

Microwave-assisted green synthesis of Gold nanoparticles and its catalytic activity

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Abstract

The present work demonstrated a green approach of synthesis of gold nanoparticles using *Eupatorium odoratum* leaf extract as reducing and stabilizing agent assisted with microwave irradiation. Effects of various concentrations of leaf extract on the preparation of gold nanoparticles have been investigated and it was monitored by undertaking UV-vis spectroscopic studies. The experimental results showed that the surface Plasmon resonance (SPR) peak blue shifted with increase in the concentration of leaf extract indicating the decrease of nanoparticles size, which was further confirmed by the Dynamic light scattering (DLS) data. Synthesized particles were further characterized by Fourier Transform Infra-Red spectroscopy (FTIR) and Transmission Electron Microscopy (TEM). All the characterization results confirmed the formation of stable spherical mono-dispersed gold nanoparticles with size ranging from 10-20 nm. The catalytic activity of prepared gold nanoparticles was checked by reducing Nitrophenol to Aminophenol in presence of an excess amount of sodium borohydride. The progress of the reaction was examined by observing the absorbance peak of UV-vis spectroscopy. The study showed positive results and it was found that gold nanoparticles synthesized with 600 μ L leaf extract have greater catalytic activity. It was also found that gold nanoparticles remained stable for long duration of time.

Keywords: Bio-synthesis; Catalytic Activity; *Eupatorium Odoratum*; Gold Nanoparticles; Nitrophenol.

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INTRODUCTION

Gold is one of the most investigated metal nanoparticles in modern days due to its size dependent chemical and physical properties such as biocompatibility, surface modifiability, and visible-NIR optical response [1-3]. Due to the electron confinement effects, such nanoparticles possess unique optical, electronic, catalytic properties [4-7]. These unique properties lead them to be used for versatile applications such as therapeutic agents, drug delivery, in catalyzing reactions, biological sensing, surface-enhanced Raman scattering, fuel cells, electro-catalytic properties toward Oxygen reduction reaction, and hydrogen storage, etc [8-12]. It is believed that the surface of gold nanoparticles play essential role in catalytic reactions [13, 14]. At the

nanoscale, the high surface-area-to-volume ratio of gold NPs facilitates them to increase chemical reactivity of many organic and inorganic reactions. Examples of organic reactions that use gold nanoparticles include oxidation of hydrocarbons, C-C coupling, hydrogenation-dehydrogenation, alkynes activation. In case of inorganic reactions, example are, the water-gas shifts reaction, ozone decomposition, selective oxidation of H_2 to H_2O_2 [15-17]. The reduction reaction of nitrophenol to aminophenol in presence of excess $NaBH_4$ is generally employed as a model reaction to investigate the catalytic activity of gold nanoparticles, because of easy monitoring of the reaction result [18]. Nitrophenols and their derivatives are widely used as intermediates for indicators and synthetic dyes, insecticides,

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herbicides in industries which are highly toxic and potential carcinogenic agents and do severe harm to the living organism of the ecosystem [18]. Hence, these pollutants are removed by various approaches such as microbial degradation, electro-coagulation, electro-Fenton, etc. Further, the reaction product, aminophenol, is an important intermediate for analgesics and antipyretics medicine [19]. Earlier, gold was not fully recognized for its catalytic ability but recent studies of gold nanoparticles have shown these particles as highly active catalysts [20]. Size of nanoparticles is crucial in determining their catalytic properties. Significant work has been done to examine the size-dependent catalytic activity of nanoparticles [15, 21]. In 1987 Haruta *et al.* showed that gold nanoparticles of size below 5 nm can be very effective catalysts [22]. But the size or surface area is not the only parameter to be considered for the catalytic behavior of gold nanoparticles. Shape and capping agent also plays active roles to enhance the catalytic activity. Kim and co-worker showed anisotropic gold nanoparticles enhance reaction rates of 4-NP to 4-AP under light irradiation [23]. The challenges are to understand how the size and shape of nanoparticles affect the catalytic activity and synthesize these nanoparticles with better control over their structure and size. Till date, various synthesis techniques have been developed, wet chemical synthesis methods to different physical methods. Though in the wet chemical methods, the yield of nanoparticles is very high it carries hazardous synthetic reagents like sodium borohydride, cetrymethyl ammonium bromide (CTAB), etc. Green synthesis of nanoparticles has overcome these problems by reducing hazardous and toxic bi-products and become an alternative to the synthetic chemicals techniques. Recently lots of works have been done on green approach to synthesize nanoparticles using biological entities like plant biomass, plant leaf extract, bacteria, fungi, and other microorganisms [24-26]. Synthesis of nano-materials using plants extracts is preferable in comparison to other biological methods like microbial procedure which require prolonged period of culturing cells [27]. Such green methods are free from any hazardous synthetic chemicals. Moreover, these methods are single step one pot, low cost and eco-friendly [28].

