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Ecofriendly green synthesis of Ag@Cu bimetallic nanoparticles using *Seidlitzia stocksii* stem extract: photocatalytic degradation of methylene blue

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Abstract: This study focuses on the synthesis of silver/copper bimetallic nanoparticles (Ag/Cu BMNPs) using *Seidlitzia stocksii* stem aqueous extract. The synthesized product was characterized by powder X-rays diffraction techniques (XRD), Fourier transform infrared spectroscopy (FTIR), Energy dispersive X-rays spectroscopy (EDX), Scanning electron microscopy (SEM). The bimetallic nature of the particles was confirmed by XRD whereas the elemental composition was verified by EDX analysis. FTIR and SEM were used to determine the presence of different functionalities and morphology of the prepared BMNPs respectively. The photocatalytic degradation of methylene blue was analyzed using solar light. Nearly 44 % degradation was observed for 70 min of irradiation with excellent rate constant. Results revealed that the synthesized material is a potential candidate in the field of photocatalysis. Moreover, this may be contributed to presence of BMNPs. It may be concluded that the prepared material could be applied as cost effective and eco-friendly catalyst for the degradation of toxic pollutants from industrial effluents.

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Keywords: bimetallic nanoparticles; dye degradation; environmental remediation; green synthesis; photocatalysis; textile effluents.

1 Introduction

Nanotechnology is a developing multi-disciplinary expertise that includes the fabrication of particles in nanoscale range. It is the production, designing, analysis and use of methods, structures and tools by adjusting their dimensions [1]. Nanotechnology is an encouraging field of research developed in the current era and is required to give main instincts to technical revolutions in a variety of industrial regions in the future [2–5].

From the point of view of material science and chemistry, as the particle size is decreased within the nanorange, essential physical and chemical properties seem to change regularly often yielding totally new and distinctive physical and chemical properties [6]. NPS can be in different shapes, such as wires, planes, spheres, rods, quantum dots, dendrimers etc. Nano-materials of variable properties can be synthesized by sol-gel, hydrothermal, co-precipitation, micro-emulsion, emulsification as well as solvo-thermal techniques. Characterization techniques such as X-ray diffraction pattern, scanning electron microscopy, transmission electron microscopy, Fourier transform Infrared spectroscopy plus dielectric measurements are performed to study the shape, morphology, crystalline structure, elemental analysis and dielectric properties of NPs [7–9].

Many metals are used for the synthesis of nanoparticles. For example, gold, silver/copper bimetallic, aluminum, cobalt, iron and nickel NPs were also reported and showed magnetic and semiconductor properties. Ag/Cu NPs have results in noteworthy catalytic, biochemical activity and atomic trend compared with larger size particles having same chemical composition [10–12].

Researchers have been showing great interest in green chemistry for the production of nanoparticles. It has been observed that nanoparticles synthesized by microbes have some demerits as compared to that of plants. There are some limitations like microorganisms that require more time and protection for their growth. The size, morphology, and structure are not easily maintained. By using bacteria for the formation of NPs require much care and protection. Therefore, plants and dried grass are utilized for the synthesis of NPs because these are cheap, simple, easily available and do not require many precautions for growth [13–15]. Silver NPs has been synthesized by using different medicinal plants like *Saccharum officinarum, Aloe vera, Capsicum annuum, Zea mays, Magnolia kobus, A. vera, Helianthus annus, Basella alba, Cinnamomum camphoraza* and seed extract of grape [3, 9, 16]. It has been investigated that each part of a plant (seeds, fruits, leaf,

peel, and roots) has different functional groups like amino acids, steroids, flavonoids, glucosinolates, polysaccharides, alkaloids, organic acids, and phenolic groups act as reducing agents to reduce Ag^+ into Ag^0 to produce silver NPs [17].

In this present investigation, *Seidlitzia stocksii* plant extract was used for the synthesis and characterization of Ag/Cu BMNPs. The main objective is to study chemical constituents of *S. stocksii* stem that help in synthesis of size controlled green synthesis of new Ag/Cu NPs using aqueous extract. Furthermore, the study is extended to find out the photocatalytic potential of eco-friendly synthesized Ag/Cu BMNPs.

