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PHOTOCATALYTIC DYE DEGREDATION USING ECOFRIENDLY SYNTHESIZED SILVER NANOPARTICLES

G. E. Ankwai*[1], S. A. Mamuru[2], O. O. Balogun[3], M. Ibrahim[1], N. J. Shamle[1]

*[1]Department of Chemistry, University of Jos, P.M.B 2084, Jos, Plateau, Nigeria
[2]Department of Pure and Applied Chemistry, Adamawa State University, Mubi, Nigeria
[3]School of Preliminary Studies, Nile University of Nigeria, Abuja
*Corresponding author: G. E. Ankwai
*E-mail: kokoeffi2010@gmail.com

ABSTRACT

Indiscriminate discharge of industrial wastewater without proper treatment pose a great threat to the environment and living things. Although various methods are presently being used to mitigate these effects however, most of these methods have some disadvantages such as high cost, low efficiency and risk of reintroducing more pollutants to the environment. Presently, nanoparticles, due to their large surface to volume ratio and excellent adsorbent properties, have been applied in wastewater treatment. Literature studies has also revealed the efficacy of nanoparticles for individual dye degradation. In this study, ecofriendly silver nanoparticles (AgNPs) was successfully synthesized using aqueous leaves extract of Lantana camara. The ecofriendly synthesized AgNPs was first confirm by the observation of a distinct colour change from colourless to light brown after addition of the aqueous leaves extract to 0.1 M silver nitrate salt solution. The AgNPs was examined using UV-vis spectroscopy which revealed a characteristics peak maxima at 430 nm. FTIR analysis revealed the possible biomolecules present in the leaves extract responsible for capping and stabilization of AgNPs. XRD analysis revealed the crystalline nature with particle size of 35.41 nm. SEM-EDS revealed the surface morphology and elemental composition with silver accounting for 71%. Dye degradation activity of AgNPs on congo red dye was also investigated which revealed that the degradation efficiency increased as concentration and exposure time increased with 80% efficiency achieved after after 6 hours. The result clearly shows the potential of ecofriendly synthesized AgNPs in enhancing the degradation of congo red dye.

Keywords: Silver nanoparticles, wastewater, Ecofriendly synthesis, Congo red dye, Photocatalysis, Dye degradation

INTRODUCTION

Most industrial effluent wastewater consist of several contaminants such as dye, heavy metals, antibiotics, pesticides, etc. [1,2]. Waste generated by textile, plastic, food, tanneries and pharmaceutical industries consist of toxic chemical dyes as part of the major pollutants including other toxic chemical components [3,4]. Some of the major dyes that are used by industries to impart color includes but not limited to; acidic, cationic basic and Azo [5].

Some of these synthetic dyes are highly toxic and carcinogenic and some of these industries discharge these dye containing effluent waste into nearby drains, rivers and lakes [6,7,8]. These effluent water generated and discarded by most of these industries are in turn used for irrigation

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purposes which in turn affects the quality of crops produced as well as becoming harmful to the soil as well as humans who consume such crops.[9]. Dye molecules can also affect aquatic lives when present in water bodies as it inhibits the penetration of sunlight into water bodies [10]. Thus, proper treatment of industrial effluent before being discarded cannot be over emphasized.

Recent developments in the field of nanoscience and nanotechnology have brought about significant attention as a result of its application in various fields such as agriculture, medicine, energy, climate including its application in the field of photocatalysis [11,12,].

In recent years, the use of nanoparticles for wastewater treatment has seen a steady surge as a result of its ability to be used as adsorbents filtration purposes [13]. This is due to the distinct property of nanoparticles having a high surface area to volume ratio [14]

Nanoparticles of metallic origin have been shown to have several physical and chemical characteristics and tendencies that aids researchers in harnessing and applying them in several field such as healthcare, food, textile, electronics, biosensors, food, environment and agriculture [15, 16]. Application of these nanoparticles of metallic origin in the degradation of dyes has drawn researches towards this field of study in recent years due to its photo catalytic tendencies [17]. Nanoparticles used for the photocatalytic degradation of dyes in the textile industries are synthesized using various processes which includes chemical, physical and biological routes [18, 19], but, these physical and chemical methods possess certain drawbacks such as high energy consumption, being toxic to the environment and complex synthesis procedures [20,21].

