

SYNTHESIS OF SILVER NANOPARTICLES USING HYDROALCOHOLIC EXTRACT OF SAMBUCUS NIGRA AND LAVANDULA ANGUSTIFOLIA

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Abstract: Technique development for getting environmentally friendly nanomaterials was the paper's main goal. Spectroscopic techniques were utilized to characterize the plant extracts used in this work (UV-Vis, FTIR and Raman). In this case, plants from the Valea Voievozilor was processed in a dry state, was used at the ideal moment and at the plant's peak maturation period. Several analytical techniques (UV-Vis, FTIR, Raman analysis) have been used to analyze the nanoparticles (Ag) created with the aid of these plant extracts. Both the bioreduction of silver ions (Ag^+ to Ag^0) and the stability of silver nanoparticles are caused by the bioactive chemicals found in plant materials.

Keywords: nanomaterials, silver nanoparticles, *Sambucus nigra*, *Lavandula angustifolia*

1. INTRODUCTION

Sambucus nigra is a 4-5 m tall shrub or it can reach 8-10 m, in the form of a bush or even a tree, with white flowers, arranged in inflorescences that form black fruits. It occurs in spontaneous flora or is cultivated in hill and plain regions, both flowers and fruits are consumed, usually in the form of preparations [1].

Sambucus nigra fruits contain large amounts of vitamins such as vitamin A, the range of vitamin B and vitamin C, flavonoids, bioflavonoids, amino acids, carotenoids, but also sugars and tannins. [1]. It is rich in anthocyanins found in the juice and extracts of *Sambucus nigra* [2]. The highest concentration of flavonoids and phenolic acids is found in mature plants. They also have a high content of minerals such as calcium, iron (approximately 9%), phosphorus and potassium (approximately 8%), fiber. [3]. The characteristic flavor of *Sambucus nigra* fruits is the result of a combination of alcohols, essential oils and esters [4].

Sambucus nigra fruits (Fig. 1.) are consumed for their therapeutic properties such as antioxidant, antipyretic, antiseptic (acts against bacteria and viruses), analgesic, adjuvant in calming coughs and facilitating expectoration, reduces inflammation, has a tonic effect, detoxifying (laxative and diuretic role), anti-inflammatory, immunomodulatory and antiallergic. [5-7].

Lavandula angustifolia is a perennial, sempervirescent plant, is a mountain species, a semi-shrub that is a little pretentious. Being an

entomophilous allogamous plant, through generative propagation (through seeds) it produces numerous forms, genotypes with morphological, phenotypic, phenological, genetic, chemical variability and, of course, with differentiated economic efficiency. [8]. The strongly scented of *Lavandula angustifolia* flowers, as in other species of the Lamiaceae family, are grouped in spiciform inflorescences (Fig. 1.), composed of a 10-20 cm long floral stem and a 3-6 cm long floral spike contains polyphenols, flavonoids, tannins, saponins, phytosterols, phenolic acids, triterpenes and cardiac glycosides, but the main organic compound is the essential oil. The content of macronutrients as well as their concentration is different depending on the variety [8].

Lavandula angustifolia flowers are used for their antiseptic (acts against bacteria), repellent (for insects), deodorant, relaxing properties. [9].

This paper presents the preparation of nanomaterials using *Sambucus nigra* fruit and *Lavandula angustifolia* flower extracts, as well as their characterization by UV-Vis, FTIR and Raman spectroscopic techniques. The phytochemical profile of these nanomaterials (the total content of flavonoids and polyphenols respectively, but also the antioxidant activity) was also carried out in an attempt to establish further practical applications.

2. EXPERIMENTAL PART

Materials

Silver nitrate, DPPH, ethanol purchased from Sigma-Aldrich (Germany) and distilled water obtained from

the facility provided by Thermo Fisher Scientific (USA) in our laboratories were used to obtain the nanomaterials.

Sambucus nigra fruits and *Lavandula angustifolia* flowers were collected from our own culture in the central area of Dambovită county.

The plant material subjected to this study was dried for a period of 24 hours at a constant temperature of 30°C using a Venticell oven. From this dried plant material, 2 g was weighed and a mixture was prepared together with ethanol and distilled water in a 25 mL Berzelius beaker. This mixture was brought to boiling point and maintained for 10 minutes. An extract was thus obtained which was filtered and allowed to cool to room temperature (~25°C).

A 10⁻³ M silver nitrate solution was prepared and mixed with the plant extracts in a ratio of 1:1 and allowed to stand until experimental investigations were carried out.

