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Green Biological Fabrication and Characterization of Highly Monodisperse Palladium Nanoparticles Using Pistacia Atlantica Fruit Broth

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ABSTRACT

The development of green and safe processes for the synthesis of nanomaterials is one of the main aspects of nanotechnology. In this study, a biological, inexpensive and rapid process for the fabrication of palladium nanoparticles using the aqueous broth of Pistacia Atlantica fruit as a novel biomass product is reported without using extra surfactant, capping agent, and template. The synthesized palladium nanoparticles were confirmed and characterized by various spectroscopic techniques including UV-Visible spectroscopy, X-ray diffraction (XRD), transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy-dispersive X-ray spectrometer, Fourier transform infrared spectroscopy and Zeta-potential measurement. The results indicate that the spherically shaped Pd nanoparticles were successfully prepared in aqueous media in accordance with the principles of green chemistry with desired stability and crystalline in nature with face centered cubic geometry. Also, the results of transmission electron microscopy (TEM) confirmed preparation of very stable nanoparticles with the small diameter below 15 nm.

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INTRODUCTION

Presently, the synthesis of metallic nanomaterials is still a considerable aspect in nanotechnology area, because of their valuable futures which make them useful and suitable in a broad range of fields such as catalysis, biological labeling, sensors technology, electronics, optics, optoelectronics and recording media [1]. Among various metal nanoparticles, palladium nanoparticles (PdNPs) have drawn particularly increased interests due to their incredible catalytic activities [2,3]. PdNPs are also having extensive applications in other diverse fields like as active membranes [4], petroleum [5], full cells [6] and sensors [7]. Anyway, fine control of the shape and size of PdNPs via novel preparation techniques is quite a paramount challenge due to the existence of the high cohesive energy between the palladium nanoclusters. Classical ways for fabrication of PdNPs in water media were described by Turkevich and Kim [8]. More studies have shown that the size, distribution, morphology, stability and properties of metal nanoparticles specially PdNPs are influenced by the experimental conditions, reducing agents and the adsorption processes of stabilizing agents with these nanoparticles. Generally, PdNPs could be synthesized by a wide variety of chemical and physical methods such as polycol reduction [9], sonochemical reduction [10], hydrothermal reaction [11], plasma [12] and laser reduction method [13]. Most of these reported methods for synthesis of PdNPs are extremely expensive and also involve the use of toxic and hazardous materials, which may pose potential environmental

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and biological risks [14]. Therefore, based on the necessity to develop a novel and environmentally friendly nanostructure preparation methods that uses less toxic precursors, fewer reagents and simpler synthetic steps, scientists are now beginning to link nanotechnology with 'green chemistry' [15].

Green chemistry has received considerable attention over the past two decades, due to efforts aimed at utilization of non-hazardous chemicals and renewable materials simultaneously with the total elimination or minimization of generated waste [15]. Biological reduction of Pd (II) ions has been known as an attractive eco-friendly and economical method because of the fact that biological systems exhibits low energy consumption at room temperatures and nearly neutral pH(s) which makes them environmentally safe routes [16].

In spite of these obvious benefits of biological preparation methods, there are limited biomimetic approaches reported for preparation of PdNPs. Indeed, the preparation of Pd^0 using green methods which uses microorganisms and plant-based techniques has not established as much as for silver and gold. On the other hand, the use of plant extracts is preferred currently due to the risk of microbial contaminations, the need to maintain cell cultures and expenditures of preparation methods in microbial based synthesis [17]. In the past years, a number of herbal and plant source materials have been studied for the synthesis of PdNPs in different sizes and shapes [18-21], but in some cases, their synthesis strategies are still complicated and the proposed approaches suffers from disadvantages such as the unsuitable regulation and the slow rate of process [17]. It should be noted that, the composition, sizes, shapes and stability of nanostructures are important factors in all of aforementioned applications. But in most cases, the bio-synthesized nanoparticles are not monodispersed enough, however this factor plays a key role in some of applications and therefore more attempts are required for their controlled fabrication [9, 22].

