

An Ecoconceptual Synthesis Process of Silver Nanoparticles Integrating the Valorization of a Food Waste: The Peanut Shell

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How to cite this paper: Massard, C. and Awitor, K.O. (2022) An Ecoconceptual Synthesis Process of Silver Nanoparticles Integrating the Valorization of a Food Waste: The Peanut Shell. *Journal of Biomaterials and Nanobiotechnology*, 13, 45-59.

<https://doi.org/10.4236/jbnb.2022.132003>

Received: April 1, 2022

Accepted: April 27, 2022

Published: April 30, 2022

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Abstract

A green synthesis process of silver nanoparticles in an eco-conceptual approach to sustainable development is reported. This eco-friendly process is based on the valorization of a food waste, the peanut shell carrying natural chemical compounds able to reduce silver ions. The physicochemical characterizations performed to allow for validation of the success of this green chemistry approach: ultraviolet-visible and infrared spectroscopies validate the formation of silver nanoparticles. X-ray diffraction ensures the good crystallinity of these biogenic nanoparticles, while transmission electron microscopy allows highlighting of the morphology of these biosourced colloids. The results of the study of photocatalytic degradation of a model molecule by these biosynthesized nanoparticles demonstrate that they can be part of a completely sustainable process of depollution from its starting point, *i.e.*, the design of the nanoparticle, to the application of pollution remediation.

Keywords

Green Chemistry, Peanuts, Silver Nanoparticle, Sustainable Development

1. Introduction

Nowadays, we are witnessing an acceleration of research in the field of nanosciences which typically deals with the structure on the 1 - 100 nm scale [1] [2] [3]. In this general theme, the design of nanoparticles is an extremely developed research activity, in order to satisfy numerous applications [4] [5]. The field of use of nanoparticles seems unlimited, from nanomedicine [6] [7] to agriculture

[8] [9], clothing [10] [11] and cosmetics [12] [13]. Since its origins, nanoparticle elaboration protocols have evolved a lot within the classical top-down [14] [15] or bottom-up approaches [16] [17]. Currently, processes are moving towards more eco-friendly approaches [18] [19] that can be part of sustainable development [20] [21]. This eco-conceptual strategy is called green chemistry [22] [23]. This specialty chemistry integrates low-energy approaches [24] [25], the use of biosourced compounds [26] [27] and the limitation of organic solvents [28] [29]. In this study, we describe a process of biogenic synthesis of silver nanoparticles which is fully in line with the paradigm of green chemistry since it takes place in a single step, at room temperature and using a food waste, in this case, peanut shells, which are already used as a valuable waste in synthesis processes [30] [31].

2. Materials and Methods

2.1. Peanut Shells Extract Preparation

A 24 g sample of peanut shells is mixed with 600 mL of permuted water. The whole mixture is brought to 80°C under magnetic stirring for one hour. An infusion is then created in the form of a light brown liquid, which is cooled to room temperature. The liquid is then filtered, a brown powder is then extracted and dried; it will allow the reduction of silver ions and thus the formation of nanoparticles.

2.2. Biosynthesis of Silver Nanoparticles Using Peanut Shells Extract

Biosynthesis of silver nanoparticles using peanut shells extract was carried out at room temperature without using the following procedure. A 20 mM solution of silver nitrate is prepared. Under magnetic agitation, 10 mg of the previously prepared peanut extract is added to the silver nitrate solution. The solution, initially colorless sees its hue evolve in the course of time: from a light yellow which denotes the reduction of silver ions to a browner color at the end of synthesis.

2.3. UltraViolet-Visible Spectroscopy

Ultraviolet-visible characterizations were made using a Jenway 7310 single beam absorption spectrophotometer which can make spectral acquisitions at a wavelength of 300 - 700 nm. Quartz cells of 1 cm optical path are used. Permuted water is taken as a reference.

2.4. X-Ray Diffraction Spectroscopy

The crystalline structure of the silver nanoparticles is determined using a Bruker D2 Phaser X-ray diffraction diffractometer with $\text{CuK}\alpha$ radiation. The interplanar distance d was calculated by the Bragg equation and the crystallite size was estimated by the Debye-Scherrer formula.

2.5. Transmission Electron Microscopy

The imaging of nanoparticles was done by using the Hitachi transmission electron microscope (H7650). The accelerating voltage used was 80 kV and the images were taken by the AMT CCD camera positioned on the side. The sampling procedure consisted in drying 10 μL of colloidal suspension deposited on a 300-mesh carbon-coated copper grid.

2.6. Thermogravimetric Analysis

The thermogravimetric analysis was done with the TGA 4000 Perkin Elmer thermogravimetric analyzer. A biogenic silver nanopowder sample of 13.313 mg was weighed into a ceramic crucible. The sample was heated under nitrogen flow from 40°C to 950°C at a rate of 15°/min. The collected data are then processed by the Pyris software.