For the determination of the phytochemical profile, solutions obtained by mixing one milliliter of plant material extract to which 4 mL of distilled water and 0.3 mL of sodium nitrite (5%) were added were used. This mixture was left under static conditions for 5

minutes at room temperature, after which 0.3 mL of aluminum chloride (10%) and 2 mL of sodium hydroxide (1M) were added. The obtained mixture was again left in static conditions at room temperature for 5 minutes, after which 2.5 mL of distilled water was added.

Equipments

With the UV-Vis Spectrometer Evolution 260 Bio, absorption spectra were obtained in the range of wavelengths 190-1100 nm for the previously obtained solutions.

The investigations to determine the functional groups were carried out with the Vertex 80 Fourier transform infrared spectrometer using the ATR (attenuated total reflection) accessory. By operating the OPUS 7.5 software, the working parameters were set (transmittance, resolution 4 cm⁻¹, 32 scans per sample in the range 400-8000 cm⁻¹). For each sample, the background spectrum in the air was obtained before the measurement. After each sample the ATR fixture was cleaned using isopropyl solution (70%) and paper towel.



Sambucus nigra



Lavandula angustifolia

Figure1. The photos of the plants

Also used was the portable Raman spectrometer (Xantus-2 from Rigaku) which can be used for two wavelengths (785 and 1064 nm) and is controlled by means of the Micro 2020 software. The analysis was performed at 1064 nm, for 3000 ms exposure averaged from three scans per sample and background performed per sample.

Determination of the phytochemical profile

To determine the total content of flavonoids, a calibration curve was obtained by preparing solutions of different concentrations using catechin as a standard. The analysis was performed on a solution consisting of a mixture of sample, distilled water, sodium nitrite, aluminum chloride and sodium hydroxide. The solution was homogenized and analyzed at 510 nm by comparison with a blank solution consisting of distilled water.

To determine the total polyphenol content was determined using Folin-Ciocalteu reagent colorimetric technique. After the calibration curve was plotted, the solution consisting of a sample mixture, Folin-Ciocalteu solution (5 mL) and sodium carbonate (4 mL) was prepared, which was homogenized and then kept under static conditions for a period of one hour at room temperature. The solution was analyzed at 765 nm and compared with the blank sample consisting of distilled water.

The DPPH method was selected for the determination of the antioxidant activity. Thus, a mixture consisting of plant extract to which DPPH was added was subjected to analysis, which was kept under continuous stirring for half an hour and then at rest in the dark and at room temperature. The control sample was prepared under similar conditions replacing the plant material extract with distilled water. The analysis was carried out at 517 nm using the UV-Vis method, and the result was calculated with the formula:

$AA \% = (A_{\text{blank}} - A_{\text{extract}} / A_{\text{blank}}) * 100$
 where: $A_{\text{blank}} = 0.5 \text{ mL distilled water} + 1 \text{ mL DPPH}$;
 $A_{\text{extract}} = 0.5 \text{ mL sample} + \text{DPPH}$

3. RESULTS AND DISCUSSION

The UV-Vis spectra of the nanomaterials formed from plant extract and silver nitrate solution are shown in Figure 2. For the two samples analyzed, an absorption maximum around 480 nm was recorded, which indicates that ionic reduction took place of silver in the analyzed solutions.

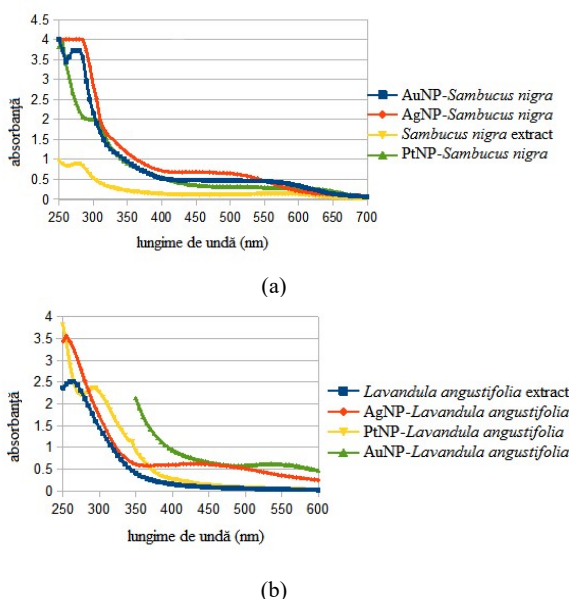


Figure 2. UV-VIS spectra for Ag nanoparticles in *Sambucus nigra* (a) and *Lavandula angustifolia* (b)

The FT-IR spectra of an extract silver nanoparticles aqueous solution indicate the existence of transmittance peaks at 3300, 1640, 1410, 1020 cm^{-1} (*Sambucus nigra*) and 3300, 1640, 1020 cm^{-1} (*Lavandula angustifolia*). The assignment of the spectral maxima is recorded in Table 1.