For these reasons and since palladium nanoparticles are nowadays widely applied to human contacting areas, in the present study, a green, easy and rapid synthesis method is introduced for the biosynthesis of highly monodispersed PdNPs using aqueous broth of Pistacia Atlantica plant's fruit at ambient conditions. To the best of our knowledge this is one of the few attempts of using Iranian local plant materials for synthesis of metallic nanoparticles. Notably, phytochemistry studies of this biomass demonstrates that it contains several organic compoonents such as fatty acids, phytosterols, phenols, tocopherol and starch which these species could strongly be act as reductant and stabilizing agents [23]. UV-Vis absorption spectroscopy, transmission electron microscopy (TEM), scanning electron microscopy (SEM), energy-dispersive X-ray spectrometer (EDS), X-ray diffraction analysis (XRD), Fourier transform infrared spectroscopy (FT-IR) and zeta potential measurements were used to characterize the as-prepared Pd nanoparticles.

MATERIALS AND METHODS

All chemicals used were of analytical grade or of the highest purity available. Palladium (II) chloride ($PdCl_2$, 99.0%) as the source of palladium ions was received from Fluka and used without further purification. All solutions were prepared with double-distilled deionized water. A solution containing HCl (0.1 M) was prepared by dissolving known amounts of concentrated HCl, purchased from Merck (Darmstadt, Germany), with deionized water to the desired concentration. The Pistacia Atlantica was used to make the biomass extract.

Preparation of the biomass and plant material

To obtain the aqueous extract of the in question plant, the fruits of the plant (biomass) were collected and washed thoroughly with sterile distilled water, dried in shade and crushed to fine powder. The attentively weighted obtained dry powder, 10.0 g, were added to 200 mL deionized water in a 500 mL Erlenmeyer flask and then boiling the mixture for 25 min in a water bath at 85 °C. Then, the obtained mixture was filtered 3 times and purified by centrifugation at 6000 rpm for 20 min prior to use for further experiments. This solution was stored at 4 °C and used within a week.

Synthesis steps of palladium nanoparticles

$PdCl_2$ solid powder was firstly dissolved by a few drops of HCl (0.1 M) with the aid of sonication. Then, the prepared solution was diluted with deionized water to obtain the desired concentrations of Pd (II). In a typical synthesis of PdNPs, 15 mL of

freshly prepared broth were added to 100 mL of 1 mM aqueous palladium (II) chloride solution. The Pd nanoparticles solution thus obtained was purified by repeated centrifugation at 10000 rpm for 20 min. The effect of temperature on the synthesis rate of palladium nanoparticles was studied by carrying out the reaction in a water bath at 25-85 °C in dark conditions. Based on the monitoring the color of prepared nanoparticles and UV-Vis spectra, better results in the case of synthesis quality, high dispersity, the amount of nanoparticles and biomass extract were obtained at 85 °C. Therefore, the optimum temperature in the preparation of PdNPs was selected 85 °C. It must be noted that expect the nanoparticles amount the effect of temperature on other characterizations of synthesized PdNPs was negligible.

Characterization and apparatus

The following procedures and equipments were used to characterize the as prepared Pd nanoparticles; UV-Vis absorption spectra were recorded using a double beam, T80+ UV-Vis spectrophotometer PG (china) with 1cm quartz cells by using wavelength between 300 to 600 nm at a resolution of 2 nm. 3 mL solution was used for each measurement. A scanning electron micrograph was recorded for the further characterization and identity the morphology of PdNPs using a Philips XL-30 electron microscope operated at 30 kV and equipped with an energy-dispersive X-ray spectrometer (EDS). To obtain elemental analysis of the as prepared nanoparticles, the freeze-dried PdNPs were mounted on specimen stubs with double-sided taps, coated with gold in a sputter coater (BAL-TEC SCD-005), and examined at 12-16 kV with a tilt angle 45˚. Transmission electron microscopy (TEM) images were obtained by a Philips EM 208 electron microscope at 100 keV. Samples for transmission electron microscopy were prepared by placing known drops of the sonicated aqueous suspension on carbon coated copper grids and allowing water to completely evaporate. X-ray diffraction (XRD) measurement for the crystalline character clarification was performed on an X-ray diffractometer (XRD; Philips PW-180, Germany) operated at a voltage of 40 kV with Cu Kα (1.5418 Å) radiation. Freeze drying of the centrifuged samples for scanning electron microscopy and FTIR was carried out using an ALPHA 1-4 freeze dryer (CHRIST, Germany) under vacuum conditions at -50˚ C for 40 h. Stability of the PdNPs and reactive matter in broth was investigated using FTIR instrument (Thermo Nicolet Nexus® 670, USA) in the transmittance mode between 400 and 4000 cm^{-1}. A Zetasizer particle size analyzer ZEN 3600 instrument (Malvern, Malvern Instruments Ltd, UK) was used to determine the Zeta (ξ) potential analysis. The measurements were performed at 25˚C in disposable plastic vials (Brand, Germany) after sonication of the samples for at least 1 h in pulsed mode.