2 Materials and methods

Analytical grade reagents, highly calibrated glassware and instruments have been employed during this scientific research project. The reagents were purchased from Sigma Aldrich. Plant material (*S. stocksii* stem) was collected from Bahawalpur area, Southern Punjab, Pakistan. The impurities, dust and other contaminants over the surface of plant sample was washed with tap water thoroughly to make sure the cleanliness of sample. The same procedure was followed with double distilled water.

The experimental plant after washing was kept under shade for drying at room temperature. The dust free glass environment was provided to *S. stocksii* for drying. After drying, the sample of *S. stocksii* stem was subjected to grinding and crushing into fine particles size by using grinder and the powder was kept in a sealed container for further experiments [9, 18].

2.1 Plant extraction

The plant extraction was done by using deionized water. The 15 g of finely powdered plant was dipped in 300 mL of deionized water in a dried beaker of volume 500 mL. It was stirred continuously by using magnetic stirrer hot plate for 6 h. During the first hour of extraction it was stirred as well as heated at constant temperature at 60 °C. In next 5 h of extraction, it was just continuously stirred only at room temperature. The mixture was then filtered to get the water extract filtrate of sample and kept in incubator at 4 °C for further experimentation.

2.2 Green synthesis of Ag/Cu NPs

Silver nanoparticles was synthesized by using precursor $AgNO_3$ salt. Green synthesis of Ag/Cu NPs was established using aqueous extract of *S. stocksii* stem for the reduction of Ag^+ to Ag^0 as well as the extract showed good capping and stabilizing abilities. Ag/Cu NPs samples were prepared by keeping the plant extract concentration constant while the concentration of salt was varied. For this purpose, 0.01 M solution of silver nitrate was prepared by adding 1.6987 g in 1000 mL of deionized water. This transparent silver nitrate solution was kept in labeled conical flask A. For copper sulfate pentahydrate, 2.4968 g of salt was added in 1000 mL of deionized water to prepare 0.01 M solution of $CuSO_4{\cdot}5H_2O$ and kept in labeled conical flask B. Two conical flasks labeled A and B ware wrapped by aluminum foil to avoid photochemical decomposition. Both solutions were

mixed. Then 200 mL aqueous extract of *S. stocksii* was added with the help of burette to 500 mL of above mixed solutions. 20–25 drops of *S. stocksii* were added slowly. Resulting solution stirred continuously for half an hour at room temperature till the dark brown color appeared, which apparently indicates the green synthesis of Ag/Cu NPs. Moreover, both samples were kept in complete darkness overnight in order for complete reduction Ag^{+} to Ag^{0} as well as stability of nanoparticles. Then NPs were poured into Falcon conical tube up to level 10 mL volume for the extraction of nanoparticles by centrifugation. Centrifugation was done at 6000 rpm by centrifuging machine for 30 min. The centrifuged solution was preserved in brown glass bottles and extracted nanoparticles were washed with doubled distilled water. After washing, sonication was performed for 15 min. Finally, the powdered Ag/Cu NPs was dried by evaporating at room temperature, weighed and kept in incubator at 4 °C in order for further characterization.

2.3 Characterization of Ag/Cu NPs

After the completion of reactions, the Ag/Cu BMNPs were subjected for analysis and characterization. The accurate ultra violet-visible (UV–Vis) spectroscopic analysis allows the further proceedings of characterization. Other techniques like X-ray diffraction XRD, Fourier Transform Infrared (FTIR) and scanning microscopy (SEM) were conducted for the characterization of Ag/Cu NPs [19–22].

2.4 Photocatalysis

The photocatalytic degradation experiment was done using 5 ppm methylene blue aqueous solution. The solution was prepared and 5 mg of Ag/Cu bimetallic nanoparticles were added and the suspension was stirred continuously for 2 h under dark. The purpose of taking solution under dark with constant stirring is to establish total adsorption-desorption phenomenon. After the completion of this step, the suspension was taken under ultraviolet light and suddenly 5 mL of the suspension was taken, centrifuged to remove nanoparticles and then UV–visible absorption spectrum was taken. Similar procedure is adopted after each and every 10 min [9, 17, 23].

