GREEN SYNTHESIS SILVER NANOPARTICLES USING SALIX SEMI-SOLID EXTRACT FOR PHOTOCATALYTIC EFFECT

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ABSTRACT

The silver nanoparticles are synthesized by the green method using Salix semi-solid extract. Fresh leaves are prepared in a semi-solid extract form that retains active substances and can be preserved for a long time. The parameters of the synthesis process are optimized by examining the effects of pH, Salix semi-solid extract concentrations of extract and $AgNO_3$ concentrations through the UV-Vis absorption spectrum of synthetic particulate solutions. The composition of atoms and surface functional groups are determined through EDX measurement and FTIR infrared absorption spectrum of Salix semi-solid extract and synthetic silver nanoparticles. Silver nanoparticles are used to study the biodegradability of organic coloring Rhodamine B (RB). The results showed that silver nanoparticles synthesized by this green method provided an optical degradation efficiency of up to 88.5%. This work shows that silver nanoparticles synthesized from Salix semi-solid extract can be used in the decomposition of dye waste that contributes to the cleansing of the water environment.

TỔNG HỢP CÁC HẠT NANO BẠC BẰNG PHƯƠNG PHÁP XANH SỬ DỤNG CAO CHIẾT LÁ LIỄU CHO CÁC ỨNG DỤNG QUANG XÚC TÁC

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TỪ KHÓA

Phương pháp xanh
Hạt nano bạc
Cao chiết lá liễu
Photocatalytic
RB

TÓM TẮT

Các hạt nano bạc được tổng hợp bằng phương pháp xanh sử dụng cao chiết lá liễu. Lá tươi được chuẩn bị dưới dạng cao chiết xuất dạng mềm để giữ lại các hoạt tính và có thể được bảo quản để sử dụng trong một thời gian dài. Các thông số của quá trình tổng hợp được tối ưu hóa bằng cách kiểm tra tác động của pH, nồng độ dịch cao chiết và nồng lượng $AgNO_3$ thông qua quang phổ hấp thụ UV-Vis của các dung dịch hạt tổng hợp được. Thành phần của các nguyên tử và các nhóm chức năng bề mặt được xác định thông qua phổ tán sắc năng lượng EDX và phổ hấp thụ hồng ngoại FTIR của dịch cao chiết và hạt nano bạc tổng hợp được. Các hạt nano bạc được sử dụng để nghiên cứu khả năng phân hủy sinh học của màu hữu cơ Rhodamine B (RB). Kết quả cho thấy các hạt nano bạc được tổng hợp bằng phương pháp xanh này có hiệu quả phân hủy quang học chất màu RB lên đến 88,5%. Công việc này cho thấy các hạt nano bạc được tổng hợp từ cao chiết lá liễu có thể được sử dụng trong việc phân hủy các chất thải nhuộm góp phần làm trong sạch môi trường nước.

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1. Introduction

Silver nanoparticles are widely utilized in a variety of industries. They may be found in a variety of consumer goods, from clothes to biomedical applications including medication delivery and biomolecules, and sensors [1] – [4]. The most prominent application of silver nanostructures is their ability to resist and kill bacteria, viruses, and fungi [5] – [8]. Silver nanostructures can release Ag^+ ions, cling to cell walls and membranes, breaking the bacterial shell [9] – [12]. Additionally, respiratory enzymes that generate reactive oxygen but obstruct the synthesis of adenosine triphosphate can be rendered inactive by silver ions. Deoxyribonucleic acid (DNA) can be changed and cell membranes can be damaged by this [13] – [15]. In addition, silver nanoparticles can accumulate in holes forming on the cell wall after they cling to the cell surface causing cell membrane deformation. At the same time, silver nanoparticles can penetrate bacterial cell walls and then change the structure of cell membranes [16], [17]. Numerous studies have shown that the bacterial destruction of silver nanostructures depends on particle shape and size [18] – [20]. Among them, bridge-shaped silver nanoparticles are good candidates for resistance and bacterial destruction.