2.7. Fourier Transform Infrared Spectroscopy

The infrared analyses were done by the Perkin Elmer Spectrum one spectrometer used in transmission mode. The sampling was done using infrared grade potassium bromide pellets. One milligram of test powder was mixed and then ground with 200 mg of infrared grade potassium bromide. The pellet was molded by applying 10 t of pressure for 30 s. The spectra were recorded in a range between 400 and 4000 cm^{-1} of wave number.

2.8. Photocatalytic Activity Test

The photocatalytic efficiency of silver nanoparticles synthesized by green chemistry was evaluated by implementing the study of the photodegradation of an organic dye, acid orange 7. A solution of this model molecule was prepared by dissolving the appropriate amount of powdered acid orange 7 to obtain a concentration of 100 ppm. 10 mg of biogenic silver nanoparticles are added with stirring to a quartz reactor containing the dye solution. The monitoring of the reaction consists in irradiating the reaction medium during defined times and at the end to record the decrease of the maximum absorbance according to time by using the visible spectrophotometry. Thus, the kinetics of photodegradation is obtained by the evolution of the characteristic peak of the AO7 located at a wavelength of 486 nm. The wavelength of the ultraviolet light is 284 nm in a configuration delivering about 1 mW/cm^2 on the surface of the quartz reactor.

3. Result and Discussions

3.1. Ultraviolet-Visible Analysis

It has been reported in several studies that peanut shells, shown in **Figure 1** have bioactive chemical functions that can be used for green synthesis [31] [32]. In this work, the use of this biobased reagent is intended to reduce silver ions in solution and at room temperature.

In the first minutes of the synthesis, the addition of the powder extracted from the peanut shell infusion does not induce any color change, the solution remains colorless. Progressively, the mixture, stirred at room temperature sees its color evolve from colorless to pale yellow then dark yellow. **Figure 2** shows this evolution.

This development reflects the reduction of silver ions in solution and the manufacture of silver nanoparticles [33] [34]. This color evolution is caused by the resonance of the surface plasmon associated with the formation of silver colloids [35] [36] [37]. In order to confirm this colored indication, samples are taken from the reaction medium and analyzed over time, the spectra are presented in **Figure 3**.

A maximum absorbance peak appears at 440 nm and its intensity increases with time, confirming the progressive formation of silver nanoparticles in solution at room temperature [38] [39]. **Figure 4** shows the evolution of the absorbance maximum at 440 nm versus time. We observed that the evolution is linear during the first 5 hours of synthesis, then it is recorded as a stall which can be attributed to the end of the reaction.



Figure 1. A photograph of a peanut shell used in this study.

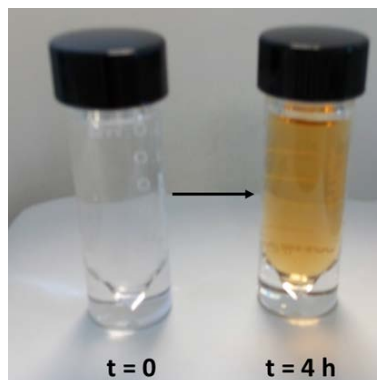


Figure 2. Evolution of the color of the reaction medium as a function of the reaction time.