3 Results and discussion

The UV-visible spectra of Ag/Cu bimetallic nanoparticles were taken at different concentrations. Dilute solution of synthesized Ag/Cu BMNPs was subjected to UV-Visible spectrophotometer to check the complete reduction of Ag^{+} to Ag^{0} and color change by indicating sharp absorbance peak between 260 and 300 nm. The spectrum was run in range from 200 to 800 nm (Figure 1). The spectrophotometer was calibrated by standard deionized water showing absence of any absorbance peak [24]. The first indication for the synthesis of Ag/Cu NPs is visual color change form light yellowish to brownish color which was developed by continuous stirring. The absorption spectra showed a sharp absorption band between 260 and 300 nm. This red shift in absorption spectra was due to the formation of metallic composite.

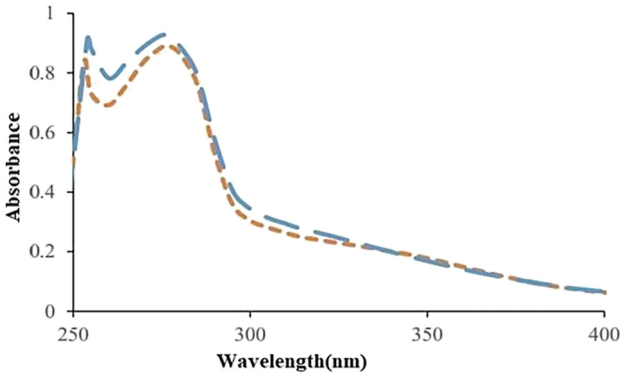

Figure 1: UV–visible spectra of Ag/Cu BMNPs taken at two different concentrations.

This unique characteristic of material was responsible its significant degradation efficiency.

3.1 Fourier transform infrared spectroscopy

FTIR spectroscopic analysis describes the functional group present in the powdered sample of nanoparticles. The basic principal of FTIR depends on different types of vibrational movements present in the different functional groups. This technique encounters the capping and stabilizing functional groups present in samples. 10 mg of both samples were prepared to meets the standard procedure for FTIR spectroscopic spectrum. In the range of 4500–500 cm^{-1} of transmittance mode operating spectrum was recorded [25]. The FTIR spectra of Ag/Cu bimetallic nanoparticles are shown in Figure 2. The peak around 3390 cm^{-1} is due to stretching vibrational modes of OH group. Whereas the peak around 1650 cm^{-1} corresponds to C–O stretching vibrations. Similarly, the peak around 1330 cm^{-1} was due to existence of CH_3 bending vibrations. The existence of these peaks were due to presence of residual solution in the final product.

3.2 X-rays diffraction analysis

XRD plays an important role in determining the crystal and powdered sample morphology. The particle size, shape, geometry, symmetry, crystalline and amorphous nature is comprehensively explained with the help XRD spectra [24]. 100 mg

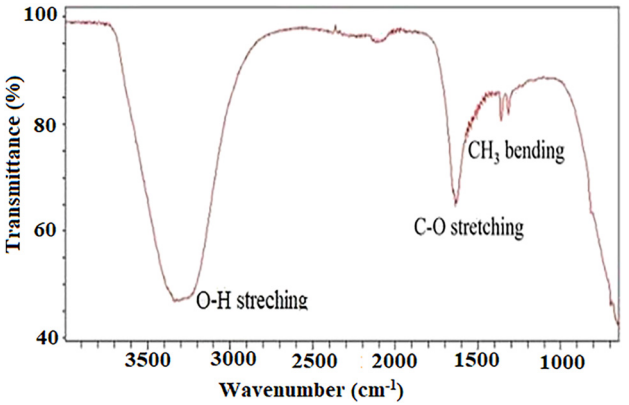

Figure 2: FTIR spectrum of Ag/Cu NPs showing different functional groups.