In the present work, we demonstrated a one-pot process of synthesis of gold nanoparticles using microwave (MW) irradiation using

Eupatorium odoratum leaf extract as reducing as well as stabilizing agent [29]. The plant is a tropical species of a flowering shrub native to North America and South America but found almost everywhere worldwide. Different parts of this plant are traditionally used as an antibacterial, anti-inflammatory, and antiseptic agent. It also has anti-oxidant, wound healing and many other significant medicinal properties [30, 31]. Leaf of the Plant has been found to be a rich source of protein, biotin, ascorbic acid, steroids flavonoids, and various polyphenolic compounds and many others molecules of medicinal importance. Among these, some are strong reducing agents and some are good stabilizing agents. In the present work, we investigated the effects of different concentrations of leaf extract on the synthesis of gold nanoparticles and evaluated the catalytic activities of these nanoparticles.

MATERIALS AND METHODS

Materials

Gold chloride pure (49%) was purchased from SRL India. 2, 4, 6 tri nitrophenol (99.99%) purchased from qualigens and sodium borohydride were purchased from SDFCL India. De-ionized water (DIW) was used for all reactions and laboratory purposes.

Leaf extract was prepared by boiling the dry leaves of *Eupatorium odoratum* with de-ionized water. The leaf extract after filtration was cooled to room temperature and kept at 4°C for future uses.

Synthesis of Gold Nanoparticles

GNPs were synthesized by reducing the HAuCl_4 solution using *Eupatorium odoratum* leaf extract under microwave heating. Six sets of the aqueous solution of HAuCl_4 (0.5mM, 5ml) were prepared in conical flasks. In each of them, 5 ml of DIW were added and then leaf extract was added to each of this solution in volumes varied from 100 μl to 600 μl in 100 μl steps. Each flask was then kept in a microwave oven at 300 watts for 60 seconds.

Characterization Techniques

UV-Vis spectroscopy was performed using Perkin Elmer Lambda 25 to observe surface Plasmon resonance peak (SPR). Hydrodynamic sizes of GNPs were evaluated using Malvern Nano-S90 Zeta sizer. Transmission electron microscopy (TEM) images were taken to confirm the formation of the nanoparticles and also to investigate their

morphology and size using JEM-2100 TEM. Fourier transform infrared spectroscopy (FTIR) of the gold nanoparticles was performed using Perkin Elmer Spectrum Two spectrometer to identify the functional groups associated with the synthesized GNPs.

Catalytic activity of Gold nanoparticles

The reduction of 2, 4, 6-tri nitrophenol by water-dispersed GNPs in the presence of NaBH₄ was carried out to examine the catalytic activity of the synthesized GNPs. In a glass cuvette 40 μL of 1 mM 2, 4, 6-tri nitrophenol was mixed with freshly prepared 400 μL 1 mM NaBH₄. Then, 300 μL of GNP solution and 3ml de-ionized water were added to the above reaction mixture. Same experimental procedure was followed to check the catalytic activity of all the six sets of GNPs. The reaction effect was observed by taking UV-vis absorbance spectrum of the mixture periodically after every 2 minutes. A controlled experiment was also carried out using mixtures of NaBH₄ and nitrophenol to observe the reduction effect of NaBH₄.