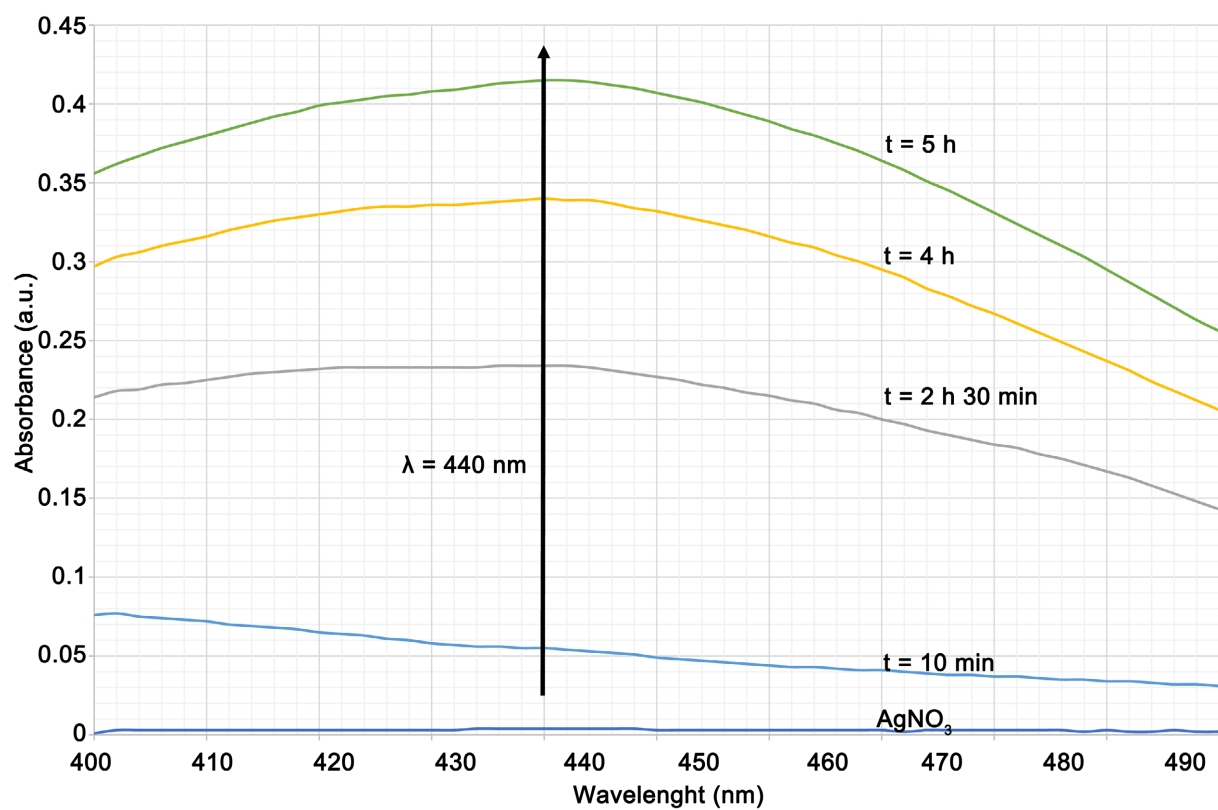


Figure 3. UV-vis spectra of silver colloid evolution of the plasmonic band as a function of time.

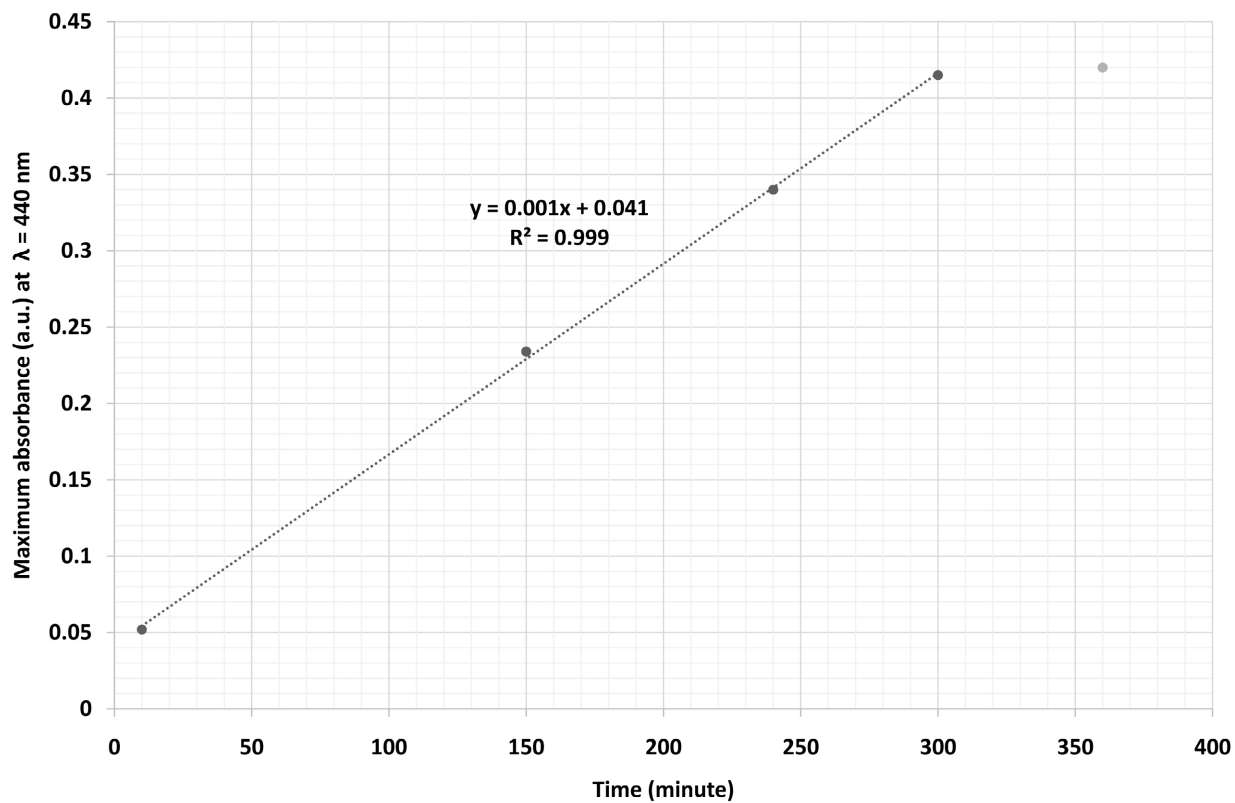


Figure 4. Evolution of the absorbance maximum at 440 nm as a function of time.

