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Valorization of rambutan (*Nephelium lappaceum* L.) peel extract for the green synthesis of silver nanoparticles

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ABSTRACT

Objective: To evaluate the use of rambutan peel extract as a reducing agent for the synthesis of Ag nanoparticles.
Design/methodology/approach: The extract of rambutan peels was prepared by adding 5 g of finely powdered dried peels to 100 mL of distilled water, and stirring the mixture for 15 min at 75 °C. The filtered extract was then used as a reducing agent for a 10 mM $AgNO_3$ solution. The effect of extract volume, reaction time and temperature were evaluated.
Results: Low amounts of extract and increased temperature promote a more effective and complete formation of AgNPs, and their band gap energy was estimated experimentally to be 2.75 eV.
Limitations on study/implications: Although UV-Vis spectroscopy confirmed the synthesis of AgNPs, additional characterization techniques should be employed in future studies.
Findings/conclusions: The use of rambutan peel extract is an effective biotechnological alternative for the green synthesis of AgNPs.

Keywords: Green synthesis, metal nanoparticles, photocatalyst, silver, sustainability.

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INTRODUCTION

Rambutan (*Nephelium lappaceum* L.) is a tropical fruit belonging to the Sapindaceae family. This fruit is native to Southeast Asia and its name derives from the Malay term “rambut,” which means “hair,” in reference to the long projections that cover its peel (Hernández-Hernández *et al.*, 2019) (Figure 1). Rambutan shares similarities with other tropical fruits such as lychee (*Litchi chinensis*) and its cultivation has expanded in tropical regions of Latin America, Africa and the Caribbean, where it has adapted favorably.

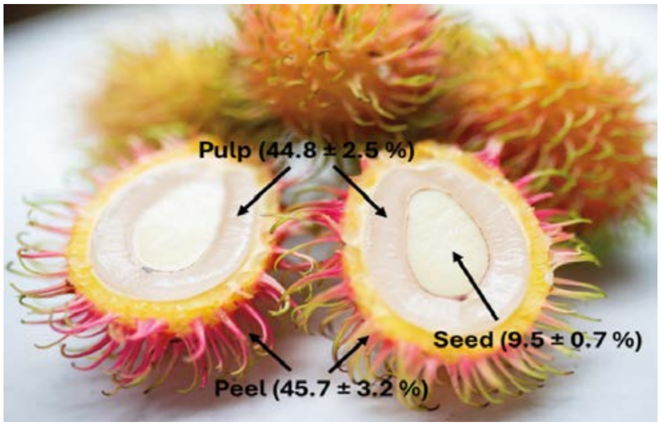

Figure 1. Rambutan fruit and percentage by weight of its constituents according to Solís-Fuentes *et al*., (2010) (image used under a Creative Commons license).

In Mexico, rambutan is cultivated in the states of Chiapas, Oaxaca, Tabasco, Guerrero, Colima, San Luis Potosí, Nayarit and Michoacán. Among them, Chiapas is the largest producer of rambutan in Mexico, where more than 90% of the crops are concentrated with an annual production of 12,200 tons (CONAGUA, 2023). In addition, the cultivation of this exotic fruit has been consolidated as an economic alternative for producers in various tropical regions, contributing to agricultural diversification and the local economy (Hernández-Hernández *et al*., 2019). However, few research has been done on the use of rambutan peel for biotechnological applications and use of its bioactive components, which could have applications in the food, pharmaceutical and cosmetic industries. Therefore, this work is focused on the valorization of rambutan peel extract for biogenic synthesis of silver nanoparticles (AgNPs), which have wide medical, biomedical and cosmetic applications, as well as an important use in the food, textile, and electronics industries, highlighting the environmental applications for water purification due to their excellent antibacterial and antiseptic properties.