Therefore, alternative methods for the synthesis of nanoparticles is of necessity. An eco-friendly method for synthesis of silver nanoparticles uses biological entities such as fungi, bacteria, and plants extracts which is being used currently. Plant-mediated approaches are straightforward, cost-effective, and environmentally friendly [22]. A plant extract contains bioactive compounds such as polyphenols, alkaloids, steroids, flavonoids, and terpenoids which act as both reducing and stabilizing agents during synthesis of nanoparticles.[23,24,25].

AgNps have been synthesized using several plants including lantana camara, moringa oleifera, Menta piperita and others for their antifungal, antimicrobial and catalytic potential [26]

Lantana camara is a perennial species of flowering plant within the verbena family, native to the American tropics. It is a scrambling, thorny, evergreen shrub that can grow up to 2 meters tall and perhaps 2.5 meters wide. The plant is often used in domestic medicine and research carried out mainly in India has shown it to contain a number of compounds with medicinal activity. It is commonly cultivated as

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an ornamental in tropical gardens, where it is also grown as a hedge plant. It is a natural pioneer species, establishing in open places and providing a suitable habitat for rainforest trees to become established. The aromatic leaves are used to make a tea. It is a very adaptable species, which can inhabit a wide variety of ecosystems; It has spread from its native range to around 50 countries, where it has become an invasive species.

Several studies have investigated the catalytic tendencies of nanoparticles against several toxic dyes such as Congo red, Tymol blue, Auramine O, Rhodamine B dye, Phloxine B, Methly orange, etc, [27, 28]. Between 90–100% degradation was recorded for various dyes through silver nanoparticles. Also, dye degradation processes involving nanoparticles is fast and eliminates the risk of toxic chemicals that accompany physical as well as chemical wastewater treatment process [29]. In recent years, researchers have shown the efficient dye degradation potential of green synthesized AgNPs for methyl orange, methyl red and congo red [30], A study of the synthesis of AgNPs from Sophora mollis leaf extract demonstrated 88% degradation of methylene blue in 160 min [31]. Also, researchers reported in a separate study the promising potential of AgNPs for the degradation of methylene blue [32].

From observation, several literature highlighted the catalytic potency of silver nanoparticles on individual dyes using chemical and other physical methods only, however, these methods in turn tends to release more toxic chemicals back into the environment which has the potential of affecting the eco system. Therefore, it is very imperative to study the photocatalytic dye degradation potential of nanoparticles synthesized in an eco-friendly nature. The present study provides an easy and eco-friendly (plant mediated; using aqueous leaf extract of lantana camara) method for the synthesis of silver nanoparticles at room temperature, which was characterized for its surface plasmon resonance and morphological features such as shape and size using UV–visible spectroscopy, SEM, XRD, etc. The eco-friendly synthesized AgNPs was further investigated for its dye degradation potential. To the best of my knowledge, this is the first report to study the photocatalytic dye degradation potential of eco-friendly synthesized AgNPs using aqueous leaf extract of *Lantana camara* at room temperature on congo red dye.

MATERIALS AND METHODS

Materials

Silver nitrate salt, Congo red salt were of analytical grade and obtained from BDH Chemicals Ltd. Poole, England. Fresh leaves of Lantana camara was obtained from the green house of Plant Science and Biotechnology Department of University of Jos, positioned in plastic bags and transported to the laboratory. The stock solution for the dye was prepared by dissolving 0.069 g of dye in 100 mL of double distilled water

Methods

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Preparation of Aqueous Leaf Extract of Lantana Camara

Fresh 10 grams of uninfected leaves of *Lantana camara* was weighed and then washed several times with running tap water and rinsed with deionized water for removal of dust particles. Afterwards, the leaves was chopped into smaller pieces to obtain better yield of the extract. The chopped and weighed material was then be transferred into a 500 ml beaker containing 100 ml of deionized water and then heat on a hotplate at 45^{o}C. The mixture was then brought to boil for 10 minutes and allowed to cool after which it was filtered using Whatman filter paper No. 1. The filtrate was kept for onward use.