3.2. X-Ray Diffraction Analysis

Figure 5 shows the X-ray diffraction pattern of the synthesized silver nanoparticles. The characteristic peaks of silver appear at $2\theta = 37.810^\circ$, 44.274° , 64.253° , 77.09° and 81.763° respectively. Using the data published in the literature [40] [41], in particular the Joint Committee on Powder Diffraction Standards (JCPDS) data card N°04-0783, they can be attributed respectively to the (111), (200), (220), (311) and (222) crystallographic planes of the face centered cubic (fcc) structure of metallic silver [42] [43]. The others (400), (311) and (420) are also attributed to metallic silver but barely emerge from the background. It also found some additional peaks at 45.955° , 54.512° and 56.429° attributed to some unknown residual impurities.

The interplanar distance d is calculated from the Bragg equation:

$$\lambda = 2d \sin \theta \quad (1)$$

Based on the (200) crystal plane of Ag nanoparticle, the interplanar distance was estimated at 2.044 \AA , which is in good agreement with other studies [44].

From the pattern, the lattice parameter is calculated: $a = b = c = 4.1 \text{ \AA}$. The relative error with respect to the JCPDS sheet N°04-0783 is 0.78% which proves a good correspondence between the experimental value and the reference data and attests to the good crystallinity of the produced nanoparticles

An estimation of the crystallite size was calculated using the Debye-Scherrer equation.

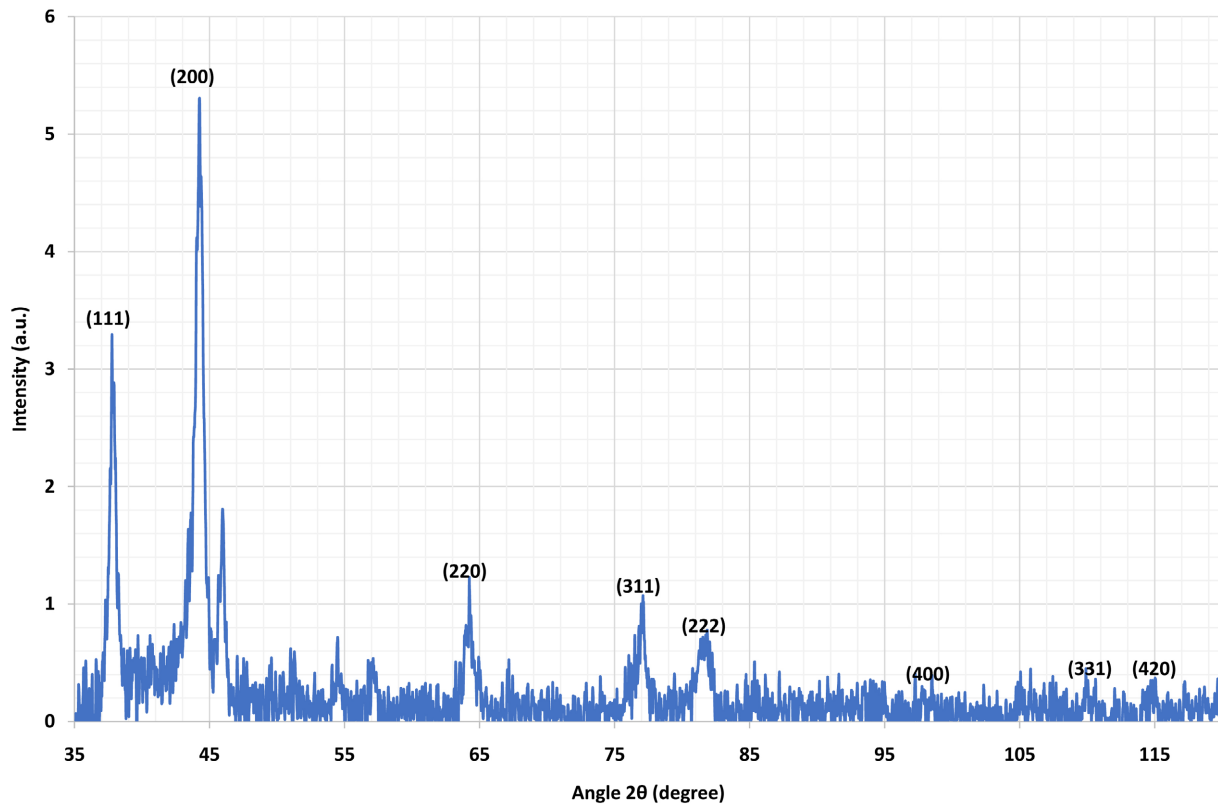


Figure 5. XRD pattern of the synthesized silver nanoparticles.

$$D = \frac{0.94\lambda}{\beta \cos \theta} \quad (2)$$

where D is the particle size in angstrom, λ is the wavelength of CuK α 1 line (1.54056 Å for radiation), β is the full-width at half max (FWHM) of a peak and θ is the Bragg diffraction angle. By calculation, we found that the size of the crystallites is 14.2 nm.