In recent years, the green synthesis of AgNPs has emerged as an innovative and environmentally friendly alternative to conventional physical and chemical methods, which often involve the use of toxic substances and high energy consumption. This approach utilizes natural extracts from plants, microorganisms, or agro-industrial products as reducing and stabilizing agents, facilitating the conversion of silver ions (Ag) into metallic nanoparticles. Furthermore, the use of agro-industrial residues, such as rambutan peels, contributes to the circular economy by valorizing by-products that would otherwise be discarded. Therefore, the green synthesis of AgNPs using aqueous rambutan peel extract not only represents a biotechnological advancement, but also a viable and sustainable strategy to address contemporary technological and environmental challenges.

Therefore, the main objective of this work was to evaluate the use of rambutan peel extract as a reducing agent for the synthesis of AgNPs, the volume of aqueous peel extract, the reaction time, and temperature of synthesis were the main factors evaluated for the effective synthesis of AgNPs.

MATERIALS AND METHODS

Materials

Rambutan peel was selected as subject of study since it is one of the principal agro-industrial residues in the Southern of México. For this purpose, fresh rambutan fruits were acquired at the local market in the Soconusco Region, Chiapas, which were transported to the laboratory and immediately processed. On the other hand, $AgNO_3$ (purity $\geq$99.0%, Meyer) was used as source of Ag^+ ions and precursor of Ag nanoparticles, while distilled water (Meyer) was used to prepare the extract of rambutan peels as well as the appropriate solutions of $AgNO_3$. To adjust the pH, a solution 10% m/v of NaOH (purity $\geq$99.0%, Meyer) was used.

Preparation of the rambutan peel extract

Rambutan peels were separated manually and washed several times with tap water followed by two washes with distilled water at room temperature to remove dust and impurities. Washed peels were dried at 60 °C overnight and then ground to a fine powder (particle size $<$1mm). The extract of rambutan peels was prepared adding 5 g of dry powder to 100 mL of distilled water and heated at 75 °C for 15 min with vigorous stirring. The extract was cooled to room temperature, vacuum filtered to remove the solid particles (cellulose filter, 30 μm pore size) and stored at 4 °C until used (7 days as maximum).

Synthesis of Ag nanoparticles

The aqueous extract of rambutan peels was used as a reducing agent to produce Ag nanoparticles in colloidal suspension. For this purpose, several volumes of extract (1, 3, and 5 mL) were added dropwise to 50 mL of 10 mM $AgNO_3$ solution, then pH was adjusted to 11 and the resulting solution was stirred at 75 °C for different durations (1, 2, 3, and 4 hours). During the reaction, a visible color change was observed (from pale yellow to dark brown) indicating the formation of AgNPs. The synthetized AgNPs were stored in a sealed glass vial and protected from light at 4 °C.

Characterization

UV-Vis spectroscopy is considered a suitable technique to characterize metal nanoparticles, and this technique was used as the first step in the verification of the AgNPs since this analysis is fast, economical, and easily applicable. The UV-Vis spectra of the prepared AgNPs were recorded using a Genesys 10S spectrophotometer (Thermo Scientific) in the range of 200-800 nm. Before analysis, each solution of AgNPs was diluted for proper analysis as follows: 1 mL of prepared samples was transferred to 50 mL volumetric flask and diluted with distilled water.

RESULTS AND DISCUSSION

In this study, AgNPs were successfully synthesized by a green method using the aqueous extract of rambutan peels, which was confirmed by UV-Vis spectroscopy. The UV-Vis spectra of the prepared AgNPs are shown in Figure 2, where the maximum absorption is observed between 350 and 450 nm, corresponding to the typical absorption region of

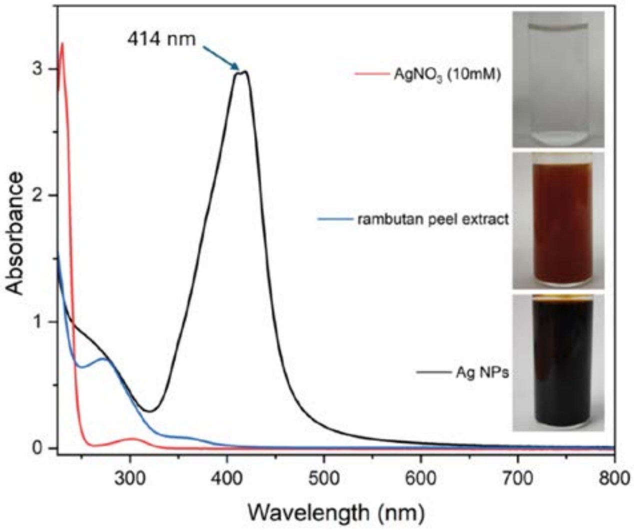

Figure 2. UV-Vis spectra of the synthesized AgNPs (black line), $AgNO_3$ 10mM (red line), and rambutan peel extract (blue line).