quantity of powdered Ag/Cu NPs sample was subjected to record XRD spectrum. Size of nanoparticles easily can be calculated with the help of Debye-Scherer equation;

$$L = K\lambda/\beta \cos\theta$$

In this equation, K = Scherer constant; λ = wavelength of X-ray; β = Bragg angles; θ = half width of peak. This equation helps in measuring the particles size and geometry. The results indicate comparative compatibles among size calculated with XRD spectra and SEM images. The structural elucidation and phase identification were completed by means of XRD. The XRD diffraction pattern of Ag/Cu bimetallic

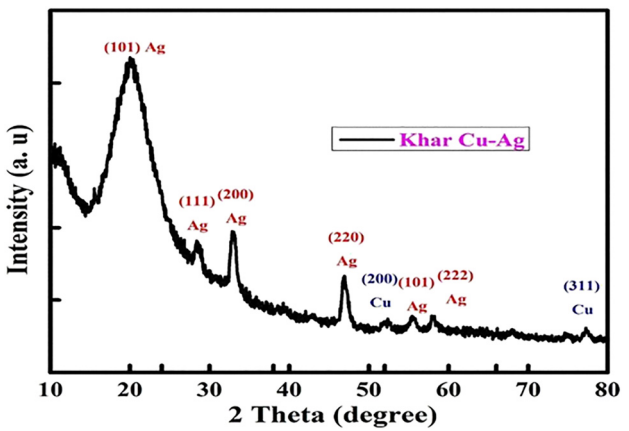

Figure 3: XRD diffraction pattern of Ag/Cu BMNPs exhibiting crystallinity of NPs.

nanoparticles is shown in Figure 3. The diffraction pattern was plotted between 2 Theta on *x*-axis and intensity on *y*-axis. The XRD diffraction pattern showed the characteristic peaks of both Ag and Cu in the material. The peaks corresponded to (101), (111), (200), (220), (101), (222) *hkl* values were due to Ag phase whereas the peaks corresponded to (200) and (311) *hkl* values were due to Cu phase in the material. As the single material is composed on two different phases, so we have claimed it as Ag/Cu bimetallic nanoparticles.

3.3 Scanning electron microscopy (SEM) and EDX

SEM image plays an important role in evaluating the morphology and size of Ag/Cu NPs. It has been a powerful tool in this perspective to determine the crystalline and amorphous nature of nanoparticles. The energy dispersive X-ray spectroscopy (EDX) analysis was performed for elemental silver analysis [26]. The SEM and EDX data was recorded of each sample by using SEM from LUMS. The SEM micrograph is shown in Figure 4. The morphology of the product can be easily recognized by the image. The biconcave morphology of the product was observed. The morphology is similar as to the red blood cells. Moreover, the aggregation of the particles is also observed. The biconcave like morphology is most suitable due to its greater surface to size ratio and thus will act excellent candidate for the degradation of organic dyes.

The energy dispersive X-rays (EDX) analysis was performed to analyze the elemental composition of the material. The EDX spectra of the Ag/Cu bimetallic nanoparticles are shown in (Figure 5). It can be seen that the material contains both Ag and Cu with 54.7 % and 16.4 % respectively. The remaining impurities includes carbon, Oxygen and chlorine with 1.7 %, 1.3 %, 0.61 % respectively.

3.4 Dye degradation

The degradation profile of methylene blue dye under ultraviolet light is shown in Figure 6. It can be noted that the absorption intensity of methylene blue dye is going to decrease with the passage of time and after 70 min. When the time was zero, then the absorption of methylene blue dye at wavelength maximum 664 was 1.38 whereas after completion of 70 min, the absorption of dye was dropped down to 0.78. Thus, the total degradation of dye solution with in 70 min of ultraviolet light irradiation was found to be 44 %. This comparable value to percent degradation makes this catalyst as a potential candidate in the field of photocatalysis. Moreover, this efficiency was attributed to bimetallic nature of the material that consist of