3.3. Transmission Electron Microscopy Analysis

Figure 6 shows the Transmission Electron Microscopy (TEM) image of the biogenic silver nanoparticles. The nanoparticles appear as aggregates of irregular geometry which may be a consequence of the sampling process of drying drops of the nanosuspension on the copper grids. These aggregates have elongated morphologies of length around 300 nm.

3.4. Thermogravimetric Analysis

The thermal behavior of silver nanoparticles synthesized using peanut extract is analyzed by thermogravimetry (TGA) under nitrogen flow. The weight loss (%) as a function of temperature is shown in **Figure 7**. In a classical way, the thermal decomposition starts initially with the elimination of the adsorbed water and of volatile organic compounds at about 150 °C [45]. The main mass loss can be attributed to the thermal decomposition of the organic compounds from the peanut extract, which is adsorbed on the surface of the silver nanoparticles and which had allowed the initial reduction of the silver ions. At the end of the heat treatment, the residual mass can be considered as metallic silver [46]. From this analysis, it is shown that the sample consists of 96% of metallic silver, the rest being attributed to the organic functionalization of the surface of the nanoparticles made by the extract of peanut.

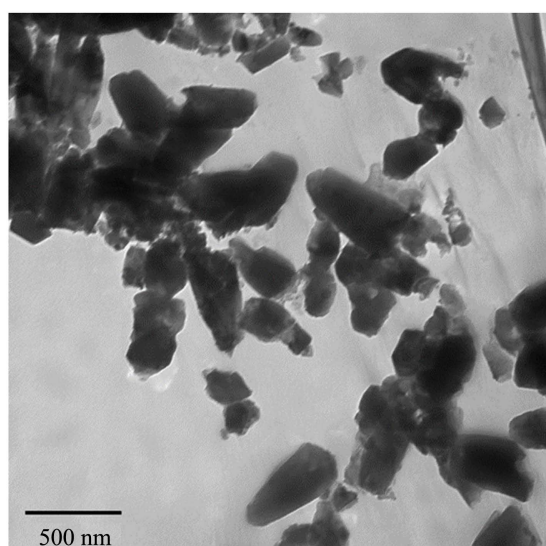


Figure 6. TEM micrograph of the synthesized silver nanoparticles.