RESULTS AND DISCUSSION

Formation monitoring and UV-Visible spectra of PdNPs

UV-Visible absorption spectroscopy is one of the fundamental techniques to characterize of the nanoparticles in aqueous suspensions [24]. Reduction of the aqueous Pd (II) ions to Pd^0 was tracked visually (changes in color) and also was monitored by UV-Vis spectroscopy in the range of 300 – 600 nm. Fig. 1 clarifies the change in the UV-Vis optical response recorded after complete reaction of constant concentration of Pistacia Atlantica extract [(line A): control] with increased concentration of Pd(II) solution [(line B): 0.0005 M, (line C): 0.001 M and (line D): 0.0015 M], respectively. The figure inset shows photographs of the vials labeled A-D containing control (biomass) and different concentrations of green synthesized and Pistacia Atlantica- reduced PdNPs, respectively. By the way, the addition of Pistacia Atlantica broth into the different and increased concentrations of palladium ions solution, exhibits the gradual change in color from transparent yellowish (Fig. 1, vial B) to dark brown (Fig. 1, vials C and D) during 5 min at 85˚C indicating the generation of PdNPs. Anyway, the final observed color (dark color) is characteristic of the surface plasmon resonance (SPR) band of PdNPs in solution and was due to the excitation of surface plasmon vibrations in the green prepared Pd^0. As can be seen, the observed peak at 382 nm in UV-Vis spectrum of 5×10^{-4} M Pd (II) solution (Fig. 1, line B) indicate the presence of the biomass with Pd (II) ions in mixture over the reduction. After the reduction process completed, the peaks shifted to shorter wavelengths along with increasing of Pd (II) concentration (Fig. 1, lines C). The absence of the absorption peaks above 300 nm in samples (C to D) revealed the complete reduction of the

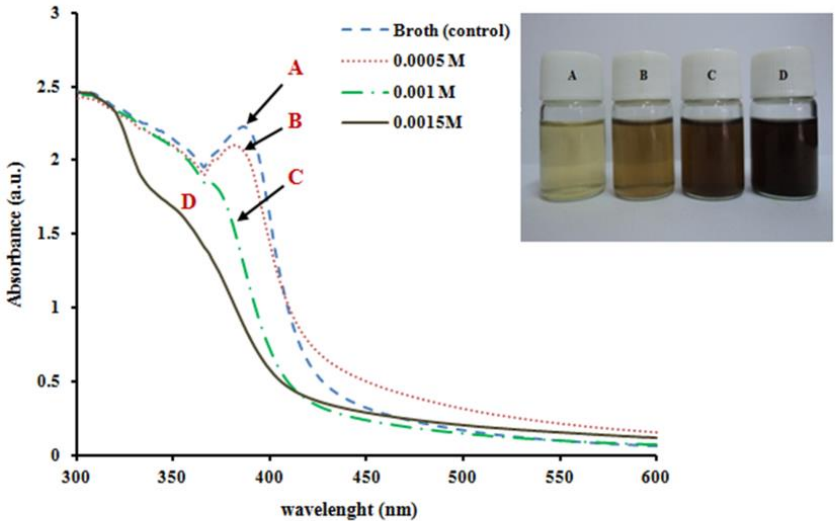

Fig. 1. UV–visible spectra of biomass and PdNPs prepared with different concentrations of $PdCl_2$ solution after bioreduction by Pistacia A broth at 85 °C: (A) UV– visible spectrum of biomass aqueous solution (control); (B) 5×10^{-4} mol L^{-1}; (C) 1×10^{-3} mol L^{-1}; and (D) 1.5×10^{-3} mol L^{-1}. The inset shows a digital image of the as-prepared palladium colloidal solution and the prepared broth solution before (A) and after reaction with Pd (II) ions (B, C and D).

initial Pd (II) ions [25]. It should be noted that, the reduced sample showed continuous absorption in the range of 300–600 nm indicating the formation of nanosized Pd particles [26].