RESULTS AND DISCUSSION

Synthesis of Gold Nanoparticles

Gold nanoparticles were synthesized as per the conventional wet chemical protocol. But instead of using synthetic chemicals, phytochemicals

were used as both reducing agents as well as stabilizing agents in the present work. After repeated experimentations in different reaction conditions, it was found that heating is one of the important parameters for increasing the kinetics of the reaction. In the present work, microwave irradiation was chosen as a source of heat over any other conventional source of heat, because microwave produces heat throughout the volume uniformly and rapidly. Just after 60 seconds of irradiation, the color of the solution changed from transparent to red. It is the indication of the formation of gold nanoparticles, as several literatures established the fact that colloidal gold solution shows intense color in nano form [25]. The effect of various concentrations of leaf extract on the synthesis of GNPs was also investigated. The effect was monitored by taking UV-vis spectra of synthesized GNPs. The variation of colors of colloidal gold for increasing concentrations of leaf extract is displayed in Fig.1a. The minimum volume ratio of precursor solution to the LE solution used is about 100:1. So it is clear that very less LE is required for the synthesis of gold nanoparticles.

Characterization of synthesized GNPs.

Uv-vis spectroscopic analysis of GNPs.

The UV-vis absorption spectra of each set of GNPs exhibited distinct surface plasmon resonance

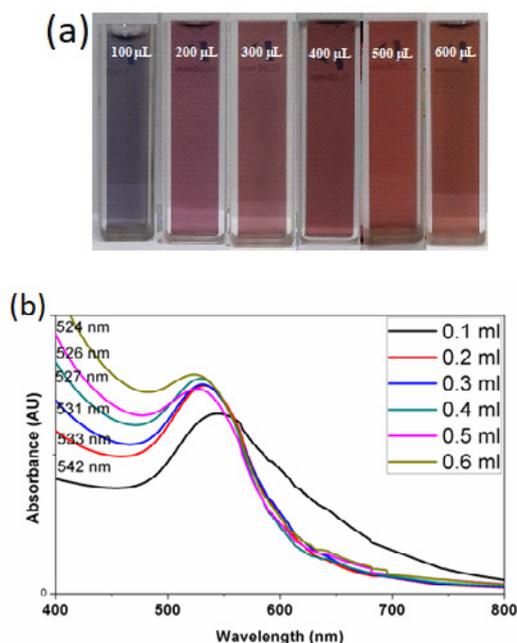


Fig. 1. (a) Color variation in synthesized colloidal gold for various concentration of LE. and (b) corresponding UV-vis graph showing SPR peak at different wavelength.



(SPR) band (Fig.1b). The figure clearly shows that the absorption peak position decreases from 542 nm to 524 nm with an increase in the leaf extract. This is actually an indication of a decrease in the size of nanoparticles. This is further confirmed by the dynamic light scattering (DLS) observation. It is also observed that the absorbance value increases for the GNPs synthesized with higher amount of leaf extracts. It is an indication of the formation of a higher number of GNPs.

Dynamic light scattering

In order to check the variation in sizes of GNPs with the increasing amounts of leaf extracts, all the six sets of NPs were characterized by DLS. It

can be seen clearly from the spectra (Fig. 2) that the hydrodynamic sizes of NPs decreased with the increasing amounts of leaf extract. The average size of GNPs synthesized with 100 μ l, 200 μ l, 300 μ l, 400 μ l, 500 μ l and 600 μ l of leaf extracts were found to be 42.64, 38.85, 30, 28.6, 26.45 and 24.30 nm respectively.

Analysis of TEM image

TEM was employed to investigate the morphology of synthesized GNPs. GNPs synthesized with 600 μ l of leaf extract were taken for TEM investigation. The TEM micrograph (Fig.3. (a)) showed that the synthesized nanoparticles were well separated from each other and of