Preparation of 0.1 M $AgNO_3$ Salt Solution

1.70 grams of silver nitrate salt was weighed and transferred into a beaker containing 100 ml of deionized water. The mixture was stirred to ensure that the salt dissolved properly. The prepared salt solutions was stored for onward use.

Synthesis of Ag Nanoparticles

The silver nanoparticles was produced by reducing the precursor salt of silver, using aqueous leaf extract of *Lantana camara*. 10 ml of freshly prepared leaf extract was added drop wise to 50 ml of 0.1 M $AgNO_3$ solution with constant stirring on a magnetic stirrer. On addition of the extract and after a period of time, a color change from colorless to pale brown was observed indicating the formation of Silver nanoparticles nanoparticles (AgNps). The AgNPs formed was then centrifuged at 3000 rpm for 15 minutes, washed with distilled water, allowed to dry at room temperature and kept for further analysis.

Solution and Surface Characterization

The presence of the synthesized silver nanoparticles was confirmed by sampling the aqueous component using GENESYS 10UV UV-visible spectrophotometer. Distilled water was used as a blank. Absorbance measurement was carried out at a wavelength of 300-800 nm at a scan interval of 5 nm in order to obtain the UV-visible spectrum of the nanoparticles. The biomolecule responsible for the reduction of AgNPs was determined using Perkin Elmer Fourier transform infrared (FT-IR) spectroscopy. The x-ray diffraction (XRD) patterns and particle size was recorded on The XRD Empyrean Malverrn Panalytical Diffractometer. The voltage and current was set at 45kV and 40 mA, with temperature, set at 21- 23º C. The surface morphology of the biosynthesized AgNPs was also determined using Agilent 5500 Atomic Force Microscopy.

Catalytic dye degradation using silver nanoparticles

The photocatalytic dye degradation potential of AgNPs was evaluated for congo red. Before the dye degradation experiment, the dye solution (1 mM congo red solution) at various exposure time intervals was scanned between 200 nm and 800 nm to obtain absorbance maxima to serve as control for the analysis. 1 mg AgNPs was added

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to 5 ml of the dye solution to prepare a 200 ppm solution and exposed to sunlight. The dye degradation/photocatalytic study was performed with the prepared sample, it was scanned regularly between 300 nm and 800 nm at different time intervals of 2 hours, 4 hours, 6 hours and 8 hours to measure the change in intensity via the λmax. The λmax value was used to determine the percentage as well as rate of degradation. This procedure was repeated for 400, 600, 800 and 1000 ppm solution of AgNPs respectively. The degradation of dyes in terms of percentage was calculated as per the following equation (1) [33]:

$$\textbf{\textit{Dye degradation}}\ \% = \frac{\mathbf{Ao - At}}{\mathbf{Ao}}\ \boldsymbol{X}\ \mathbf{100} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots \textbf{(1)}$$

Where Ao denotes absorbance at zero time; At denotes absorbance at t time.

RESULTS AND DISCUSSION

Optical Property

The optical properties of silver nitrate solution, Lantana camara leaf extract, and silver nanoparticle are shown in plate 1. After addition of 10 mL of Lantana camara leaf extract, the color of 0.1 M silver nitrate solution changed from colorless to pale brown, then light brown, indicating the formation of silver nanoparticles. Silver nanoparticles are known to have a distinctive brown color. The excitation of the surface plasmon resonance action in silver nanoparticles is responsible for the color changes. The size and shape of metal surface plasma oscillations, as well as their optical properties, are well known.