Possible mechanism of PdNPs formation

It is important to understand the biosynthetic mechanism involved in the fabrication of PdNPs mediated by a biological system in order to gain better control of the process and products. So far, little is known about the interaction between bio-molecules and PdNPs, though several analyses have been made. We believe that the biological compounds such as phenolic and triterpenoidic groups that are available in the biomass broth could be probably responsible for this bioreduction approach and the sugar compounds such as starch that can be found in prepared extract acts as a stabilizing agent which could avoid the aggregation of synthesized nanoparticles in water media [27, 28]. The reaction between biomass and the Pd (II) species might occur according to the following equation [20]:

$$nPd(II) + 2R{-}(OH)n \rightarrow nPd(0) + 2nR{=}O + 2nH^{+}$$

Where, R and n represent heterocycle or alkyl groups and the number of the hydroxyl groups oxidized by Pd (II) species, respectively [20]. Hereby, it is reasonable to speculate that polyol components in the Pistacia A. fruit were oxidized into aldehydes or ketones while Pd (II) ions were reduced to elemental palladium. It should be noted that, the slight decreasing in pH values from 3 (before reduction of Pd (II) ion solution) to 1.6 (after reduction completed) confirmed the proposed mechanism for assembly of PdNPs by this green approach.

Crystalline phase study

The crystalline properties of the prepared palladium nanoparticles were investigated by X-ray diffraction (XRD). The intense peaks due to (111), (200) , (220) and (311) Bragg reflection at 2θ= 40.48°, 48.72° , 67.8° and 82.0, respectively, that confirmed the formation of palladium nanoparticles with face centered cubic (fcc) crystalline structure (Fig. 2).

The average particle size of the palladium crystallites is evidenced by Debye–Scherrer's formula as follows [20, 21]:

$$d=0.89(\lambda)/\beta_{2\theta}\cos\theta$$

Where, λ is the X-ray wavelength (1.5418 Å), θ is the Bragg diffraction angle, and $\beta_{2\theta}$ is the peak full width at half-maximum (FWHM). The calculated crystallite size of the synthesized PdNPs using peak broadening profile of (111) peak at 40.48° is 3.63 nm. The observed peak broadening and noise

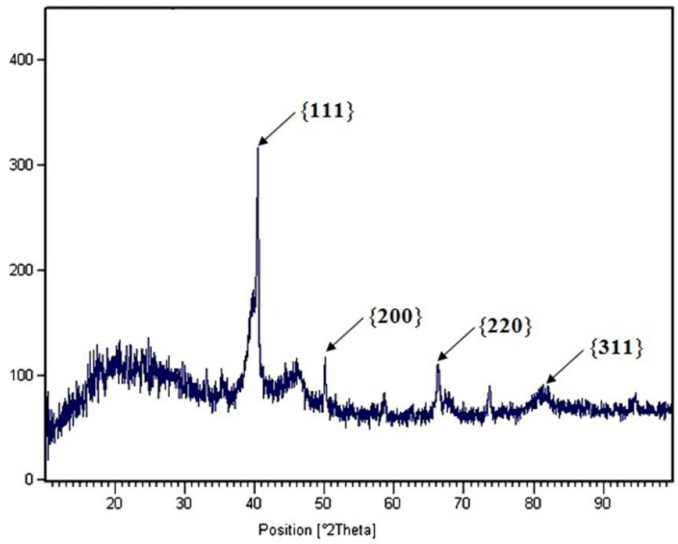

Fig. 2. X-ray diffraction pattern (XRD) of the as- prepared palladium nanoparticles.