AgNPs (Lestari *et al*., 2018). Also, both UV-Vis spectra of the rambutan peel extract and the solution of $AgNO_3$ (10 mM) were included in the same figure for comparative purposes. Figure 2 shows that the maximum absorption of AgNPs is located at 414 nm, this peak in the visible and near-ultraviolet spectrum is associated with the surface plasmon resonance (SPR) on AgNPs, a common phenomenon in metallic nanoparticles where electrons in the surface layer are excited by photons, generating collective oscillations of free electrons that travel parallel to the surface and can give rise to reactive oxygen species (ROS). Additionally, according to the literature, the formation of AgNPs was indicated by the color change from straw yellow to dark brown, as result of the reduction and stabilization of Ag^+ ions (Dua *et al*., 2023; Kumar *et al*., 2015), which is depicted in the inset of Figure 2.

In previous studies, it has been reported that biomolecules/phytochemicals such as anthocyanins, ellagitannins, ellagic acid, corilagin, geraniin, syringic acid, *p*-coumaric acid, and phenolic compounds contained in the extract of rambutan peel could play a key role as reducing agent of Ag^+ ions, mainly by interaction with -OH and -CHO groups, which are electron donors and also responsible for the stabilization of the AgNPs by the interacting with the oxidized forms (-C=O, -COOH) (Kumar *et al*., 2015). Thus, the biomolecules present in the aqueous extract of rambutan peels are responsible for the reduction of Ag^+ ions to Ag^0 in a single step:

$$AgNO_3 \rightarrow Ag^+ + NO_3^- \tag{1}$$

$$Ag^+ + reducing\ extract\left(e^-\right) \rightarrow Ag^0 \tag{2}$$

The effect of the extract volume, reaction time and temperature for the synthesis of AgNPs were evaluated. Figure 3a shows the UV-Vis spectra of AgNPs prepared adding 1, 3, and 5 mL of rambutan peel extract, and it was observed that the increase of volume extract (5 mL) produce a broad and asymmetrical peak from 325 to 520, indicating the formation of Ag particles with a broad interval of size and non-uniform shape, which is consistent with those reported in other studies for AgNPs synthesis (Lestari *et al.*, 2018). Furthermore, a red shift is observed (blue line) from 414 to 424 nm, which is also associated with the increase of size of AgNPs. According to Mie theory the absorption bandwidth and peak position depends on the size of the AgNPs, as the diameter of the particles increases (agglomeration or aggregation), the polarization of them with light is not homogeneous, which is expressed in the shifts and broadening signals (Sharma *et al.*, 2016). On the contrary, prepared samples with 1 and 3 mL of extract exhibited the highest intensity on the absorbance peak at 414 nm, indicating a more complete transformation of the Ag^+ ions into AgNPs. Based on these results, 1 mL of rambutan peel extract was used as the appropriate volume for the synthesis of AgNPs. These observations underscore the critical role of the extract concentration for the green preparation of AgNPs, where low amounts of the reducing agent facilitate a more effective and complete formation of AgNPs.

In addition, the reaction time is a critical factor influencing the formation, stability, and morphology of AgNPs during green synthesis, as mentioned for (Ghaffari-Moghaddam *et al.*, 2014; Huq *et al.*, 2022). In this study, the synthesis of AgNPs using rambutan peel extract was monitored over various reaction times (1, 2, 3, and 4 hours) under constant temperature (75 °C) and stirring conditions. It is worth mentioning that the color change of the reaction mixture from pale yellow to dark brown occurred immediately after pH adjustment, indicating the reduction of Ag^+ ions and the formation of AgNPs. This visual observation was supported by UV-Vis spectroscopy, which showed the characteristic SPR peak around 414 nm (Figure 3b). The data suggest that nucleation, growth and stabilization of AgNPs occurred predominantly within the first two hours of reaction. No substantial increase in absorbance was observed after this time, indicating that Ag^+ ions reduction had reached saturation. Extended reaction times beyond this point (two hours) did not enhance nanoparticle yield but could potentially lead to agglomeration or changes in size distribution. Therefore, in this work, two hours was selected as the appropriate reaction time for the green synthesis of AgNPs.

