was added into the aqueous solution of AgNO₃ under standard conditions. The mixture was constantly stirred at 70°C in a homemade UV-lamp containing a single wavelength of 265 nm of white light for four hours by using magnetic stirrer. The color change from milky white to yellow and finally orange was showing the formation of the silver nanowires. Ag-nanowires were obtained on filtration and followed by calcination at 50°C for 30 minutes (Scheme 1 and Fig. 1).

2.4. Catalytic activity of MO dye

Catalytic-degradation of MO dye was supervised over UV/Vis spectrophotometer using *Psidium Guajava* seed extract's stabilized Ag-NWs. The degradation was done by time dependent kinetic studies with constant 3 minutes' time interval. 0.4 mL quartz cuvettes were utilized during all reactions. 0.6 mL of 17.6 mM constant concentration of NaBH₄ was used for the optimized model reaction as well as for both factors study. For catalyst Ag-NWs dosage factor, 0.4 mL of different concentrations of Ag-NWs (0.64 to 3.84 mg/mL) were used. Similarly, 1.6 mL of MO dye having concentration ranges from 0.062 to 0.102 mM were used for MO dye's factor study. All the reactions follow Pseudo first order reaction with Langmuir-Hinshelwood mechanism (LHM) as the 200% more NaBH₄ was used in contrast with MO dye.

$$\ln (A_t/A_0) = -k_{\text{obs}} t (\text{NaBH}_4 \gg \text{MO dye})$$

Reaction mixture without Ag-NWs was observed over UV/Vis spectra ranges from 300–800 nm as reference to compare with reaction mixture containing Ag-NW's UV/Vis spectra for the confirmation of Ag-NWs.

3. Characterization Of Ag-nws

The fabrication of AgNWs was preliminarily established by recording the absorbance in UV/Vis spectra at a range of 300–800 nm. The change in Surface Plasmon Resonance (SPR) of nanoparticles in the

dispersion was recorded using UV/Vis spectrophotometer by CECIL-Aquarius 7400 ce UK. The XRD patterns of Ag-nanowires were collected on Bruker AXS-D8 Advanced X-Ray diffractometer with *Cu K α* radiations of $\lambda = 1.5406 \text{ \AA}$ and scanning angle 2θ over the range of $10-80^\circ$. Crystallite size was calculated by using Scherer Equation $CS = K\lambda/\beta \cos \theta$, where CS is the crystallite size, constant $K = 0.94$, β is the full width at half maximum (FWHM), ($\beta = \text{FWHM} \times \pi/180$), $\lambda = 1.5406 \times 10^{-10}$ and $\cos \theta = \text{Braggs angle}$. Fourier Transformation Infrared Spectroscopy (FTIR) was used to characterize the nanoparticles using the powder sample by ATR in the range of $400-4000 \text{ cm}^{-1}$. Scanning electron microscopy (SEM) images were recorded using FEI-NOVA-450 Nano-SEM (FE-SEM) by USA. The functional group determination was carried out by utilizing Alpha-II FTIR-ATR by BRUKERS Internationals (USA).

4. Results And Discussion

UV-visible spectroscopy is a convenient, preliminary, and indirect method for characterization of Ag-NWs based on optical properties called surface plasmon resonance (SPR) (Pathak & Singh, 2020). The Ag-nanowires bands occur at 435.0 nm with progressive increase in absorbance for 30 minutes (Fig. 2) (Chung et al., 2021). From this spectrum, it is clear that there is no peak in Psidium Guajava seed's curve which illustrates the absence of Ag-NWs. But, the peak in AgNWs-Guajava curve confirmed the creation of nanowires of Ag with wide size distribution. (Parente et al., 2020).

4.1. FTIR spectra of Ag NWs

FTIR spectrum of as-prepared Ag-NWs was conceded to pinpoint the biomolecules accountable for the reduction and capping of silver ions (Fig. 3). The existence of peaks at 3341.8 , 2927.4 and 1234 cm^{-1} may perhaps be due to $-N-H$ stretching of the secondary amine and its salts and primary amine, respectively. The peak 3341.8 cm^{-1} shrink in case of silver due to interaction $-OH$ group of nanowires with amines. The peaks around 2197.4 cm^{-1} and 1619 cm^{-1} affirms the presence of α , β -substituted unsaturated ketones. Two peaks observed at 2161.3 cm^{-1} and 2009.4 cm^{-1} confirms the iso-cyanate and thio-cyanate functionalities, respectively. The peaks at 1735 cm^{-1} and 1647 cm^{-1} confirm the presence of ortho-substituted six-membered lactone. The $-OH$ bending of α , β -unsaturated carboxylic was recorded at 1419.6 cm^{-1} . Secondary amine its salts and α , β -unsaturated carboxylic acts as reduced silver nitrate to silver ions also work as a capping agent and stabilizes the Ag-NWs (Le et al., 2021).