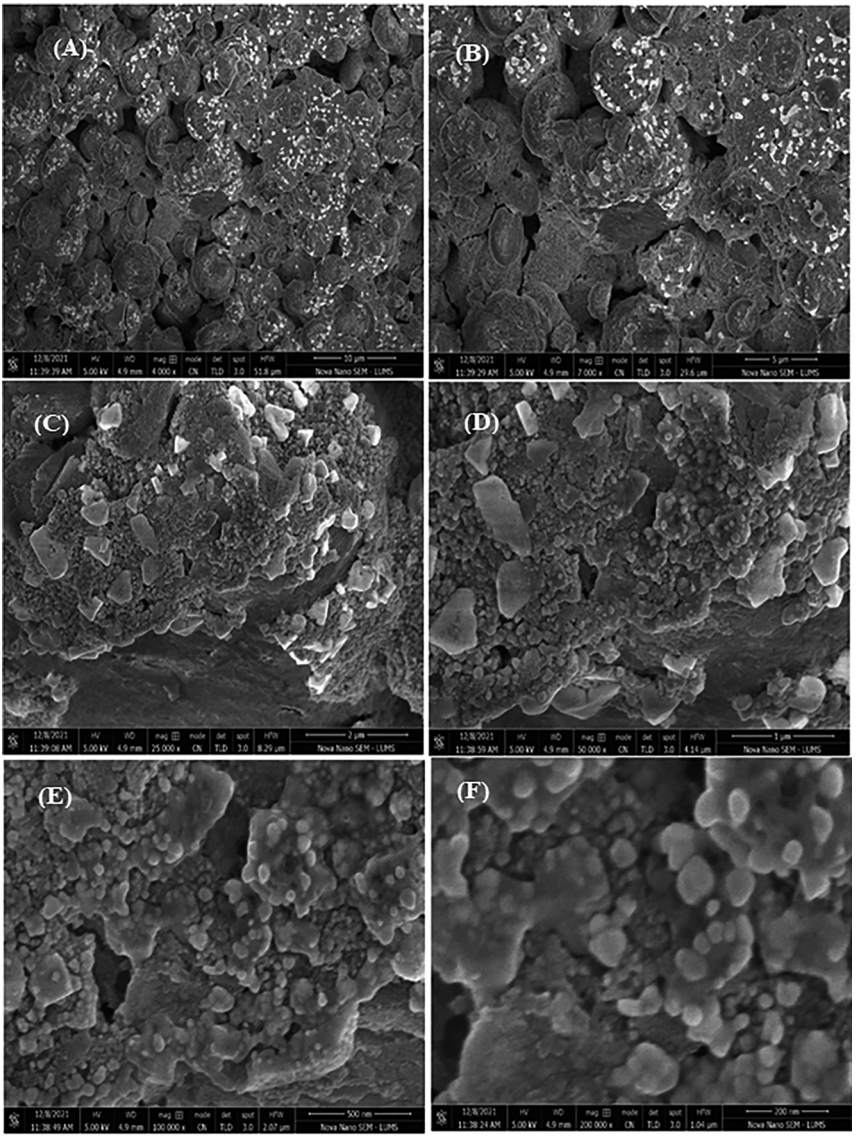

Figure 4: SEM micrograph of Ag/Cu bimetallic nanoparticles showing the morphology of NPs.

both Ag and Cu phases within the same matrix. The graph between time versus A/A_o (depicted in Figure 7) is also plotted that shows the relative decrease in intensity of material with the passage of time.