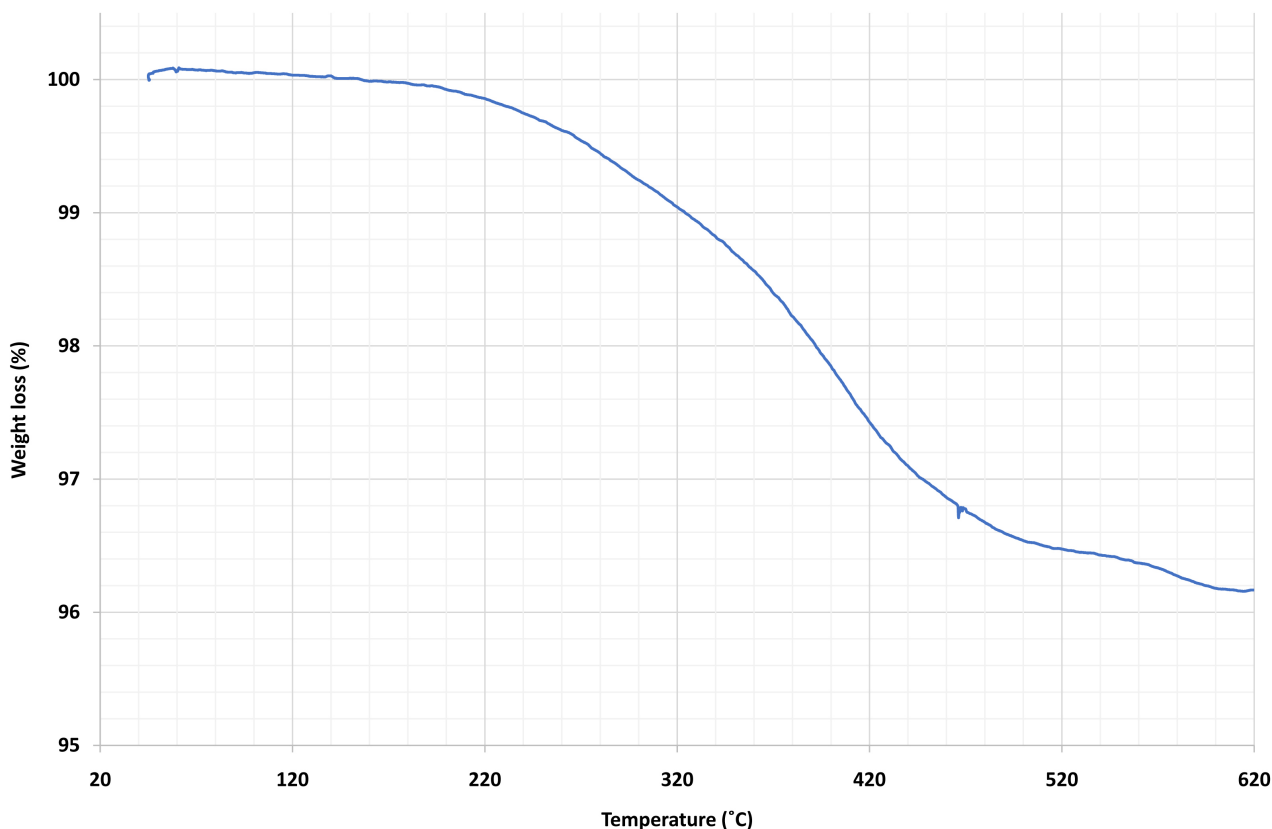


Figure 7. Thermogravimetric analysis of silver nanoparticles recorded at a scan rate of 15 degree per minute.

3.5. Fourier Transform Infrared Analysis

Fourier Transform Infrared spectra of the peanut extract and the biosynthesized silver nanoparticles are presented in **Figure 8** in split view. The peak at 3430 cm^{-1} is attributed to the hydroxyl groups (-OH) [47] which can be present in the alcoholic, phenolic and carboxylic compounds are coming from the peanut extract. The peaks near 2920 cm^{-1} could be the signature of (C-H) asymmetrical stretching bands of olefins, aromatics or aliphatic organic compounds [48]. The major band at 1636 cm^{-1} may correspond to adsorbed water molecules [47]. The peak at 1384 cm^{-1} evidenced the presence of NO_3^- [49] while the band recorded around 1094 cm^{-1} is assigned to C-O-C groups [50] from the proteins of the peanut extract. In general, the direct comparison of the two spectra shows that the same bands are present in the two compounds, which means that there is no chemical bonding between the metallic core and the biosourced reductant during the synthesis. The intensity fluctuations as well as the small shifts in wave number recorded are caused by the relative proportions between the reducing agent and the silver nanoparticles.

3.6. Evaluation of Photocatalytic Capabilities of the Biosourced Silver Nanoparticles Produced

The photocatalytic activity of these biogenic silver nanoparticles is evaluated by a photodegradation test of an organic dye named Acid Orange 7 (AO7), a model