4.2. Scanning Electron Microscopy

Morphology and surface properties of the product was determined by field emission scanning electron microscopy (FE-SEM) (Fig. 4). It is evident that the product has wires i.e., morphology mainly formed and stabilized due to the action of unsaturated linear ketones. The well separated wires have $\sim 12 - 8 \text{ }\mu\text{m}$ length and $\sim 500 - 300 \text{ nm}$ diameter. This unique morphology results an excellent surface area expansion, mainly responsible for the brilliant catalytic reduction of the organic dyes. The performance of a catalyst chiefly dependent on surface area, stability, surface charge, and shape of the molecules of a catalyst. Amongst them surface area and shape have shown more powerful role for the adsorption and

reduction of the adsorbed entities in photocatalysis phenomena as mentioned in previous works (Satsangi, 2020).

4.3. XRD analysis of Ag-NWs

As-obtained Ag-NWs were further characterized by X-ray diffraction method to elucidate the crystal structure and crystallite size (Fig. 5). The finger-print pattern has four typical diffraction features corresponds to (111), (200), (020), (220), and (131) planes, and all the four peaks might be indexed to standard cubic phase of silver (JCPDS-870720) (Nagasundari, Muthu, Kaviyarasu, Al Farraj, & Alkufeidy, 2021). The final product owes 100% purity as there was no peak detected for reflection, mainly corresponds to nitrate ions and other impurities. The peak intensity profile was individual of simple cubic construction of Ag-nanowires. The size of Ag-NWs was determined by using Debye-Scherrer equation-1 (Lim, Marks, & Rowles, 2020).

$$\text{Particle size}(D) = \frac{k.D}{\beta.Cos\theta} \quad (\text{eq-1})$$

is the crystalline size, k is the wavelength of x-ray used, b is the full width at half maximum light of the maximum intensity peak and h is the Bragg's angle. The crystallite size of the synthesized Ag-nanowires is estimated ~ 19.63 nm.

5. Catalytic Reduction Of Methyl Orange (Mo)

Methyl Orange (MO) is widely used as an indicator as well as a textile dye (azo-dye) for dyeing of textile fabrics (Carolin, Kumar, & Joshiba, 2021). The reduction of MO was calculated by using freshly prepared Ag-NWs in excessive NaBH_4 . The rate of reduction of methyl orange without catalyst in the presence of NaBH_4 is very slow (Fig. 6-a). This poor performance is due to the presence of high energy barrier of mutually repulsive interactions between the borohydride anion and methyl orange ion, which should be overcome only by catalyst (Wu et al., 2020). Moreover, in the presence of catalyst only no reduction occurs due to the same interactions as mentioned above (Fig. 6-b). However, in the presence of catalyst and NaBH_4 , reduction of azo-dye takes place (Model reaction). Initially, NaBH_4 and catalyst will adsorb on the surface of the dye and then reduction reaction will proceed at faster rate. The characteristic peak of MO solution was recorded ~ 458 nm and catalytic reduction was observed by a sharp decline in intensity merely in 25 minutes (Fig. 6-c).

6. Kinetic Studies

6.1. Effect of Catalyst dosage

The effect of concentration of catalyst-dosage was determined by changing the amount of Ag-NWs from 0.60–3.84 mg/mL (Fig. 7-a). The rate of reaction increases fast from 0.60–1.28 mg/mL because the adsorption takes place exponentially in the start of reaction due to the presence of active sites. Later on

the reaction rate becomes slow from 2.56–3.84 mg/mL due to the occupation of the available active sites (Rodwihok et al., 2020). The effective degradation out of all the adsorbent's concentration was monitored at 1.28 mg/mL by the help of k_{obs} graph plotted between k_{obs} vs concentration of Ag-NWs, while keeping the NaBH_4 concentration (17.6 mM) and MO-dye (0.082 mM) constant.

It is evident from the Fig. 7-b, that from 0–5 minutes' reaction was started with a very slow speed as there are molecules moving towards the surface of Ag-NWs and speed-up from 6–20 minutes due to their interaction at the surface of catalyst, then ultimately reaches to completion after 20–25 minutes. The inset graph shows the negative slopes which used for the determination of k_{obs} (Shan et al., 2020).

6.2. Effect of MO dye

Figure (8-a) illustrates the graph between k_{obs} on ordinate and concentration of MO-dye on abscissa, while keeping catalyst dosage (1.28 mg/mL) and NaBH_4 (17.6 mM) constant. This graph explains that initially k_{obs} value remains constant while, at 0.082 mM its concentration increases to maximum k_{obs} value. Which is due to the maximum adsorption of incoming MO-dye's molecules on the surface of catalytically enhanced by Ag-NWs. This point is fruitful in optimization of reaction. After this concentration the decrease in k_{obs} 's value is due to the excessive amount of MO-dye molecules on the surface of limited Ag-NWs. Here the double layer adsorption occurs on the surface of Ag-NWs, which restricts the effective adsorption.