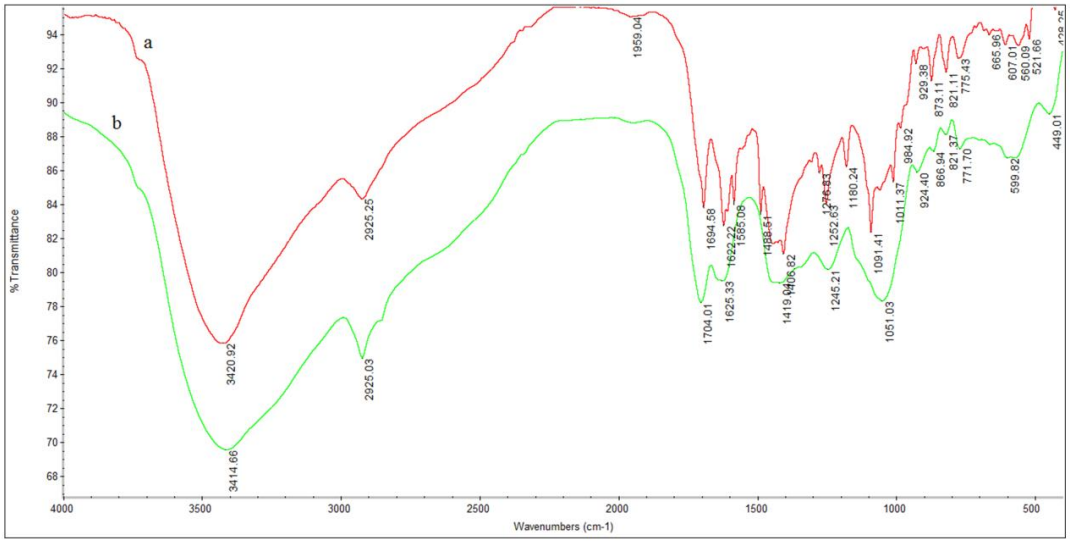

Fig. 3. FTIR spectra of (a): Pistacia Atlantica broth (b): obtained PdNPs powder + broth.

were probably related to the effect of nanosized particles and the presence of various crystalline biological macromolecules in the plant extracts.

FTIR analysis

FTIR analysis was carried out to investigate the interactions of functional groups and different bio-molecules in aqueous extract of biomass with palladium (II) ions at room temperature. A typical FTIR spectrum of the freeze-dried powder of obtained PdNPs is shown in Fig. 3 (band-b). The control spectra [(Fig. 3, band (a) not treated with palladium chloride)] showed several peaks indicating a complex nature of the biological material including some compounds e.g., phytosterols, tocopherol, polyphenol and fatty acids. As can be seen, the FTIR band at 3420 cm^{-1} and 2925 cm^{-1} could be assigned to the stretch vibration of OH related to phytosterols and polyphenols in biomass broth. However, the bands in 1091 cm^{-1} probably indicate the vibration of C-O-C in tocopherols and 1694 cm^{-1}

incorporated to the stretch vibration C=O of carboxylic acids (oleic acid and linoleic acid). Moreover the absorbance band centered at 1585 cm^{-1} and 1488 cm^{-1} can be assigned to the vibration of –C=C (aromatic ring). By the comparison of these results with earlier reports [29, 30], we proposed that the reduction of the palladium ions may be performed by the oxidation of hydroxyl groups to carbonyl groups that may be participating in the process of nanoparticles synthesis. Furthermore, the observed bands in 599 cm^{-1} to 1625 cm^{-1} in Fig. 3 (band-b) are related to various functional groups of starch and fatty acids of broth that may be able to attach through its hydroxyl groups to the surface of the synthesized nanoparticles that cause to their stability against the aggregation.

Surface morphology and elemental composition studies

To gain further insight into the features of the green synthesized PdNPs, analysis of the samples was performed using scanning electron microscopy (SEM), and EDX techniques. The morphology and size of PdNPs in the colloidal solutions and their size distribution were also investigated by transmission electron microscopy (TEM).

Transmission electron micrograph in Fig. 4(A) displays palladium particles obtained under optimum conditions. From transmission electron micrograph, it has been clarified that PdNPs mostly exists in spherical shapes with the average size of 10 nm. Fig. 4(B) also shows the scanning electron micrograph of PdNPs obtained from the proposed bioreduction method at 30000 – fold magnification. The EDX (Fig. 4C) recorded in the spot-profile mode from one of the densely populated nanoparticles region shows approximately strong signals of Pd^0 and no peaks belonging impurities were detected.

Zeta potential measurement

Pd nanoparticles were characterized by dynamic light scattering (DLS) technique to check the Zeta (ξ) potential (surface charge). ξ -potential is a fundamental parameter for the characterization of stability in aqueous nano-suspension, which is