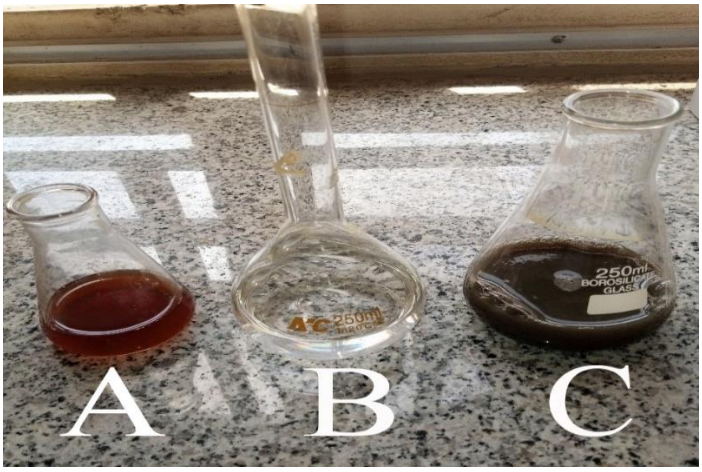

Plate 1: (a) Lantana camara aqueous leaves extract (b), silver nitrate solution (c), solution of silver nanoparticle

UV-Vis spectrophotometry

UV-visible spectroscopy was used to monitor the reduction of aqueous silver metal ions after treatment with Lantana camara leaf extract which acted as the reducing agent. The UV-visible spectrum of the reaction mixture indicated the presence of surface plasmon resonance (SPR)

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absorption band of approximately 425-435 nm with maximum absorbance at 430 nm which can be attributed to the presence of silver nanoparticles. The nearly symmetrical structure of the plasmon band can be attributed to the fact that the nanoparticles are no homogenous and also not uniformly dispersed. This non-uniformity of the nanoparticles might be responsible for the slightly broad absorption peak. The particle size, shapes, homogeneity and capping agent determines the position of the plasmon resonance.

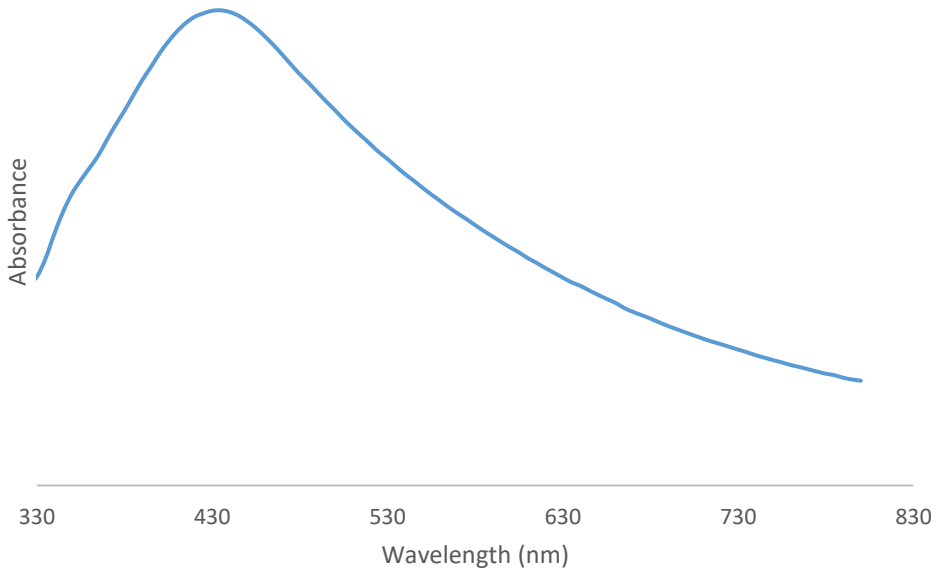

Figure 1: UV-visible spectrum of silver nanoparticle

Scanning Electron Microscope and Energy-Dispersive X-ray Spectroscopy Analysis

The SEM-EDS analysis was utilized to examine the surface roughness and morphology of the Ag nanoparticles as shown in plate 2. The morphology shows the intricacy of the particle size to be densely packed with slightly rough surface and revealed the cuboidal nature of the nanoparticles, this surface roughness might be attributed to biomolecules from the leaf extract that are bound to the Ag nanoparticles A similar shape was reported by Banerjee *et al.*, [34] in this work using tulsi extract. The EDS analysis revealed a strong signal in the silver region with a percentage weight of 71% and this confirms the formation of silver nanoparticles Elumalia *et al.*, [35]. The EDS profile also shows weak signals of oxygen, carbon, Silicon, sulphur, and gold, which may have originated from the biomolecules bound to the surface of the silver nanoparticle.

