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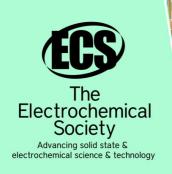
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Chromatographic analysis of phytochemicals components present in mangifera indica leaves for the synthesis of silver nanoparticles by AgNO₃ reduction

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Abstract. It was studied the green synthesis of silver nanoparticles (AgNPs) from the reduction of a silver nitrate solution (1 and 10mM) in the presence of an extract of mangifera indica leaves. Phytochemicals components present in extracts of mango leaves were determined using a GC-MS chromatograph. The results showed the presence of the phenolic compound pyrogallol (26.9% wt/5mL of extract) and oleic acid (29.1% wt/5mL of extract), which are useful for the reduction of the metallic salt AgNO₃ and the stabilization of silver nanoparticles. The synthesized nanoparticles were characterized by UV visible spectroscopy (UV-vis), evidencing absorbances at wavelengths of 417nm (AgNPs-1) and 414nm (AgNPs-10), which are characteristic peaks of this metallic nanoparticles. Scanning Electron Microscopy (SEM) was used to determine the size of the synthesized nanoparticles. A particle size of about 28±7nm was observed for the AgNPs-1 sample and 26±5nm for the AgNPs-10. This suggests the advantages of green chemistry to obtain silver nanoparticles with a narrow size distribution.

1. Introduction

Synthesis of silver nanoparticles represents a matter of great interest within the scientific community nowadays thanks to their chemical, optical and morphological properties. Their large volumetric surface area allows the liberation of silver ions, which inhibit bacterial growth, adding microbial resistance to the material [1,2]. These nanoparticles can be synthesized by green chemistry methodologies using plant extracts with phytochemical components that promote the reduction of the silver nitrate metallic salt [3]. One of the advantages of this method is that it avoids the use of organic solvents such as hydrazine, which can produce volatile toxic residues like toluene and chloroform [4].

The purpose of this research was the synthesis of silver nanoparticles by using mango (mangifera indica) leaves extracts as reducing agent for the silver nitrate salt (AgNO₃). For that reason, a chromatographic analysis of the extract was performed, aiming to identify the phytochemical components that are present in the biomass. Such substances could promote both reduction of the metallic salt and stability of the suspended nanoparticles. A gas chromatograph coupled to a mass spectrometer was used for this analysis. Synthesis of the silver nanoparticles was carried out at room temperature, varying the concentration of silver nitrate from 1.0 to 10mM. The synthesized nanoparticles were characterized by means of both UV-visible spectroscopy and Scanning Electron Microscopy (SEM) to determine the size distribution of the nanoparticles as a function of the concentration of the precursor reagent.

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2. Materials and methods

Mango (mangifera indica) fruit trees were selected from the rural population of Arroyo Grande (Bolívar, Colombia) for the gathering of leaves. This biomaterial was used for the preparation of the aqueous extract and the characterization of phytochemical components. Infusion technique was used when preparing the extract. In order to do that, the leaves were washed, dried and grinded. Then, 100g of biomass were added to 1L of distilled water that had been previously heated at 95°C. The infusion was left to cool down to 60°C and then it was filtered to remove fibrous material. Finally, this aqueous solution was concentrated until achieving a volume of 100mL.

The synthesis of silver nanoparticles involved the preparation of an initial concentrated solution of 100mM silver nitrate (AgNO₃ 99%, purchased from PanReac AppliChem) by diluting 1.7g of the metallic salt in 100mL of distilled water. Afterwards, 1.0mM and 10mM diluted solutions were obtained from the original. Green chemistry methodology was implemented for the synthesis of silver nanoparticles by mixing 50mL of AgNO₃ solution (1.0mM and 10mM) with 5mL of the mangifera indica extract. Reaction mixture was carried out at room temperature with magnetic agitation for 1 hour. Then, silver nanoparticles were centrifuged at 5500rpm, washed with distilled water and ethanol, and dried at room temperature.

3. Results

3.1. Phytochemical components in the mangifera indica extract

Phytochemical components of mangifera indica leaves extracts were determined by using a gas chromatograph coupled to a mass spectrometer (GC/MS). This analysis allowed the identification of chemical compounds that could promote the reduction of the metallic salt during the synthesis of the silver nanoparticles. This measurement required an organic extraction of the phytochemical components using a HPLC grade ethanol. This organic extract was concentrated by using a Stuart Vertical Condensor rotary evaporator at 60°C for 15 minutes until obtaining a volume of 5mL. Chromatographic analysis was carried out by direct injection of the extract into organic phase, using an AT6890 Series Plus Agilent Technologies gas chromatograph coupled to a AT MSD 5975 Inert XL selective mass detector operating in radiofrequency full scan mode. An additional DB-5MS (J&W Scientific) 60m×0.25mm×0.25µm column with 5% phenyl-poly(dimethylsiloxane) was used for this purpose. Injection was performed in split mode (30:1) with an injection volume of 2µL.