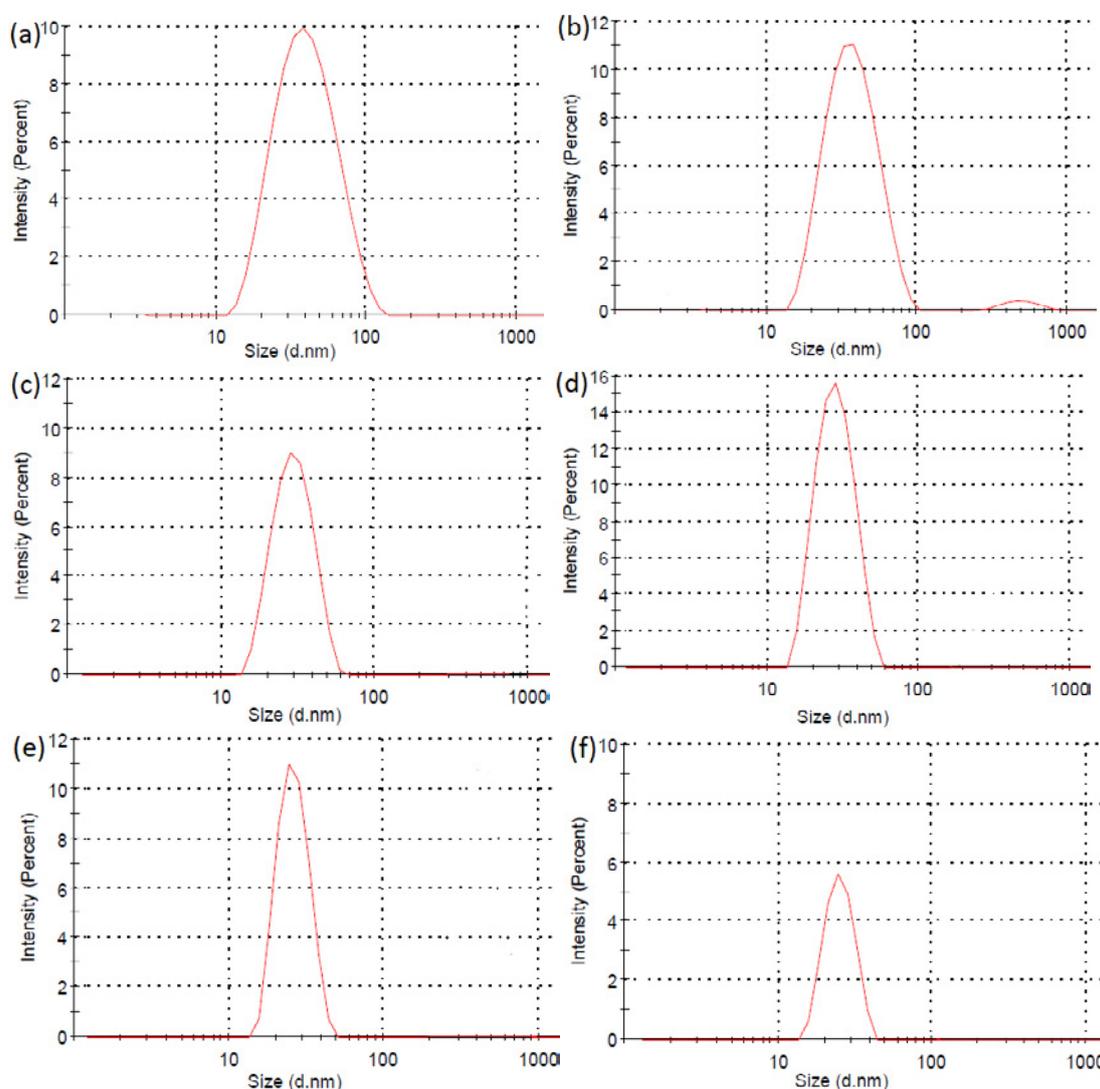


Fig. 2. Dynamic light scattering (DLS) spectra of synthesized GNPs for (a) 100 μ l, (b) 200 μ l, (c)300 μ l, (d) 400 μ l,(e) 500 μ l and (f) 600 μ l of *Eupatorium odoratum* leaf extract.

uniform shape and size, indicating the presence of a strong stabilizing agent in the leaf extract. The sizes of nanoparticles were found to be in the range 10 - 20 nm as shown in the size distribution histogram (Fig. 3b).

Crystallinity analysis of GNPs by XRD

Data of X-ray diffraction (XRD) analysis of the gold nanoparticles in Fig.3.(c) matched well with the diffractions from metallic face-centered cubic (fcc) at $2\theta = 20^\circ, 28.92^\circ, 38.20^\circ, 44.62^\circ, 64.84^\circ$ and 74.06° , which are arises from the (200), (220), (111), (311), (220) planes (JCPDS 04-0784). The intensity of the diffraction peak of (111) was much stronger than the other peaks. The high-resolution TEM (HRTEM) image further revealed that the measured lattice fringe distance (0.22 nm) corresponded well to the (111) lattice spacing of the fcc gold.

FTIR spectral analysis of synthesized GNPs.