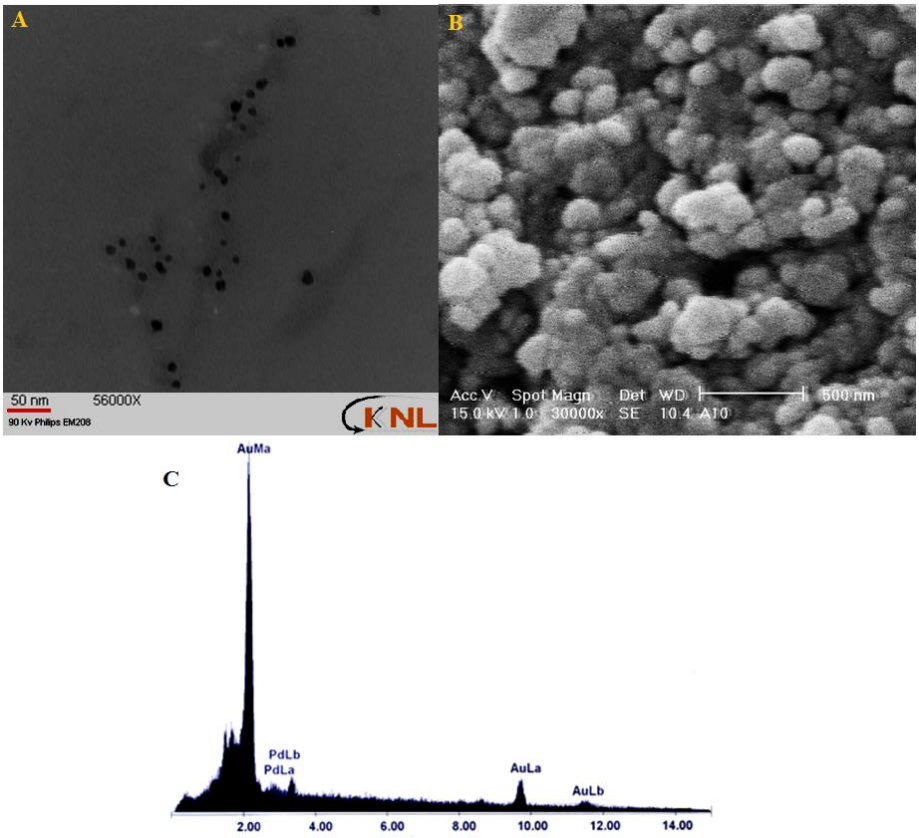

Fig. 4. Transmission electron microscopy (A), scanning electron microscopy (B) and EDX (C) analysis of synthesized PdNPs.

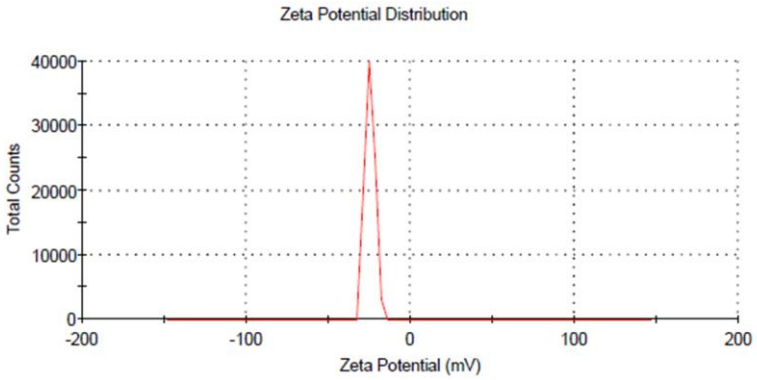

Fig. 5. Zeta potential measurement histogram of green prepared PdNPs.

related to both surface charge and the functional group around the particles. For the ξ -potential measurements, a known concentration of samples were prepared after 1 h sonication, leaching and subsequent centrifugation. Fig. 5 depicts the zeta potential analysis of prepared PdNPs. A minimum of zeta potential (>25 mV or < -25 mV) demonstrates stable nanostructures. As the zeta potential approaches "0" or the point of zero charge, the attraction between nanomaterials exceeds the repulsive forces between the structures resulting in agglomeration [31-33]. As appeared in Fig. 5, the zeta potential of -24.5 obtained which shows a large amount of negative surface charge of nanoparticles and inhibit their accumulation in the nano-suspension form.

CONCLUSIONS

To conclude, Pd nanoparticles were successfully prepared using Pistacia Atlantica extract mediated rapid bio-synthesis process. The oxidation of hydroxyl group in biomass could be responsible for the reduction of Pd (II) ions and the stabilizing agent such as starch and fatty acids led to inhibit the aggregation of fabricated nanoparticles. The synthesized PdNPs were characterized using XRD and confirmed the fcc crystalline structure. The prepared nanoparticles were also characterized by a variety of other standard analytical techniques. The results of scanning electron microscopy (SEM), transmission electron microscopy (TEM) and EDS analysis confirmed successfully synthesize of stable PdNPs with the small diameter below 15 nm, desired stability and high purity chemical phase. The zeta potential analysis of nanoparticles showed the negative surface charge (-24.5 mV) of nanoparticles.

CONFLICT OF INTEREST

The authors declare that there are no conflicts of interest regarding the publication of this manuscript.

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