On other hand, the effect of the temperature for the synthesis of AgNPs also was evaluated at two levels: at room temperature (24 °C) and 75 °C (to minimize water evaporation). Figure 3c shows the results of these experiments and it is observed that the increase of temperature promotes a complete and more effective formation of AgNPs, which is agree to those reported by (Dua *et al.*, 2023) who synthetized AgNPs using green extract, and indicates that temperature between 60-80 °C contributes to obtain nanoparticles with sizes <100 nm. In this work, the particle size of the prepared AgNPs was calculated from the UV-Vis spectra using Mie theory, which is a suitable approximation to describing how particles scatter light, depending on their size and optical properties, through the following equation:

$$2R = \frac{\lambda_{\max}^2 V_f}{\pi c \omega} \tag{3}$$

Where R is the radious of the particle, $\lambda_{\max}^{2}$ is the wavelength at maximum intensity of the SPR, V_f is the velocity of the electron at Fermi levels (1.4×10^6 ms^{-1} for Ag), c is the velocity of light, and is the FWHM of the SPR signal (full width at half maximum) (Pragatheeswaran *et al.*, 2010). The size of the AgNPs was determined in the range between 3.7 and 4.5 nm under the following conditions: 50 mL of 10mM $AgNO_3$ + 1 mL of rambutan peel extract, pH 11, and 2 hours of synthesis at 75 °C with magnetic stirring. Which agree with those reported in other studies where size of the synthetized AgNPs were experimentally measured by transmission electronic microscopy (TEM) ranging between 3.2 and 7.8 nm (Gharibshahi *et al.*, 2017).

Among the several applications of the AgNPs, the use for water and food disinfection is the most outstanding. Even, AgNPs has been evaluated as photocatalyst for water decontamination under sunlight (Dua *et al.*, 2023). For this reason, the optical band gap (Eg) of the prepared AgNPs was estimated experimentally from the UV-Vis spectra through the Tauc's equation:

$$(\alpha h\nu)^{n} = A\left(h\nu - E_{g}\right) \tag{4}$$

where α is the absorption coefficient, h is Planck constant, ν is frequency, A is proportionality constant, E_g is the optical band gap, and n is Tauc exponent (n=2 for indirect allowed transition). Practically, this method includes plotting $(\alpha h\nu)^{n}$ *vs.* $h\nu$, and extrapolating the