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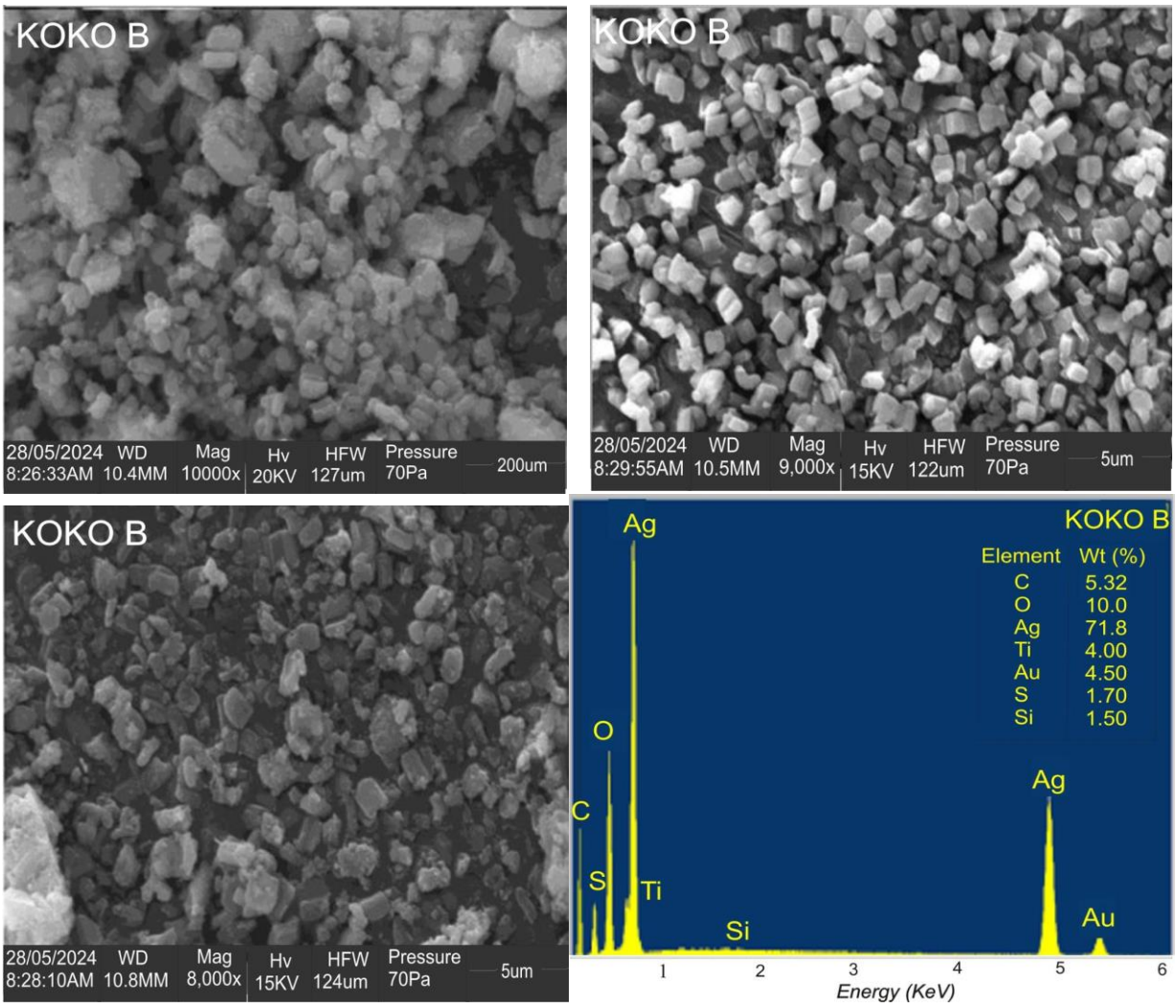