FTIR spectroscopy of leaf extract and leaf extract synthesized GNPs were performed in order to get the information about surface modification

of GNPs by leaf extract. FTIR graph of both leaf extract and GNP showed almost similar absorption peak (Fig.3 (d)). A broad absorption at 3389 cm^{-1} for LE and GNP is due to O-H bond present in alcohol, a weak absorption near 2900 cm^{-1} for LE corresponding to C-H stretching vibration in methylene group, vibration near 1624 cm^{-1} for the C=C band, absorption near 1405 cm^{-1} indicating the presence of hydrocarbons of methylene group [32]. While FTIR spectra of both LE and GNPs exhibited similar absorption peaks, the only peak corresponding to 2942 cm^{-1} disappeared from the GNPs spectrum indicating that functional group C-H in methylene group may be involved as a capping agent of GNPs.

The catalytic activity of synthesized GNPs.

In order to study the catalytic activity of the synthesized particles, first 2, 4, 6-tri Nitrophenol aqueous solution was mixed with a freshly prepared aqueous solution of NaBH_4 in a standard quartz cuvette. Time-dependent UV-vis absorption spectra were monitored throughout the reduction process in the absence or presence

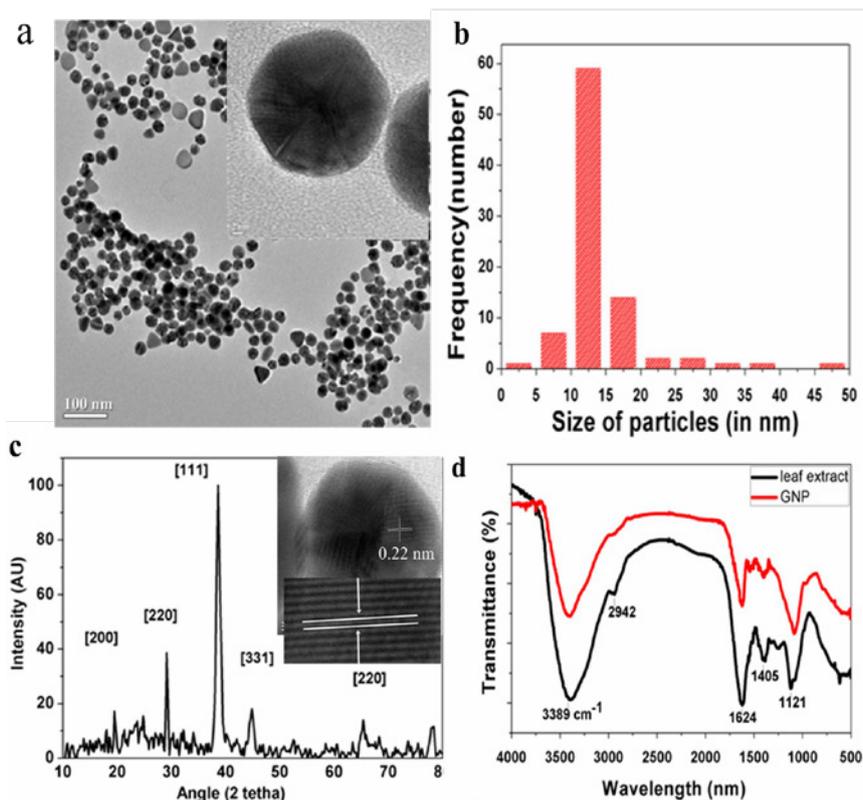


Fig. 3. (a) TEM micrograph of GNPs (600 μl LE), (b) Size distribution histogram (measuring more than 100 particles from TEM image using Image J software). (c) XRD patterns of GNPs (600 μl LE), HRTEM images (insert), (d) FTIR spectra of GNPs (600 μl LE) and pure leaf extract.