Not only do silver nanostructures act as catalysts under the stimulation of light that disintegrate toxic organic colors [21] – [23]. Under the influence of light, silver nanostructures can release electrons, these electrons in a state of stimulation when interacting with electromagnetic waves, forming a pair of electrons - holes. The OH^- radicals act as oxidizing agents that break down organic color molecules. This nature of silver nanostructures makes them optical catalysts that break down organic dyes in dye wastewater, contributing to the purification of the water environment. For purposes aimed at the application of the decomposition of organic colors, silver nanoparticles synthesized by the green method are preferably used at an environmentally friendly level. Silver nanoparticles can be synthesized using liquid extracts of plants, fungi, and bacteria [24] – [29]. Each type of extract gives different synthetic results depending on the nature of the extract and how the parameters are adjusted during the synthesis process.

In the Salix semi-solid extract, there are organic compounds such as phenolics, flavonoids and steroids [27]. In addition, the leaf extract also has ingredients such as hydroxyl phenol, coumarin, lactone, flavonols, quinone, saponin polyphenol, triterpenes, and steroid compounds [29]. At the same time, according to some studies, Salix semi-solid extract has antioxidant activities. Therefore, the use of Salix semi-solid extract instead of fresh Salix leaf extract helps to keep the activities of willow leaves stable over time and helps silver nanoparticles to be more stable in the solution after synthesis. In this article, we synthesize silver nanoparticles (S.S.AgNPs) using *Salix* semi-solid extract and optimize reaction participation parameters while studying the optical catalysis of silver nanoparticles with RB dye.

2. Experiment

2.1. Materials

Silver nitrate ($AgNO_3$, 99%), Sodium hydroxide (NaOH, 98%), and Hydrochloric Acid (HCl, 37-38%) were purchased from Sigma Aldrich. Rhodamine B ($C_{28}H_{31}ClN_2O_3$, 99%) and Ethanol (C_2H_6O, 99.5%) was provided by Merck, Germany. The deionized water (DI) with a resistance of 18.2 MΩ was used in all processes. Fresh Salix leaves were obtained from the experimental garden of Thai Nguyen University of Education.

2.2. Method

Two kg of fresh samples of Salix leaves were chopped, dried to obtain 1 kg dry and refluxed with 99.5% ethanol at 80 °C for 4 hours, distilled to recover the extracted solvent. Finally, the extract was stored at 4 °C to synthesize S.S.AgNPs. To find the optimal parameters for the synthesis process, we changed the pH of the extract with NaOH and HCl solutions from the initial pH value of pH 4 to pH 8. After selecting the optimal pH value for the reaction, $AgNO_3$ concentrations and volumes of *Salix* semi-solid extract were surveyed. $AgNO_3$ concentrations vary with values of 0.8 mM, 0.9 mM, 1.0 mM and 1.2 mM while the volume of the *Salix* semi-solid extract is constantly 1.5 ml. When the constant concentration of $AgNO_3$ was 1.0 mM, the volume of *Salix* semi-solid extract was altered with values of 0.25 ml, 0.5 ml, 1.0 ml, 1.5 ml and 2.0 ml. At the same time, to know the appropriate reaction time, we surveyed the absorption spectrum of the solution at reaction times from the 40th minute to the 120th minute.

2.3. Photocatalyst applications

S.S.AgNPs are also used as catalysts to investigate RB's optical degradation under the stimulation of white light due to a 50W LED light source (λ > 400 nm, λ_{max}= 464 nm) [30], 6500 K color temperature. The obtained S.S.AgNPs were centrifuged to remove the residues after the reaction and re-dispersed in deionized water with the same initial volume. In each experiment, 2 ml of S.S.AgNPs solution was added to 50 ml of 10 ppm RB solution. The mixture was stirred in the dark about two hours for absorption equilibration. The mixture was then illuminated with a white light source in the presence or absence of a 20% H_2O_2 reducing agent. The thermal equilibrium of the system is maintained throughout the reaction by exposing the reactor vessel to heat with water. The temperature of the system is always checked with a thermometer. The decrease in RB absorption was assessed on the UV-Vis-absorption spectrum in a lighting time of approximately 2 hours.