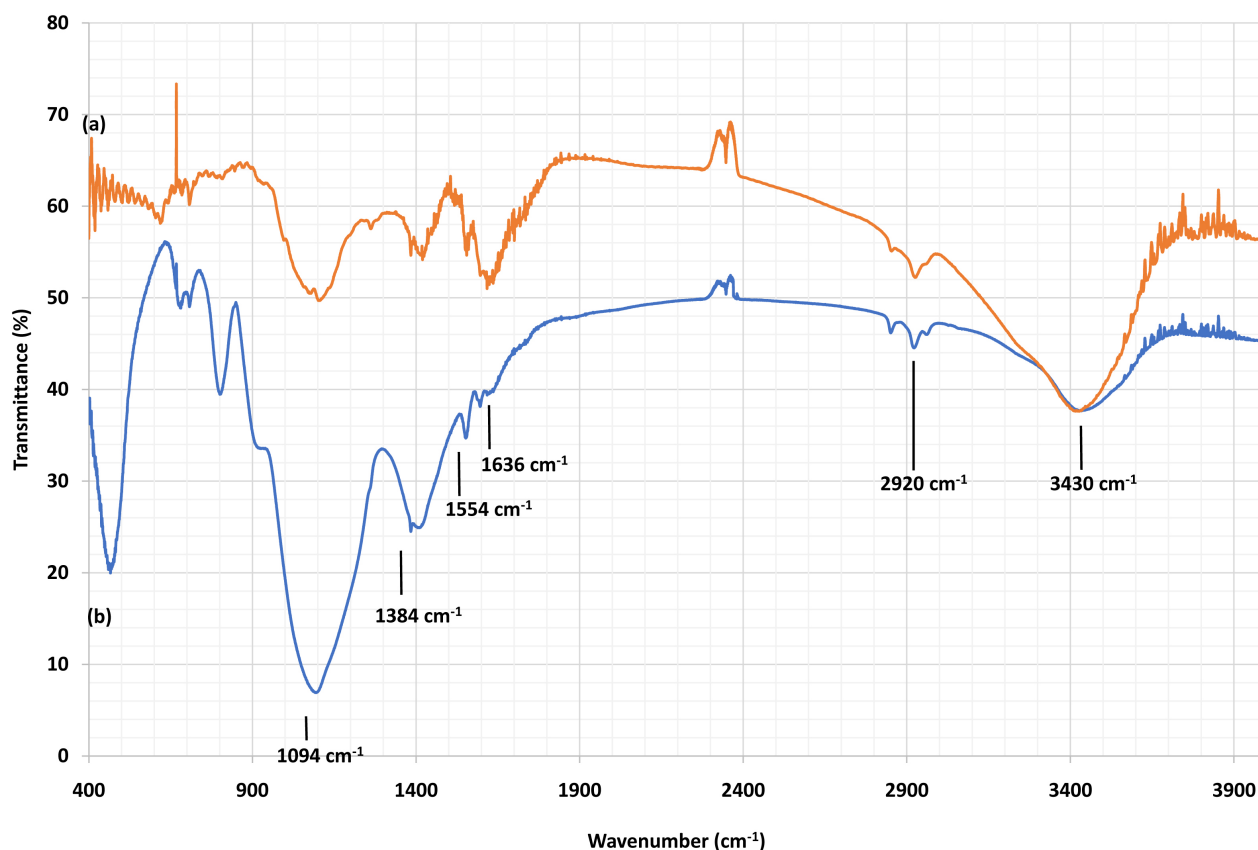


Figure 8. FTIR spectra was shown at wavenumber of 400 - 3900 cm^{-1} in presence of: (a) peanut extract (b) biosynthesized silver nanoparticles.

azo dye [51] under ultraviolet irradiation at 254 nm. In UV-visible spectroscopy, the AO7 molecule is characterized by a maximum absorbance peak positioned at the wavelength of $\lambda = 486 \text{ nm}$ [52]. **Figure 9** summarizes the gradual photocatalytic degradation of the dye AO7 by the decrease of the absorbance peak at $\lambda = 486 \text{ nm}$ during 8 hours of monitoring, while the control remains unchanged, see **Figure 9**.

The exploitation of the series of ultraviolet spectra allows to calculate the percentage of degradation of the AO7 dye versus time of irradiation using the formula [53]:

$$\text{AO7 degradation (\%)} = \frac{C_0 - C_t}{C_0} \times 100 \quad (3)$$

C_0 is the initial concentration of the AO7 dye solution and C_t is the concentration of the AO7 dye solution after t minutes of UV irradiation respectively. Using Beer Lambert's law, all dye concentrations are directly proportional to the absorbance of the peak at 486 nm. All the results are reported in **Figure 10**.

The photodegradation process is initiated by the interaction of ultraviolet photons and silver nanoparticles. This results in an electronic transfer phenomenon towards the conduction band [54]. The surface electrons can then react with the dissolved dioxygen to form hydroxyl radicals. These radicals then oxidize