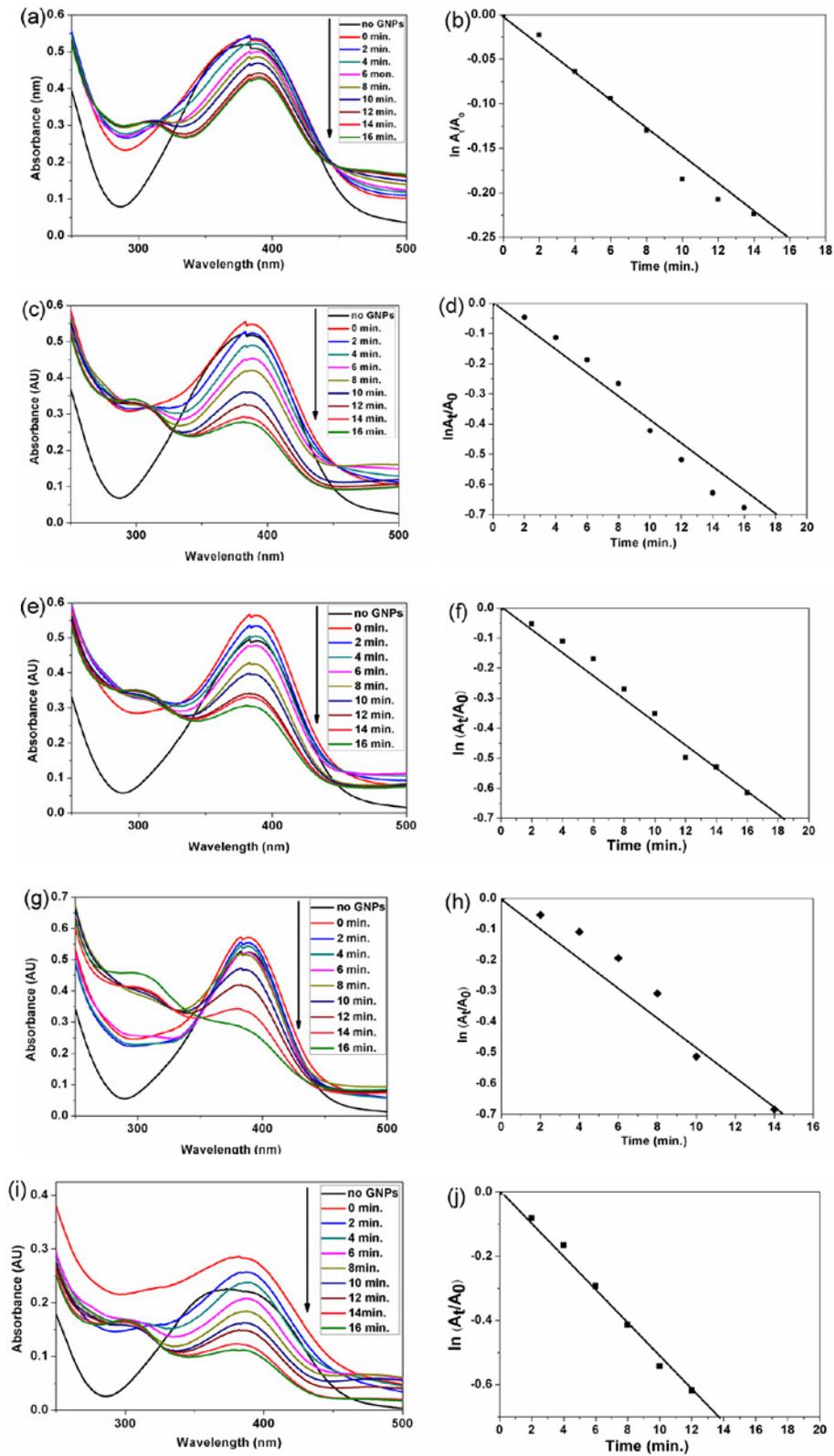


Fig. 4. (a,c,e,g,i) UV-Vis spectra showing the successive reduction of nitro phenol to amino phenol by GNP-synthesised by various concentration of LE. (200 μ l -600 μ l) in presence of NaBH_4 and the corresponding plots of ' $\ln A_t/A_0$ ' versus time (b,d,f,h,j).