MO dye's concentrations were taken, ranges from 0.072 to 0.102 mM with 0.010 mM difference. Figure 8-b shows the induction, reaction and completion times of the catalytic reaction between MO-dye and NaBH_4 on the surface of Ag-NWs. As, the concentration of MO-dye increases, while keeping the concentrations of Ag-NWs and NaBH_4 constant, the time of degradation increases (Ramos, Luyo, Sánchez, Gomez, & Rodriguez, 2020). The reason behind this factor is the constant concentration of Ag-NWs, which illustrates the limitation of surface active sites for incoming reactants (Raj, Singh, Trivedi, & Soni, 2020b). The figure inset (6) shows the negative slopes for finding the apparent rate constant for Pseudo first order reaction following Langmuir-Hinshelwood mechanism (LHM) (Kumari & Meena, 2020).

Table 1
Comparison of time duration of MO-dye degradation by NW and nanoparticles.

Reduction Source	Nanostructures (NS)	Dimensions of (NS)	Time (minutes)	Ref.
<i>Lavandula angustifolia</i> plant extract	Nanowires	100 μm (L) 60–130 nm (d)		(Villalpando, Saavedra-Molina, & Rosas, 2020)
<i>Terminalia arjuna</i> leaf extract	Nanoparticles	10–15 nm	29	(Raj, Singh, Trivedi, & Soni, 2020a)
<i>Nervalia zeylanica</i> leaf extract	Nanoparticles	34 nm	60	(Vijayan, Joseph, & Mathew, 2019)
Ag@AgBr	Nanowires	54 nm	160	(Y. Wang, 2016)
Ag@Cu ₂ O	Nanowires	10 μm (L) 100 nm (d)	140	(Xiong et al., 2014)
<i>Psidium guajava</i> seed extract	Nanowires	~ 19 nm	25	This Work

Conclusion

The Ag-NWs were successfully fabricated using AgNO₃ as precursor and *Psidium guajava* seed extract under continuous irradiation of UV light. The extract strongly acted as reducing as well as stabilizing agent. UV-visible spectra confirm the formation of the Ag-NWs. The average crystallite size of Ag-NWs was 19.63 nm and morphology were cubic face having 12-8 μm length and internal diameter of 300-500 nm. Synthesized Ag-NWs was then used for catalytic degradation MO-dye, NaBH₄ and Ag-NWs. The reaction completed in 25 minutes and kinetic studies of the data confirmed pseudo first order reaction. Hence, *Psidium guajava* seed extract can be used for the synthesis of Ag-NWs. The authors recommended that Ag-NWs can be exploited for degradation of azo-dyes that can be a good tool for treatment of water from textile industry.

Declarations

Ethical Approval

All the data and information in this manuscript original and not been published in any other Journal before this.

Conflict of Interest

Author declares no conflict of interest with anyone.

Consent to Participate

Not applicable.

Consent to Publish

Authors are willing to publish this data.

Authors Contributions

Faisal Ali: Synthesized and characterized the compounds. **Zahid Ali:** Data curation, Writing – original draft. **Ghulam Mooin-ud-din:** Theoretical calculation and Writing – theoretical part.

Umer Younas: Final formatting and cross-review. **Muhammad Pervaiz:** Software validation. **Faiza Hassan:** Dye-degradation studies. **Hafiz Muhammad Yasir:** Technical support. **Adnan Ashraf:** Discussion on theoretical results. **Muhammad Waqas Ishaq:** Conceptualization, Methodology.

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Availability of data and materials

All original data and materials are available on demand.

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Figures

Figure 1

Milky white solution (a), Orange colored Ag-Nanowires (b), Ag-NW powder oven dried (c)

Figure 2

Ag-NWs confirmation with UV/Vis spectra and comparison with Psidium guajava UV-visible spectroscopy

Figure 3

FTIR comparison of Psidium guajava and Ag-NWs

Figure 4

FE-SEM images of Ag-NWs stabilized Psidium guajava seeds (a, b) length of nanowires ~8-12 μm and (c, d) average diameter ranges between 300-500 nm

Figure 5

XRD pattern of Ag-NWs

Figure 6

Reaction monitoring over UV/Vis; in the absence of catalyst (a), in the absence of NaBH₄ (b) and Model reaction at optimized conditions; NaBH₄ = [17.6 mM], MO = [0.082 mM], Ag-NW (catalyst) = [1.84 mg/mL]

Figure 7

Effect of Ag-NWs-Catalyst dosage for kinetic study at 0.60, 1.28, 2.56, 3.84 mg/mL concentrations, NaBH₄ = 17.6mM, Ag-NWs = 1.84 mg/mL (a) and k_{obs} determination (b)

Figure 8

Effect of MO dye for kinetic study at 0.062, 0.072, 0.082, 0.092, 0.102 mM concentrations, NaBH₄ = 17.6mM, Ag-NWs = 1.84 mg/mL (a) and observed-rate constant determination (b)

Supplementary Files

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- [Scheme01.png](#)