Plate 2: SEM-EDS Analysis of silver nanoparticles

Fourier Transform Infra-Red Spectroscopy (FTIR) analysis

Figure (2A and 2B) shows the FTIR spectra of Lantana camara aqueous leaf extract and silver nanoparticles respectively. The former shows the distinct absorption bands for the biomolecules present in the plant extract involved in the reduction of silver ions. Absorbance peaks were recorded at 3750, 3350, 2900, 1650 and 1050 cm^{-1}. The band at 3750 cm^{-1}, corresponds to hydroxyl (–OH) medium stretch for alcohols. Aliphatic amine N-H medium stretching is responsible for the bands at 33501cm^{-1}. The 2900 cm^{-1} can be attributed to C-H stretching for medium alkanes, C=C stretch for conjugated alkenes can be attributed to the bands at 1650 cm^{-1}. While 1050 cm^{-1} is attributed to N-H stretching for amides. From the FTIR spectrum of Ag nanoparticles

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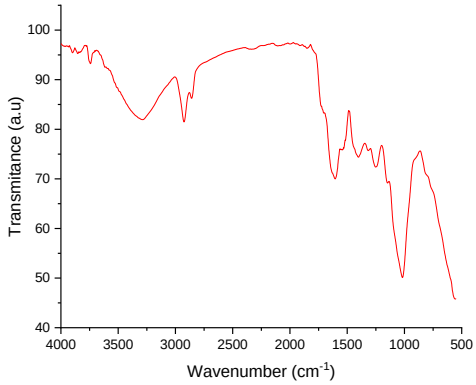
(figure 2B), it was observed that there was a marked reduction in the intensities of the peaks compared to that of the aqueous leaves extract of *Lantana camara* which suggests that the biomolecules present in the extract have been used up in the synthesis of the silver nanoparticles. Aqueous leaf extract of Lantana camara is known to contain tannins, alkaloids and carbohydrates and flavonoids which are rich in antioxidant properties capable of donating atoms of hydrogen and protein moieties that can bind with silver and capable of reducing Ag+ to Ag^0 and also serve as stabilizing agents to the synthesized AgNPs [36].



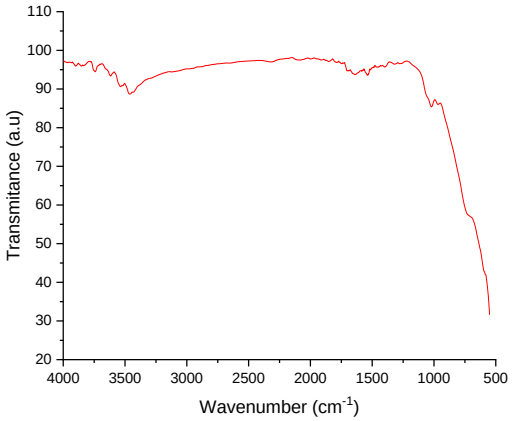


Figure 2A: FTIR spectrum of *Lantana camara* leaves extract. Figure 2B: FTIR Spectrum of Ag nanoparticles

X-ray Diffraction (XRD) Analysis

The XRD is used in the determination of the size, shape, fraction analysis and parameter determination of the sample, the position of the peaks obtained from diffraction pattern also gives information about the translational size and shape of the crystal. The average size of the silver nanoparticles was calculated using the Debye-Scherrer equation

$$D = \frac{K\lambda}{\beta\cos\theta}$$

Where, D= is the thickness of the crystal,

k= constant,

ƛ = wavelength of the X ray's incident on the crystal,

β= is the width at half maxima at (111) reflection at Bragg's angle 2θ, while θ is the Bragg angle and the constant (k) has a value of 0.94 and the wavelength (ƛ) of 1.5418

The XRD pattern of Ag nanoparticles with crystal planes of (111), (200), (220) and (222) showed diffraction peaks at 23, 29, 31 and 44 indexed as crystalline silver. The average size of the synthesized nanoparticles was determined to be 35.41 nm, which was calculated from (table 1). This corresponds with the average size the silver

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nanoparticles synthesized using Acalypha indica leaf extracts by Krishnaraj et al. [37]. The values of the crystals planes of the biosynthesized silver nanoparticle observed were compared with the standard powder and diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS) which showed that the biosynthesized silver nanoparticles exhibit a Face center cubic crystals. The other peaks might be as a result of some bioorganic compounds or proteins present in the plant extract. [38]