Table 1. Assignment of FTIR spectral maxima

The value of the spectral maximum (cm^{-1})	<i>Sambucus nigra</i>	<i>Lavandula angustifolia</i>	Assignment of spectral maxima [10-15]
3300	x	x	O-H stretching
1640	x	x	C=C bonds
1410	x	-	C-H bond
1020	x	x	OH bonds

Since the spectra recorded for the two samples of nanomaterials are similar, Figure 3 shows the spectrum obtained for the nanomaterial with *Sambucus nigra* extract.

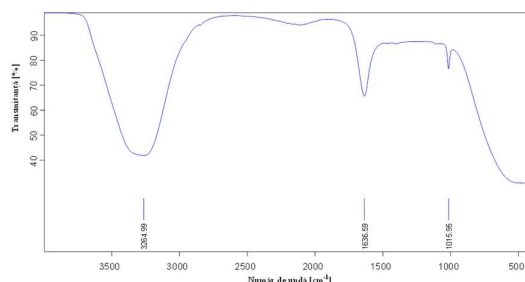


Figure 3. The FTIR spectra of Ag nanoparticles in *Sambucus nigra*

The spectra of the nanomaterials analyzed by Raman spectroscopy are presented in Figure 4 and the results are recorded in table 2.

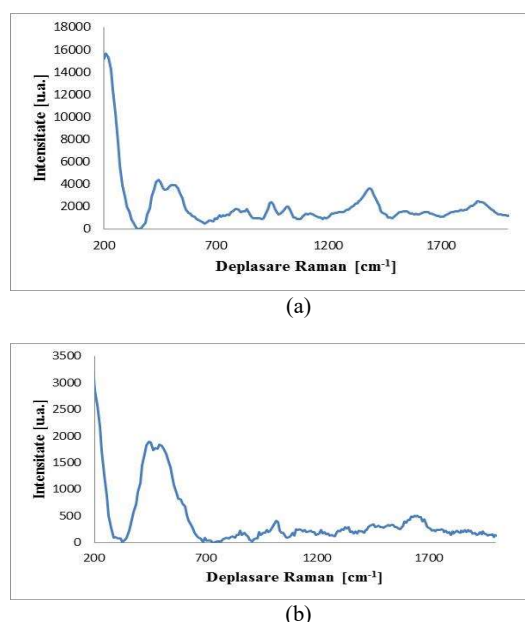


Figure 4. The Raman spectra of *Sambucus nigra* (a) and *Lavandula angustifolia* (b) extracts

Table 2. Assignment of Raman spectral maxima

The value of the spectral maximum for <i>Sambucus nigra</i> (cm^{-1})	The value of the spectral maximum for <i>Lavandula angustifolia</i> (cm^{-1})	Assignment of spectral maxima [10-15]
442	444	C-O, C-OH bonds
510	492	C-O bonds
945	-	C-H bonds
1020	1014	C-C bonds
1381	-	C-C bonds
-	1644	C=C bonds
1870	-	carbon chain

It is generally known that most phenolic phytochemicals, including flavonoids and tannins, contain hydroxyl functionality as a key component. The plant material analyzed in the present study was selected following

recent research that analyzed antioxidant properties. [10-16].

The results of the phytochemical profile analysis indicated the following values presented in table 3:

Table 3. The results of the phytochemical profile

Extract	Total flavonoid content (mg C.E./L)	Total polyphenol content (mg GAE/L)	Antioxidant activity (%)
<i>Sambucus nigra</i>	252.20	2702.54	91.30
<i>Lavandula angustifolia</i>	77.13	1118.64	82.39

By rigorously analyzing the most recent research in this field, corroborated with the results obtained and presented above, we can conclude that the high values recorded for the phytochemical parameters favor the formation process of silver nanoparticles and thus the obtaining of nanomaterials using the plant material selected for this study.

4. CONCLUSIONS

In this study, hydroalcoholic extracts from the 2 plants were prepared and analyzed to determine the phytochemical parameters (T.F.C., T.P.C. and AA). AgNO₃ ionic solution (10⁻³ M) was added and they were analyzed by UV-Vis spectroscopy where they showed an absorption band at 480 nm for silver nanoparticles. The obtaining of silver nanoparticles was confirmed by UV-Vis spectrometry.

With the support of FTIR and Raman spectroscopy we were able to confirm the presence of phytochemical compounds in the analyzed samples and as mentioned above we can conclude that they contributed to facilitating the formation of silver nanoparticles.

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