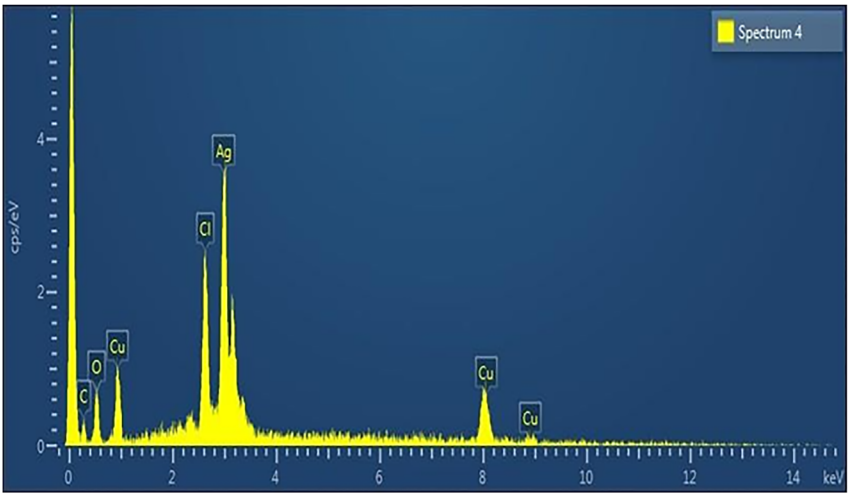

Figure 5: EDX spectrum of Ag/Cu bimetallic nanoparticles illustrating elemental composition.

Figure 6 shows the increase in percent degradation with the passage of time. Furthermore, the kinetics of the degradation experiment was also studied by applying pseudo first order rate kinetics. The graph between time on *x*-axis and $-\log(A/A_o)$ on *y*-axis was plotted and shown in Figure 7. The slope of the graph is corresponded to rate constant of the reaction. The 0.0089 min^{-1} was found the rate constant of the reaction.

Previous studies report the preparation of NPs from different plant sources and their application as a photocatalyst to degrade toxic pollutant from industrial effluents. Nair et al. [27] have successfully managed to develop ecofriendly technique for the preparation of well characterized shaped nanoparticles. The most favorable method for this purpose to use organisms. Plants must be placed at top priority among the other candidate for biosynthesis as they are appropriate for large scale biosynthesis of metallic nanoparticles. Synthesized nanoparticles are most stable by using plant and provide easy, faster and environment friendly method then using microorganisms. Furthermore, the nanoparticles have different shape and size as compared to those synthesized by other organisms. The sequence and mechanisms of metal ions uptake, bio-reduction by plant and to draw the possible mechanisms for the synthesis of metallic nanoparticles in plant, by using their different parts of plants has developed much interest in mind of researchers.

Torresdey and his collaborators have used Alfalfa sprouts plant for the first time to synthesize of metallic nanoparticles. They have provided a comparison of different parts of plant extract to the synthesis of Ag nanoparticles. It was found that the roots