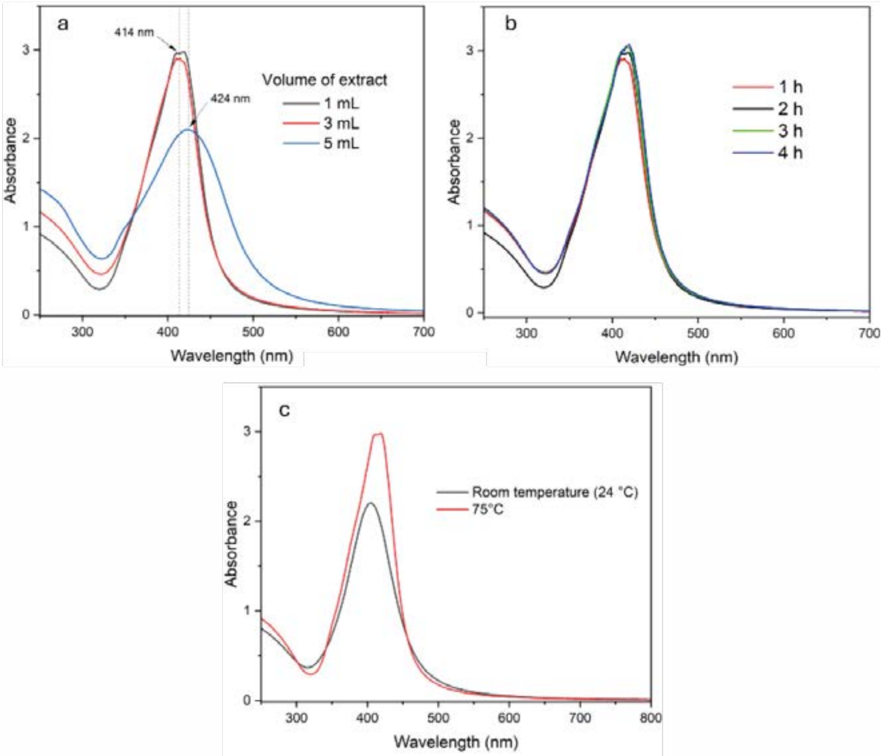

Figure 3. a) UV-Vis spectra of AgNPs prepared with different extract volumes, b) UV-Vis spectra of AgNPs prepared at different reaction times, c) effect of temperature for the synthesis of AgNPs.

linear range beyond the absorption edge yielding the value of the E_g on the abscissa axis, as shown in Figure 4.

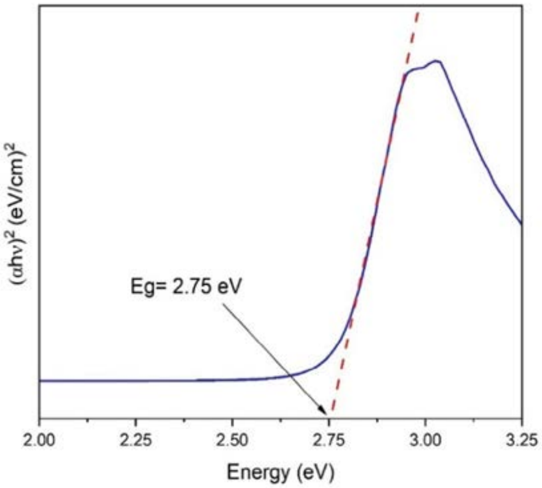

Figure 4. Estimated band gap of AgNPs using Tauc plot from UV-Vis spectra.

In this work, 2.75 eV (λ=450.8 nm) was experimentally estimated as the optical E_g of AgNPs, which is according to those reported by (Arumai Selvan *et al.*, 2018) who prepared AgNPs using several vegetable extracts, obtaining E_g values between 2.75 and 2.77 eV. In a few words, E_g can be explained as the minimum energy a photon must have to excite an electron to high energy bands, which can promote charge separation and transfer, ideal for photocatalytic systems. In the case of Ag nanoparticles, they primarily exhibit a metallic band structure, and their optical properties are related to plasmonic behavior and interband transitions, where the plasmonic absorption often dominates the optical spectra of AgNPs. However, in photocatalytic applications for water decontamination, it has been widely reported that electrons in the 4d states of AgNPs are excited to high-energy 5sp bands under UV-Visible radiation. Thus, the excited electrons can reduce the dissolved oxygen molecules to form superoxide radicals, while photoinduced holes left in the inner d bands can abstract the electrons of the polluting molecules (Varghese Alex *et al.*, 2020). Both superoxide radicals and photo-holes are the agents responsible for the degradation of the pollutants, giving rise to an interesting and promising alternative for environmental applications using AgNPs under a UV-Visible light source such as natural solar light.

CONCLUSIONS

The use of rambutan peel extract is an effective alternative for green synthesis of Ag nanoparticles, which contributes to the valorization of agro-industrial residues. The production of AgNPs was confirmed by UV-Vis spectroscopy as the first step, but further studies should employ other characterization techniques such as DRX, SEM-EDX, and FTIR spectroscopy. Based on the results, extract volume and temperature are crucial parameters for the preparation of AgNPs, highlighting that low amounts of the reducing agent and increased temperature facilitate more effective and complete AgNPs formation.

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