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ORIGINAL ARTICLE

Green synthesis of silver nanoparticles using Andean blackberry fruit extract

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Abstract Green synthesis of nanoparticles using various plant materials opens a new scope for the phytochemist and discourages the use of toxic chemicals. In this article, we report an eco-friendly and low-cost method for the synthesis of silver nanoparticles (AgNPs) using Andean blackberry fruit extracts as both a reducing and capping agent. The green synthesized AgNPs were characterized by various analytical instruments like UV–visible, transmission electron microscopy (TEM), dynamic light scattering (DLS), X-ray diffraction (XRD) and Fourier transform infrared (FTIR) spectroscopy. The formation of AgNPs was analyzed by UV–vis spectroscopy at λ_{max} = 435 nm. TEM analysis of AgNPs showed the formation of a crystalline, spherical shape and 12–50 nm size, whereas XRD peaks at 38.04°, 44.06°, 64.34° and 77.17° confirmed the crystalline nature of AgNPs. FTIR analysis was done to identify the functional groups responsible for the synthesis of the AgNPs. Furthermore, it was found that the AgNPs showed good antioxidant efficacy (>78%, 0.1 mM) against 1,1-diphenyl-2-picrylhydrazyl. The process of synthesis is environmentally compatible and the synthesized AgNPs could be a promising candidate for many biomedical applications.

1. Introduction

In the past decade, green synthesis of nanomaterials using various plant materials is an emerging field in Nanoscience, with emphasis to avoid the use of toxic chemicals and supports the development of ecofriendly technique. Nanomaterials (1–100 nm) of different sizes and shapes have attracted considerable attention because of their unique electronic, chemical and optical properties compared to the bulk materials (Henglein, 1989; Pileni, 1997). Recently, there has been a considerable interest in colloidal noble metal nanoparticles (MNPs) such as silver, gold and platinum in industrial applications because they exhibit different colors depending on the shape, size, and the tendency of aggregation (Lee and El-Sayed, 2006). Among noble MNPs, silver nanoparticles (AgNPs) have been given more attention due to their numerous applications in catalysis (Santos et al., 2012), biomolecular detection and diagnostic (Schultz et al., 2000), therapeutic (Eckhardt et al., 2013), micro-electronics fields (Gittins et al., 2000), sensing (Kate et al., 2011) etc.

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Several complicated and expensive methods have been employed for the synthesis of AgNPs, such as sonochemical (Kumar et al., 2014a), microwave (Yao et al., 2010), γ-rays (Rao et al., 2010), hydrothermal (Zou et al., 2007), wet chemical (Banerjee et al., 2014), laser ablation (Abid et al., 2002) and sol–gel (Gamez et al., 2013), which involve either toxic chemicals or require high capital costs, and also generate hazardous toxic wastes. Recently, the studies are focused toward greener methods for the production of large amounts of nanoparticles in non-toxic aqueous medium. Plant materials, including leaf (Kumar et al., 2014b), bark (Mehmood et al., 2014), fruit (Kumar et al., 2015a), peel (Kumar et al., 2015b), seed (Kumar et al., 2014c), and root (Shameli et al., 2012) extracts work so well in the green synthesis of AgNPs under mild experimental conditions and replacing hazardous chemicals by polyphenols, flavonoids, proteins, saponins or sugar as reducing agents as well as capping agents.

An important example of such a plant material is the dark-red color, juicy, and flavored Andean blackberry (*Rubus glaucus* Benth.) fruit. It is consumed mainly in Ecuador, Peru and Colombia as fresh, jam, juice, frozen pulp and to a minor extent as wines (Kumar et al., 2015c). We hypothesized that flavonoids, ellagitannins and anthocyanins could be applied in the green synthesis of AgNPs. Although, green synthesis of AgNPs using different plant extracts has been already explored by our research group (Kumar et al., 2014b,c, 2015a,b). In the present study, spherical AgNPs were prepared efficiently using Andean blackberry fruit extract (ABFE) as a bioreductant and stabilizer. The synthesized AgNPs were further characterized using different analytical instruments and discussed. In addition, the antioxidant efficacy of synthesized AgNPs was also evaluated against 1,1-diphenyl-2-picrylhydrazyl (DPPH·).