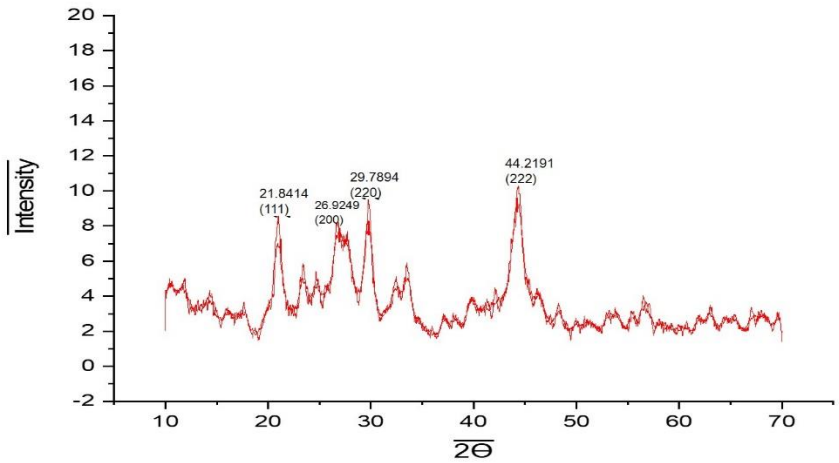

Figure 3: XRD spectrum of silver nanoparticle

Table 1: Average size analysis of silver nanoparticles from XRD analysis

Position (nm)	Height (Abs)	FWHM (radians)	d-Spacing (Å)	Particle size (nm)
12.135	5.101	0.725	0.179	9.201
21.184	9.123	0.636	0.137	20.977
23.681	5.890	0.720	0.317	26.955
26.924	8.023	0.210	0.278	29.795
29.789	9.207	0.265	0.387	33.089
33.416	5.602	0.425	0.167	44.270
44.219	11.001	0.328	0.241	56.659
57.321	4.000	0.352	0.195	63.047

Catalytic Activity of Silver Nanoparticles

Congo red dye is harmful and toxic to a wide variety of organisms including humans and can

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be carcinogenic and mutagenic. The efficiency of the photocatalytic degradation of Congo red dye by the eco-friendly synthesized Ag nanoparticles was investigated using UV-vis spectroscopy. (Figures 4A – 4E) shows the degradation of Congo red dye after addition of AgNPs of different concentrations (200, 400, 600, 800 and 1000 ppm), the degradation was observed at uniform time intervals of 2 to 8 hours in each case. Dye degradation was observe to increase with increase in concentration of nanoparticles and exposure time with maximum dye degradation of 80% observed at (1000 PPM) after 6 hours exposure time, complete degradation was not observed. Dye degradation process was observed to reduce after 6 hours at all concentration, this might be due to the binding of degraded materials to the nanoparticle surface. Shroog et al. [39] reported on the facile bio-fabrication of silver nanoparticles using Salvia officinalis leaf extract and its catalytic activity towards Congo red dye degradation. Rajkumar et. al. [40] also reported on the Bio-synthesized silver nanoparticles for effective photo-catalytic degradation of Congo red dye in aqueous solution: optimization studies using response surface methodology.

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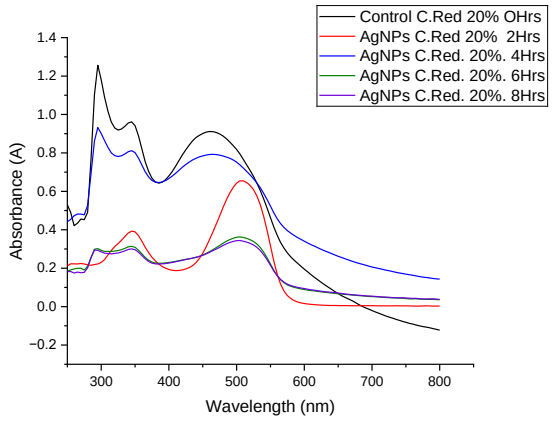
A