3. Results and discussion

Figure 1 is the absorption spectrum of the S.S.AgNPs solution obtained when changing the pH parameters, the volume of *Salix* semi-solid extract, $AgNO_3$ concentration, and reaction time. The results showed that the pH of the *Salix* semi-solid extract had a major influence on the S.S.AgNPs. Absorption spectra without a clear peak in the visible area demonstrate the very low synthesis performance of S.S.AgNPs when the pH of the solution is equal to 4. When the pH of the solution is greater than 5, the absorption spectra have a characteristic form of silver nanospheres with a resonance peak at 416 nm, corresponding to silver nanoparticles with a diameter of about 30 nm. However, the highest absorption peak intensity and the narrower full width at half maximum (FWHM) are at pH 7. This suggests that S.S.AgNPs form the highest performance when synthesized in pH 7 neutral environments (Figure 1a).

Absorption in the near-external region is the contribution of the *Salix* semi-solid extract. The more extracts in the solution, the higher the absorption in this area. The results in Figure 1b show that the extracted volume of 0.5 ml is the most reasonable to synthesize S.S. AgNPs uniformly in shape and size. This is demonstrated by the narrow absorption spectra and the greatest absorption strength at 416 nm. Similarly, when analyzing the absorption spectrum in the case of changes in $AgNO_3$ concentrations, we observed 1.2 mM $AgNO_3$ concentrations for the highest synthesis performance (Figure 1c).

The reaction was monitored for approximately 2 hours using the absorption spectrum (Figure 1d). The results showed that the spectrum shape did not change much from 40 minutes to 120 minutes. This suggests that the reaction occurs relatively quickly compared to other green synthesizing methods, especially those using fungi or bacteria with reaction times of up to several days [31].

The size and size distribution of S.S.AgNPs were investigated through DLS particle size measurement and TEM images (Figure 2). The results show that the S.S.AgNPs particles have an average hydrodynamic diameter of 31.50 nm with a PdI index of 0.20. The hydrodynamic size is also equivalent to the TEM size of 30 nm ± 5 nm. This shows that the obtained S.S.AgNPs have an average size of about 30 nm (consistent with the size predicted from the absorption spectrum) and are well distributed in solution.