2. Materials and methods

2.1. Synthesis of AgNPs

Silver nitrate, $AgNO_3$, 99.0% was purchased from Spectrum, USA and DPPH·, >99.5% was purchased from Sigma Aldrich, USA. The ABFE was prepared by the earlier method (Kumar et al., 2015c). The collected fresh blackberry fruit (5 g) was washed thoroughly and heated (62–65 °C) in 50 mL of deionized water for 60 min. After cooling, the red color extract was filtered using Whatman paper No. 1. For green synthesis, 1.0 mL of ABFE was mixed with $AgNO_3$ (10 mL, 1 mM) solution and kept at 25 °C. Green synthesis of AgNPs was confirmed by the appearance of yellowish-orange solution with lapse of time.

2.2. Radical scavenging activity

The free radical scavenging activity of the AgNPs was measured by using the DPPH·-method adapted from Kumar et al. (2014b,d) with slight modifications. An aliquot (1000–200 μL) of AgNPs or control and (1000–1800 μL) of H_2O was mixed with 2.0 mL of 20 μM (DPPH·, 0.2 N) in absolute methanol. The mixture was vortexed vigorously and allowed to stand at room temperature for 30 min in the dark. Absorbance of the mixture was measured spectrophotometrically at 517 nm, and the free radical scavenging activity was calculated using Eq. (1):

$$\text{Scavenging effect } (\%) = [1 - \{\text{absorbance of sample}/\text{absorbance of control}\}] \times 100 \quad (1)$$

The scavenging percentage of all samples was plotted. The final result was expressed as % of DPPH· free radical scavenging activity (mM).

2.3. Characterization of AgNPs

The synthesized AgNps were characterized with the help of a UV–visible single beam spectrophotometer (Thermo Spectronic, GENESYS™ 8, England). Transmission electron microscopy (TEM) and selected area electron diffraction (SAED) were recorded digitally (FEI Tecnai G2 spirit twin). The hydrodynamic size distributions and polydispersity index (PDI) of nanoparticles were analyzed by using dynamic light scattering (DLS) instrumentation (HORIBA LB -550). X-ray diffraction (XRD) studies on thin films of the nanoparticle were carried out using a PANalytical brand θ–2θ configuration (generator-detector) X-ray tube copper $\lambda = 1.54$ Å and EMPYREAN diffractometer. Fourier transform infrared (FTIR-ATR) spectra were recorded on a Perkin Elmer (Spectrum two) spectrophotometer to detect the functional groups involved in nanoparticles synthesis.

3. Results and discussion

3.1. Visual and UV–visible study

Fig. 1 shows the visual studies of the AgNPs synthesis for 48 h at room temperature. The addition of ABFE to the aqueous $AgNO_3$ solution resulted in the yellowish orange color due to the surface plasmon resonance (SPR), that strongly depends on the particle size, dielectric medium and chemical surroundings (Kumar et al., 2014b,a; Kumar et al., 2015b). The reduction of aqueous Ag^+ ions by the ABFE was easily analyzed by UV–visible spectroscopy. In the 0.5 h of synthesis, the absorption spectrum shows no peaks in the range of 380–480 nm but after 3.5 h, a new peak appears around 380–480 nm. The result shows the synthesis of AgNPs started within 3.5 h after Ag^+ ions contact with the ABFE. A broad absorption peak

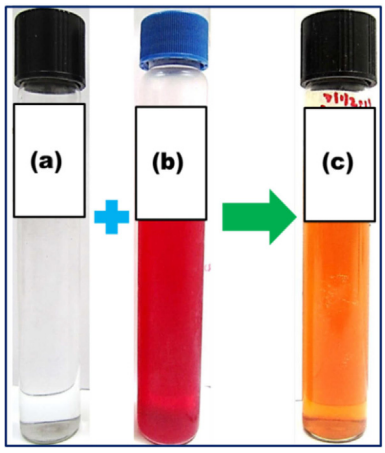

Figure 1 (a) 1 mM $AgNO_3$, (b) ABFE and (c) AgNPs.