Figure 1 displays the chromatogram obtained from this measurement and the Table 1 shows a list of the main components that were detected in this analysis. Chromatographic analysis indicated the main presence of pyrogallol (26.9%) and hydroquinone (5.1%), which can promote the reduction of silver ions, facilitating the formation of nanoparticles [5]. In the case of pyrogallol, (1,2,3-benzenoitriol) accounts for a highly reducing agent for metals. The properties of this phenol derivative allow the transformation of metallic materials into their basic electronegativity state, which corresponds to a nanometric scale. This electrochemical change causes free ions to attract each other and form nanoparticles (clusters) [6]. On the other hand, the presence of oleic acid (29.1%) can contribute to the stabilization of AgNPs by acting as a primary surfactant during the nucleation stage of this nanomaterial [7]. It is an organic monosaturated acid that is primarily found in plants. This fatty acid can be coupled to the external structure of metallic nanoparticles through carboxylate bonds (COO⁻) which can limitate size growth of the material [8].

In addition, it is important to note that chromatographic analysis showed the presence of 23.7% unidentified oxygenated compounds. This result makes it possible to hypothesize the presence of other phytochemicals such as flavonoids, aldehydes, or ketones that can be involved in the formation of the silver nanoparticles. Finally, the presence of diethyl phthalate (4.9%) was also detected. This phenomenon is associated to a plastic component. It is possible that a sample contamination had occurred as mango leaves were collected in plastic bags, and they were also stored in plastic Ziploc bags after the grinding process.

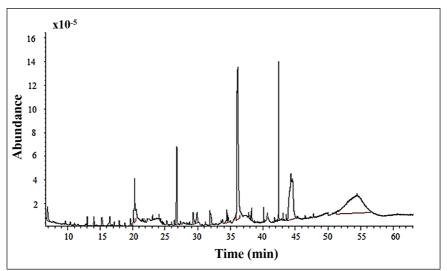


Figure 1. Sequencing chromatogram obtained for mango leaves extract.

t _R (min)	Component identification	Rel. Quantification %wt/5 mL
6.87	Acetaldehyde	1.5
13.0	Furfural	0.6
14.03	2-Furanometanol	0.8
20.32	Phenol	3.1
26.83	2,3-Dihidro-3,5-dihifroxi-6-metil-4H-Piran-4-ona	5.1
29.24	o-Catechol	1.3
31.89	Hydroquinone	1.7
36.12	Pyrogallol	26.9
38.27	2-Hydroxyacetophenone	1.0
42.46	Diethyl phthalate	4.9
54.43	Oleic acid	29.1

Table 1. Chromatography analysis for the mangifera indica leaves extract

3.2. Characterization of silver nanoparticles

The synthesized nanoparticles were characterized by UV-visible spectroscopy using a Labomed, Inc. UV 2650 equipment. Figure 2 shows the results of this analysis. There, absorption peaks in the wavelengths of 417nm (AgNPs-1) and 414nm (AgNPs-10), were observed. Such peaks are characteristic of these metallic nanoparticles [9,10].

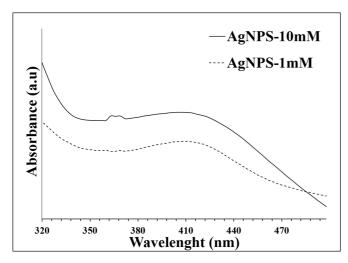


Figure 2. UV-vis spectroscopy of the silver nanoparticles that were synthesized from 1.0 and 10mM silver nitrate solution, using a mango leaves extract as reducing agent.

Figure 3 shows images of the nanoparticles that were obtained with a Quanta FEG 650 Scanning Electron Microscope (SEM). The ImageJ software was used for the determination of size distribution. Average sizes of 28±7nm and 26±5nm were obtained for the AgNPs-1 and AgNPs-10 samples, respectively. This indicates that the concentration of metallic salt did not influence the size distribution of the nanoparticles.

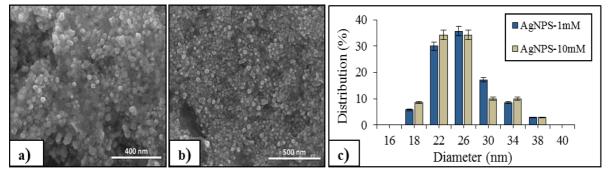


Figure 3. SEM images of silver nanoparticles synthesized by green chemistry from silver nitrate at concentrations of (a) 1.0mM, (b) 10mM, and (c) frequency histogram of the average particle size.

4. Conclusions

This research proved that the phytochemicals components present in the mangifera indica leaves extract (pyrogallol, hydroquinone, and oleic acid) can promote the formation of silver nanoparticles at room temperature with a uniform size distribution. This methodology is characterized by its easy performance and its low cost, which contributes to the environmental wealth that is achieved when no toxic compounds are used during the synthesis stage.

Acknowledgments

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