of different size of Nanoparticles. Although NaBH_4 is a strong reducing agent, very little decrease of the absorbance at 390 nm (corresponding to nitrophenolate anions) was observed in 16 min without the addition of a catalyst. Moreover, during this process, the solution in clear yellow color turned into yellow-brownish rapidly and constantly released bubbles because of the formation of nitrophenolate in alkaline solution [15, 33]. The absorbance peak did not change with time indicating that the NaBH_4 was unable to convert Nitrophenol to Aminophenol. Whereas on addition of 0.3ml of colloidal gold nanoparticles into the Nitrophenol/ NaBH_4 solution, the UV-vis absorption intensity of nitrophenolate at 390 nm decreased gradually as the reduction proceeded, and simultaneously a new absorption peak at 304 nm appeared (Fig. 4), which was an indication of the reduction from NP to AP [34]. The catalytic activities of all the six sets of gold nanoparticles were evaluated under similar reaction condition as described above and displayed in Figs. 4(a, c, e, g, i). A linear relationship found between $\ln(A_t/A_0)$ and time (min.) (Figs. 4 (b, d, f, h, j)) suggests that the reaction followed pseudo-first-order kinetics. The reaction rate was measured from the plot between $\ln(A_t/A_0)$ and time (t) for all the six sets of the experiment. Rate constant values of gold nanoparticles of different size have been summarised in Table 1. Rate constant

values revealed that the catalytic activities of gold nanoparticles are enhanced when synthesized with higher concentrations of leaf extract. The rate constant (k) was obtained from the graph using the linear equation:

$$-kt = \ln\left(\frac{C_t}{C_0}\right) = \ln\left(\frac{A_t}{A_0}\right) = \ln A$$

Here the NPs concentration at time 0 and time t was expressed as C_0 and C_t , respectively. A_t and A_0 are substituted for C_t and C_0 , respectively. A_t and A_0 are the absorbance of Nitrophenol at 390 nm at time t and time 0, respectively in UV-vis spectral graph.

Stability of synthesized GNPs

Stability of GNPs was evaluated by observing the SPR absorbance peak at different intervals of time. Fig 5 shows the UV-vis spectra of GNPs (600 μl of LE) at the time of synthesis, after passage of one year and after two years. No significant change of SPR peak has been observed corresponding to wavelength revealing that the size of the nanoparticles did not change with time and no aggregation took place. A little decrease in absorbance can be observed indicating that the concentration of nanoparticles decreased little bit but very slowly. Thus synthesized gold nanoparticles were found highly stable.

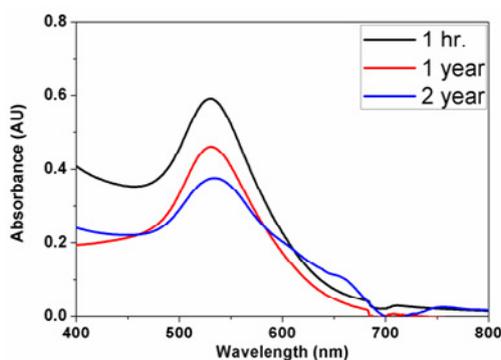


Fig. 5. Uv-vis spectra showing the stability of GNPs synthesized using 600 μl of *Eupatorium Odoratum* leaf extract.

Table 1. SPR absorbance peaks of different sets of GNPs (synthesized for different LE concentration), their corresponding hydrodynamic sizes as measured from DLS study and values of their reaction rate constant to evaluate the catalytic activity.

LE concentration(μl)	100 μl	200 μl	300 μl	400 μl	500 μl	600 μl
SPR peak (nm)	542 nm	533 nm	531 nm	527 nm	526 nm	524 nm
Average Size (nm)	42.64 nm	38.85 nm	30 nm	28.86 nm	26.45 nm	24.30 nm
Rate constant $K(\text{Sec}^{-1})$	----	2.67×10^{-4}	6.17×10^{-4}	6.37×10^{-4}	8.17×10^{-4}	8.7×10^{-4}



CONCLUSIONS

In summary, we reported a low cost one pot and controlled green synthesis of GNPs using *Eupatorium odoratum* leaf extract assisted with microwave irradiation. The characterization of the prepared nanoparticles by Uv-Vis spectroscopy, DLS, TEM, FTIR confirmed the formation of well small size spherical stable gold nanoparticles and presence of bio-molecules responsible for the formation of the nanoparticles. Moreover, the gold nanoparticles remained stable for long days. The size of the gold nanoparticles can be tuned by varying the concentration of leaf extract solution. The method is very rapid, it takes only a minute for the synthesis of nanoparticles and a very small amount of leaf extract is sufficient for the synthesis. The prepared GNPs were tested to check the catalytic behaviour on 2, 4, 6-tri Nitrophenol and it showed positive results in catalyzing the reaction of reduction of Nitrophenol to Aminophenol in presence of sodium borohydride.

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CONFLICT OF INTEREST

The authors declare that they have no competing interests.

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