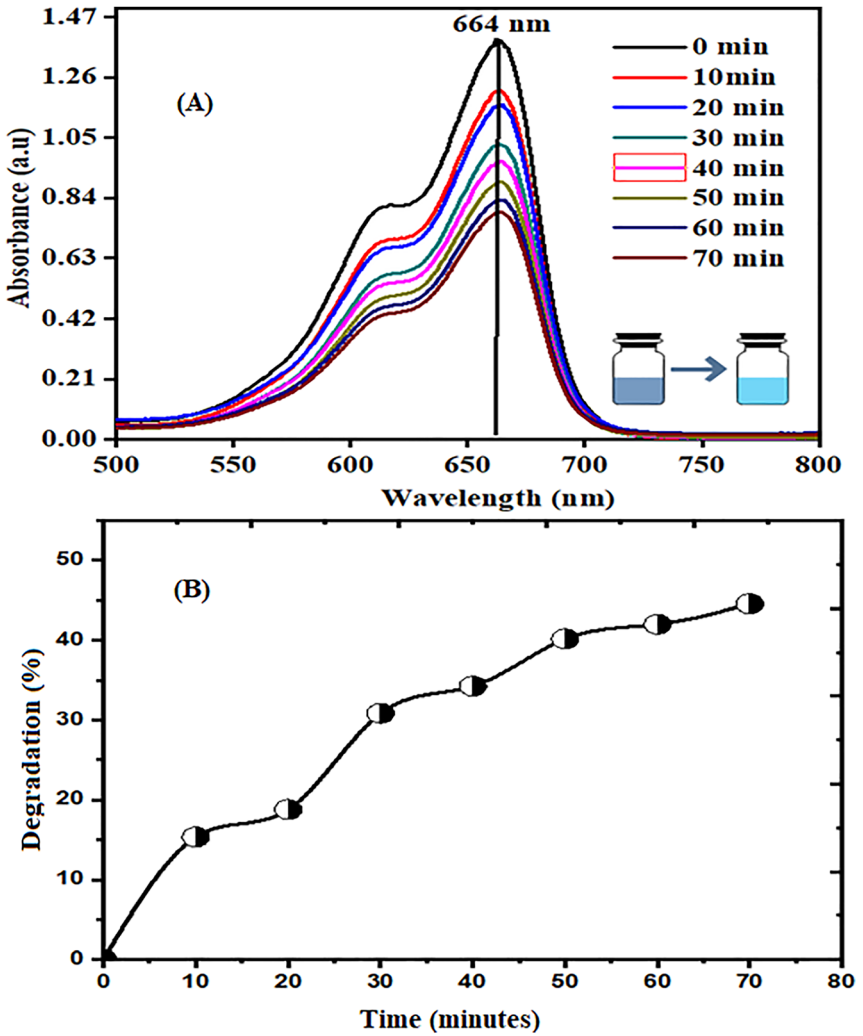

Figure 6: Degradation profile of methylene blue with the passage of time.

of this plant has maximum capabilities to absorb silver and to supply it in different parts of plants cells and organs in same oxidation state [28]. Ankamwar et al. [29] have proposed a series of plants extract for the green synthesis of both Ag, Au and their bimetallic NPs. It was found that *Emblica officinalis* fruit extract showed good stability toward metallic nanoparticles extracellularly. Similarly, Ghodake et al. [30] have also found that the fruit of this plant could be used for biosynthesis of triangular