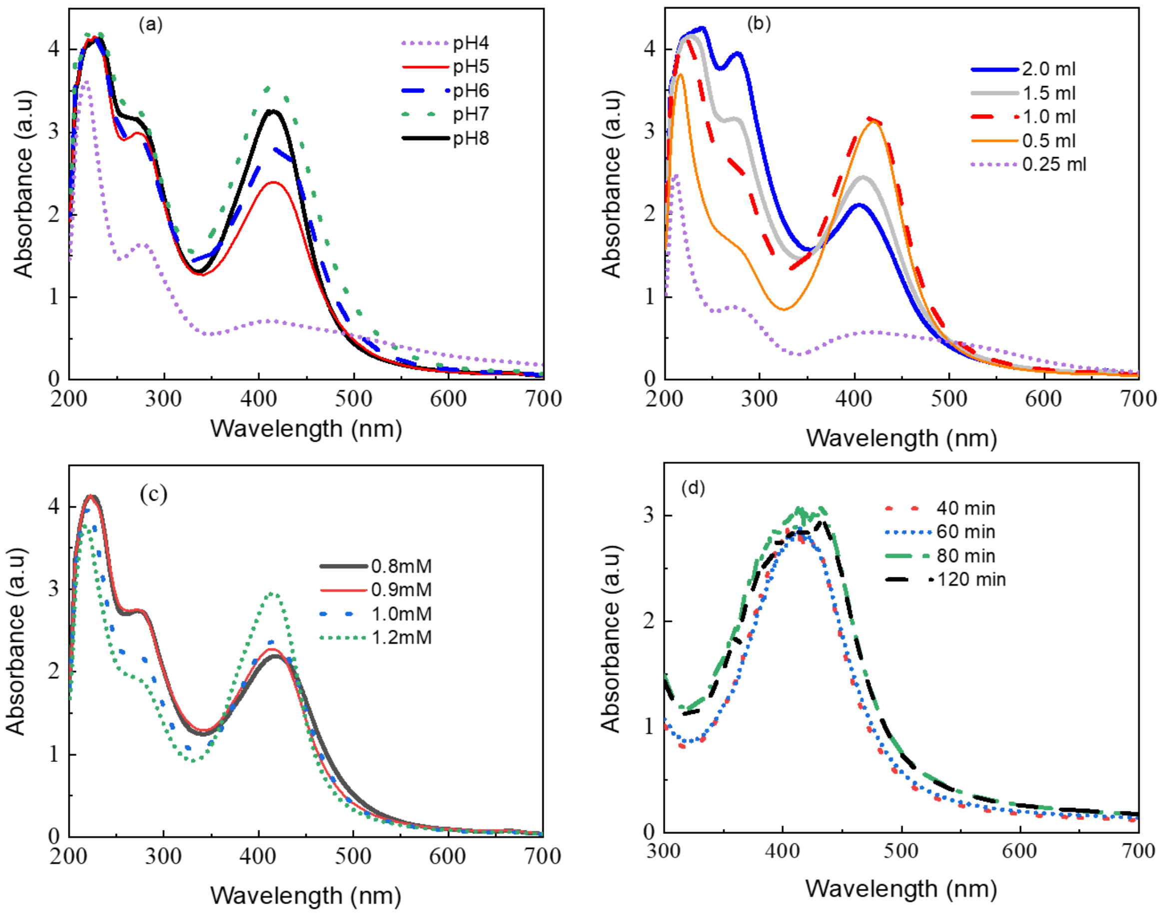

Figure 1. *UV-Vis absorption of S.S.AgNPs solutions obtained when the pH of the extract (a), the volume of the extract (b), $AgNO_3$ concentration (c) and reaction time change (d)*

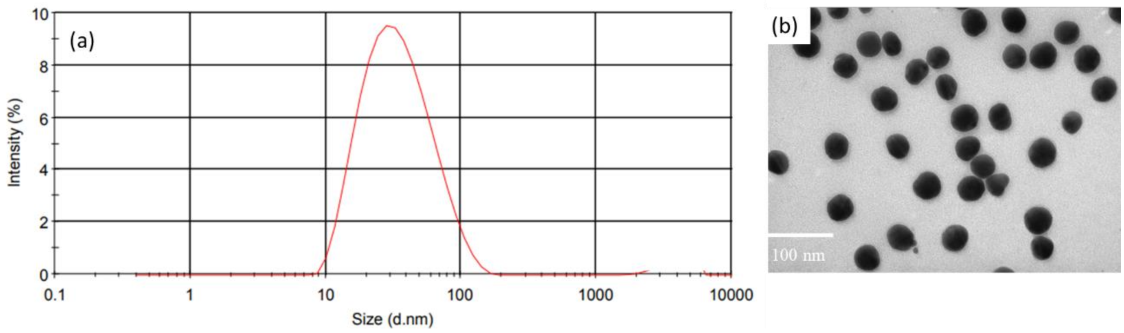

Figure 2. *Particle size distribution of S.S.AgNPs (a) and Image TEM of S.S.AgNPs (b)*

The energy spectrum of the particle sample was determined by EDX measurement with the characteristic energy of the Ag atom at 3 keV, accounting for 20.32% in mass (Figure 3). The presence of atoms such as Si, O, C, S and some metals is due to the samples that are spread over the glass and the substances that are fused into the sample analysis process.

The FTIR spectrum of pneumonia and silver nanoparticles is presented in Figures 4. The characteristic absorption peaks of *Salix* semi-solid extract show the functional groups in the compounds of the extract. A wide range with a peak at 3325 cm^{-1} can be seen as attributed to the symmetrical oscillation mode of the OH groups [27].