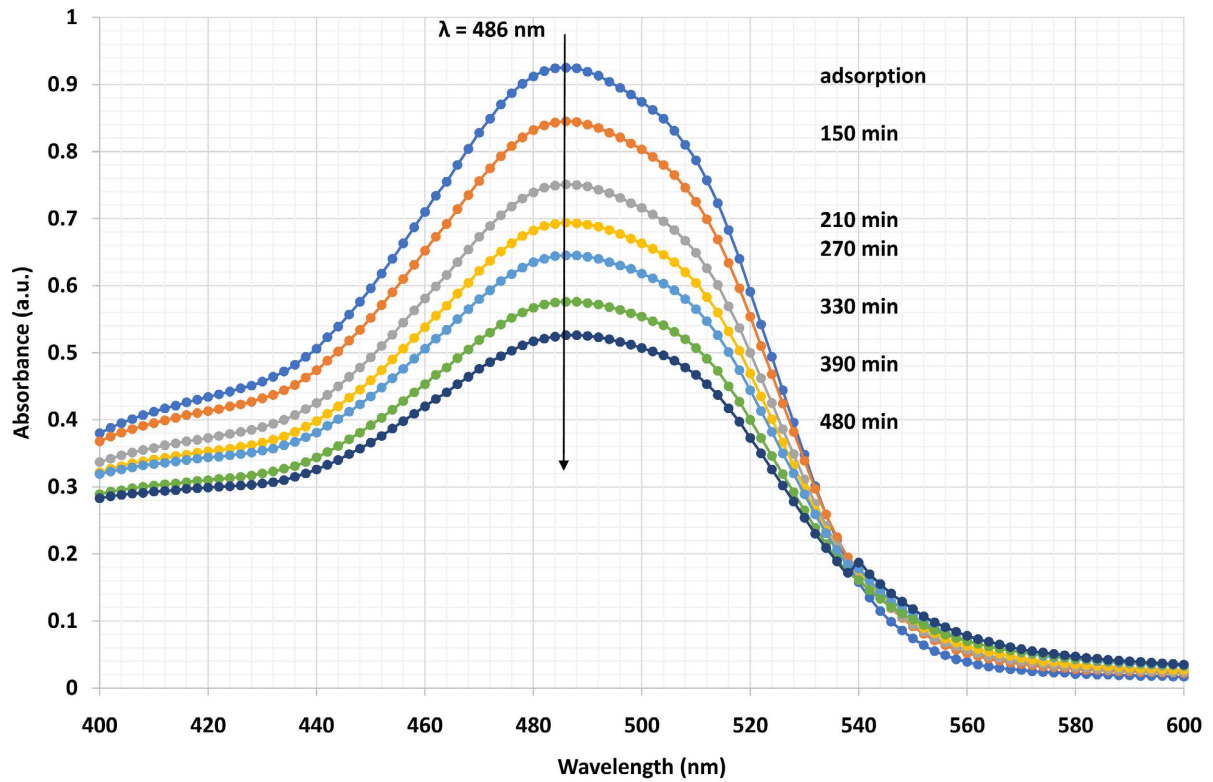


Figure 9. FTIR spectra was shown at wavenumber of 400 - 3900 cm^{-1} in presence of: (a) peanut extract (b) biosynthesized silver nanoparticles.

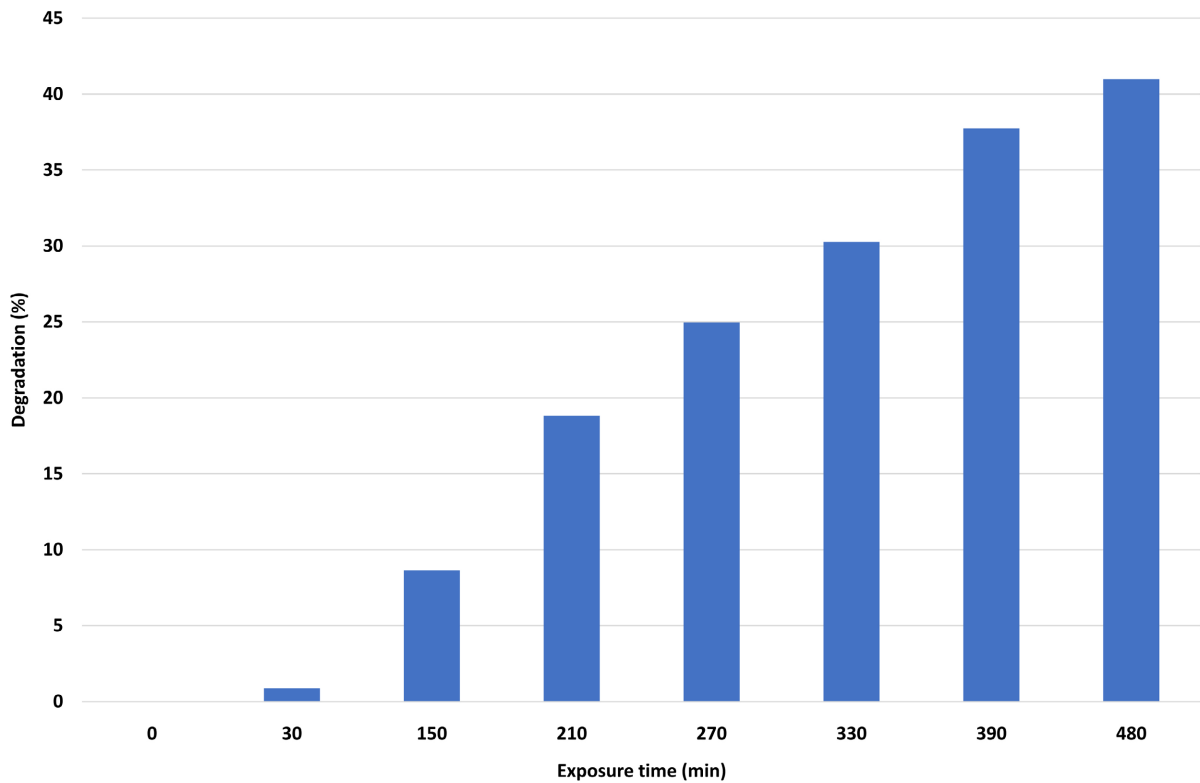


Figure 10. Degradation % of AO7 dye with UV irradiation time.

the AO7 molecules adsorbed on the surface of the silver nanoparticles, which leads to the formation of by-products [55]. This observation allows to highlight the efficiency of our biosourced silver nanoparticles synthesized in a photodegradation process applicable to depollution methods.

4. Conclusion

This research work has demonstrated the feasibility of an eco-conceptual, low-energy silver nanoparticle synthesis process from a sustainable development perspective. Moreover, this study demonstrates the possibility of the valorization of a food waste: the peanut shell. Compared to conventional synthesis processes that require both reflux heating and synthesis reagents, the process we have described here is free of these limitations, which is a step forward in the implementation of environmentally responsible processes. All the physicochemical characterizations carried out have validated this green chemistry process and opened the way to its integration in many application fields, in particular pollution control, illustrated in this paper by the results of the photodegradation study of an organic dye.

Acknowledgements

We thank Christelle Blavignac from the CICS (Centre d'Imagerie Cellulaire Santé) Clermont Auvergne University for the transmission electronic microscopy (TEM) pictures of the silver nanoparticles.

Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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