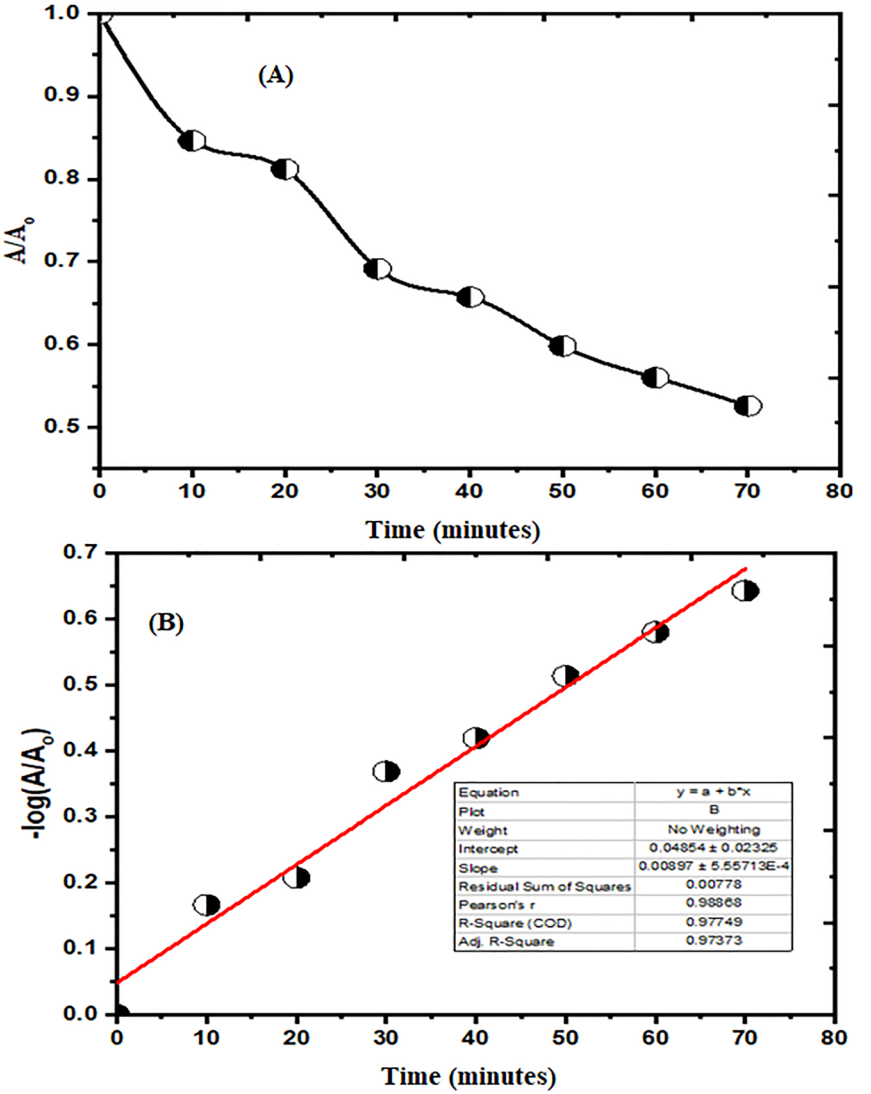

Figure 7: Degradation of methylene blue (A) decrease in absorption with the passage of time (B) reaction kinetics.

and hexagonal structure of noble metal nanoparticles. The ability of this plant to reduce metal is due to the presence of many important classes of compounds like emulsifying agents, organic acid, peptides, proteins, carbohydrates and amino acid.

Khan et al. [31] have synthesized Ag/Fe_2O_3 bimetallic nanocomposite using Algaia Monozyga leaves extract. The synthesized product was tested for biocidal and photocatalytic applications. The biocidal applications were evaluated using *E. Coli* and *Basilus subtilis* whereas photocatalytic degradation efficiency was tested using methylene blue dye as a source of ideal pollutant. Fauzia et al. [32] have synthesized Au/Ag bimetallic nanoparticles on ZnO surface using hydrothermal methods. The product was tested for degradation of organic pollutants using ultraviolet light. The product experienced higher degradation efficiency resulted due to the reduction in recombination of photo emitted species.

Sheny et al. [33] have synthesized trimetallic Gold-Ag/Cu NPs using leaf extract of *Anacardium occidentale* at lower to higher temperature in order to achieve optimum condition for the synthesis. The reaction at greater temperature requires less leaf extract for the synthesis of stable nanoparticles as compared to that of reaction at low temperature. It is also observed more stable and bigger size nanoparticles can be synthesized at higher temperature with increase in reduction ability as well.

Dubey et al. [34] have proposed that the metal ion concentration helps to get a proper shape, size and reduction processes of nanoparticles. Various concentration of metal ion (1–3 mM) was used for the synthesis of gold and Ag/Cu NPs by using tansy (*Tanacetum vulgare*) fruit extract. It was examined that the bigger size Ag/Cu NPs was obtained by increasing silver ion concentration, which also leads to the increase in absorption peak of Ag/Cu NPs while increase in Au ion concentration develop sharpen peak up to 2 mM Au ion concentration followed by decrease in absorption peak at 3 mM. In comparison with Gold and Ag/Cu NPs, Au NPs has bigger size then Ag/Cu NPs which was confirmed by TEM. Similar results were reported by Dwivedi and Gopal on Ag/Cu NPs and Au NPs by *Chenopodium album* leaf extract [35].

4 Conclusions

The Ag/Cu BMNPs were successfully synthesized by simple biological method. For the synthesis of material, *S. stocksii* stem aqueous extract was used for emulsifying and charge neutralizing agent. The synthesized product was examined by XRD, FTIR, SEM and EDX. The XRD results showed that the material consists of binary phases. The Ag and Cu phases are found in the XRD result and thus we have claimed it as Ag/Cu bimetallic nanoparticles. Furthermore, the EDX results supported the XRD results. The FTIR spectra shows the presence of different organic nature impurities that were also detected in EDX spectra. The SEM results showed the biconcave morphology similar to our red blood cell. The photocatalytic degradation efficiency of the material was tested using sun light. It was found that nearly 44 %

degradation of methylene blue dye takes place within 70 min of irradiation. It is concluded that these synthesized NPs could possibly be potential candidate for degradation of toxic pollutants.

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