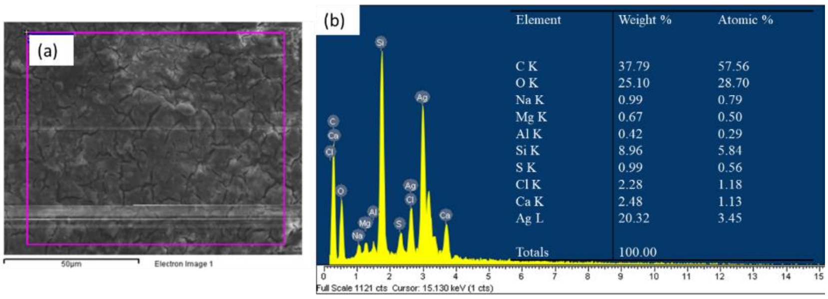

Figure 3. *SEM image of the zone defining atomic composition (a) and the EDX spectrum defining the atomic component and the mass, atomic ratio table of the particle sample (b)*

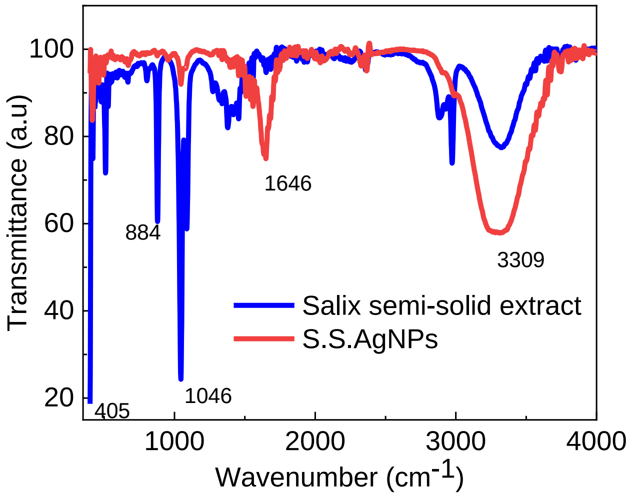

Figure 4. *The FTIR spectra of Salix semi-solid extract and S.S. AgNPs*

The smaller intensity peaks in the range from 2881 cm^{-1} to 2973 cm^{-1} are the contributions of the bonds = C-H and -C-H that are present in the aromatic phenolic and aliphatic alcohol groups from the hexose and tannin compounds [27]. C–O bonds from alcohols or aliphatic ethers can be indicated at the absorption peaks of 1045 cm^{-1} and 1086 cm^{-1} [32]. The absorption bands in the FTIR spectrum of the S.S.AgNPs are also in similar locations in the FTIR of the *Salix* semi-solid extract with slight displacement and there are variations in intensity. However, in the FTIR spectrum of the S.S.AgNPs appear absorption peaks in the 1650 cm^{-1} range that are attributed to corresponding to the bond of the carbonyl group with aromatic rings C=C [32].

Figure 5 is the results obtained when using the S.S.AgNPs to decompose the RB dye. The absorption spectra of the RB dye when there are no S.S. AgNPs but only the H_2O_2 deodorant decreases over time are shown in Figure 5a. The results showed that the absorption peak at 554 nm of RB decreased slightly over time from 1.05 to 0.91 in 2 hours. Meanwhile, the absorption of RB in the presence of S.S.AgNPs decreased sharply over the illumination time from 2.0 to 0.23 (Figure 5b). It can be seen that the absorbance of RB can only be reduced to a certain limit without complete decomposition. This is because in the solution there is competition between the catalysts and Ag ions, causing the decomposition of the pigment to be stopped [33]. The relative absorption of the RB dye decreases with the lighting time shown in Figure 5c. The difference in color degradation is clearly noticeable in the presence and absence of S.S.AgNPs. The decomposition performance is determined by the formula: H = $\frac{A_0 - A_t}{A_0} \times 100\%$. Where A_t is the absorption of RB at the time of t, and A_0 is the originally absorbed RB. Accordingly, color degradation performance with and without S.S.AgNPs was 88.5% and 13.3%, respectively. The mechanism of optical degradation of silver nanoparticles has been mentioned in many references [21]–[23]. Silver nanoparticles under the stimulation of white light are capable of releasing e and forming free hydroxyl groups. Color molecules react with these OH-free radicals and are broken down in the solution.