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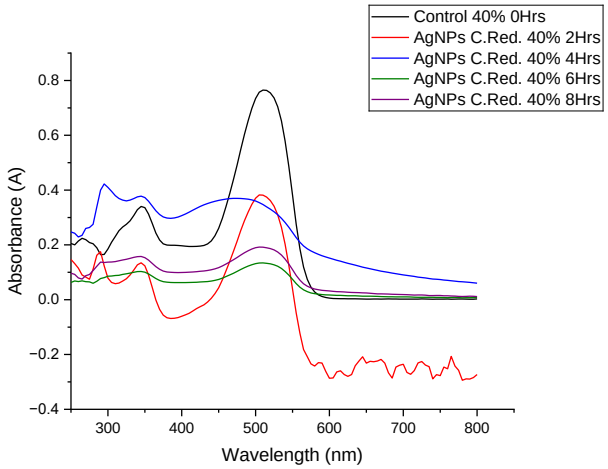

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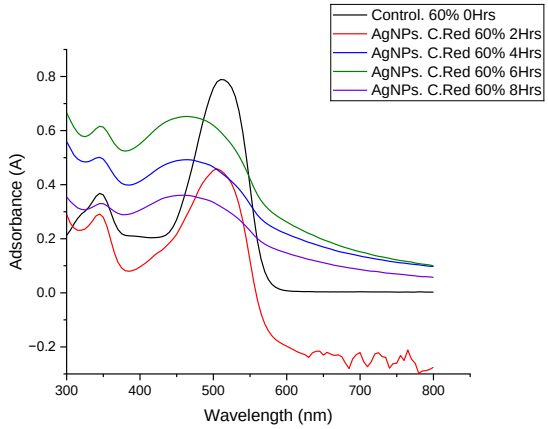

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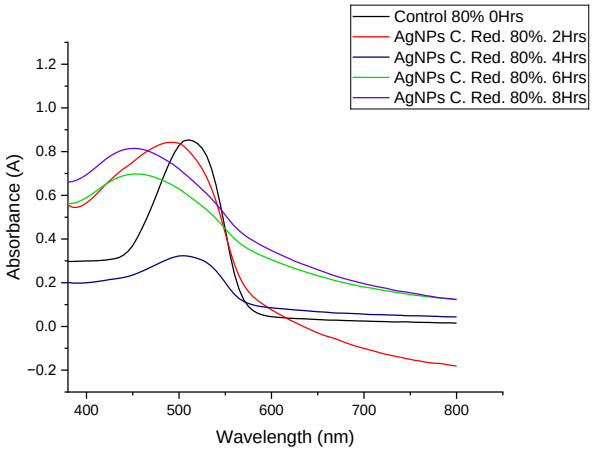

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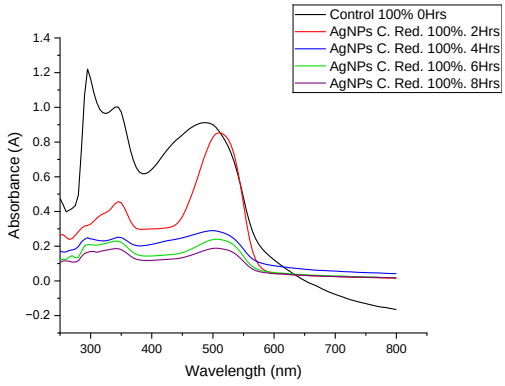

Figure 4 A-E: Photocatalytic dye degradation of Congo red dye at various concentrations of AgNPs and exposure time**.**

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CONCLUSION

In this study, aqueous leaves extract of *Lantana camara* was used via a facile and cost effective eco-friendly route at room temperature to synthesize silver (AgNPs) nanoparticles. The plant extract acted as a reducing and stabilizing agent to the nanoparticles owing to its rich biomolecule content. Photocatalytic dye degradation potential of the synthesized AgNPs showed about 80% degradation of Congo red dye after 6 hours exposure time. Furthermore, most literature studies have shown the use of other less eco-friendly methods of synthesis which are either expensive or toxic to the environment. Therefore, this study could serve as an indicator for researchers to explore more eco-friendly route in the synthesis of nanoparticles for large scale industrial application.

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