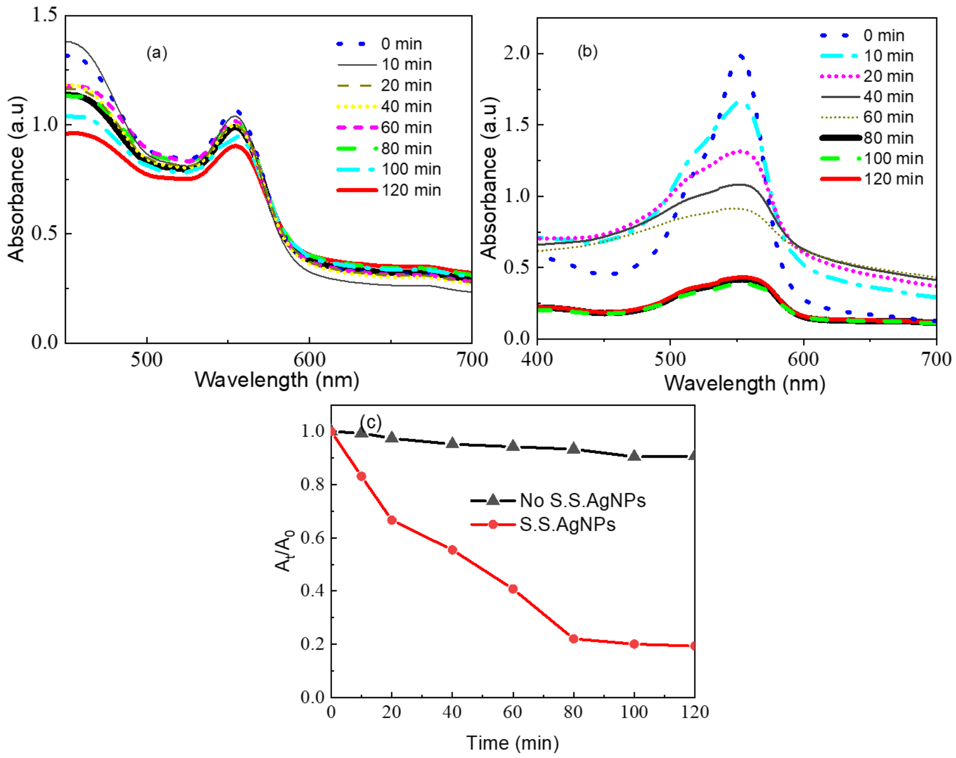

Figure 5. *The absorption spectra of RB over time when there are no S.S.AgNPs (a), when there are S. S. AgNPs (b) and the dependency of RB's relative absorption (A_t/A_0) in lighting time with S.S.AgNP (red line) and no AgNP (black line)*

4. Conclusion

The S.S.AgNPs are synthesized using *Salix* semi-solid extract with a spherical shape with an average size of about 30 nm. The synthesis performance is optimized when adjusting the high pH of the extract in a pH 7 neutral environment, $AgNO_3$ concentration of 1.2 mM, and the volume of the *Salix* semi-solid extract is 0.5 ml. The functional groups on the S.S.AgNPs on the FTIR spectrum represent the full functional group of extracts that are present in the characteristic compounds of the *Salix* semi-solid extract. The S.S.AgNPs were used to study the ability to decompose RB dye when using a 50W LED light source and a 6500K color temperature as a source of stimulation. The results showed that the photodegrading effectiveness of S.S.AgNPs was up to 88.5% much higher than when using H_2O_2 detergents alone.

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