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Green synthesis of Gold Nanoparticles from flower extract of *Tagetes erecta* at room temperature and physiological pH and their Nanoscopic Characterization by Spectroscopy and Microscopy

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ABSTRACT

The synthesis of nontoxic and eco-friendly metallic nanoparticle is a promising area of research due to its potential applications in the field of biomedical and therapeutics. Here, we report an easy and rapid aqueous synthesis of gold (Au) nanoparticle (NPs) from flower extract of Tagetes erecta at room temperature and physiological pH. The flower extract was act as a reducing and stabilizing agent of the NPs. The synthesis of nanoparticle was characterized by UV-Vis spectroscopy, Transmission electron microscopy (TEM), and X-ray diffraction (XRD) analysis. Gold NPs had a surface plasmon resonance peak (SPR) at 525 nm. TEM micrograph revealed that the NPs were well dispersed and spherical in shape. The average particle size was 6.5 ± 0.5 nm. Fourier transform infrared spectrum (FTIR) of the nano composite (Au NPs-extract) revealed that phenolic and other aromatic compounds of the flower extract were responsible for reductive synthesis of NPs. Use of non toxic extract, environmental friendly solvent and physiological condition are making it a suitable alternative over the convention methods for the synthesis of Au NPs.

Key words: *Tagetes flower extract, synthesis, Gold nanoparticles, Room temp, Physiological pH*
Abbreviations: *Au-Gold, NPs-Nanoparticles, TEM-Transmission electron microscopy, XRD-X-ray diffraction, SPR-Surface plasmon resonance, FTIR-Fourier transform infrared spectrum*

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1. INTRODUCTION

In the last few decades, synthesis of desired silver (Ag) and gold (Au) nanoparticles of different size and shape have been gaining intense interests because of their various applications such as imaging, catalysis, information storage, biodiagnostics and many more. There are several chemical and physical methods for the synthesis of the metal nanoparticles. However, these are multistep procedures and required sophisticated instruments and toxic reagents with potential environmental and biological risks. Therefore, it is imperative to develop alternative methods for generation of large scale biodegradable and eco-friendly nanoparticles. Recently, several reports came out on the synthesis of metal nanoparticles from biological molecules such as DNA, Protein, carbohydrates as well as whole cells such as from *Coriandrum sativum* leaf extract [1], Plant Extract of Papaver somniferum [2], aqueous extract of *Momordica cochinchinensis* rhizome [3], leaf extract of *Nepenthes khasiana* [4], fungi [5], bacteria [6] etc. These approaches provided facile and convenient way for generation of benign nano material by reducing the production of hazardous substances. But, the methods involved expensive reagents, significant time and multiple steps for synthesis of NPs. In addition, the extreme temperature and pH require for synthesis might cause denaturation or loss of activity of the biomacromolecules. For example, the Au NPs were successfully synthesized from the green fluorescence protein (GFP) but the physicochemical conditions maintained during synthesis caused the denaturation of protein which subsequently loss its fluorescence. To address these issues, the synthesis of nanoparticles from the plant extracts may be a possible solution. Because, the plant extract contains large amount of widely distributed metabolic fluxes (both primary as well as secondary) which can easily reduce and stabilize the NPs. In addition, this 'green synthesis method' provides several advantages over chemical and physical method due to its low cost, easy availability and scale up [7]. In addition, the plants used in the isolation of extracts do not require any special care during culturing and extraction for the synthesis of

NPs. Here we have reported the synthesis of Au NPs from the flower extract of *Tagetes erecta* commonly known as Marigold.

2. MATERIALS AND METHODS

Tagetes is an annual and perennial herbaceous plant. Its metabolites have several chemical and medical uses. It can be used as a food additive agent to add the natural color and flavor. It is also used to treat stomach ache, vomiting, indigestion as well as pesticides. Fresh flower (25g) of *Tagetis erecta* were washed, finely cut and soaked in 150ml boiling water for 15-20 min and filtered through cheese cloth followed by whatman filter paper no. 42. For preparation of gold nanoparticles, 20 ml of flower extract and 0.02ml of 0.3 M NaOH were added to 40 ml of ddH_2O followed by 1ml of 10^{-2} M $HAuCl_4$ (sigma) solution and kept at room temperature for 10 min. Then UV-Vis spectra were recorded by using Perkin-Elmer lambda 45 spectrophotometer (Lambda 45, Perkin-Elmer, Fremont, CA) for SPR analysis. The composite was analyzed using a high resolution Transmission electron microscope (TEM) (JEM 2100; Jeol, Peabody, MA, USA) operating at a maximum accelerating voltage of 200 KeV. For that, 7 μL of the synthesized composite was dropped on carbon coated copper grid, air dried and analyzed under TEM. Selected area electron diffraction (SAED) and energy-dispersive X-ray (EDX) spectroscopy (EDS) were performed using the same instrument and on the same samples. For X-ray diffraction (XRD) measurements the sample was evaporated on microscope glass slides before recording the diffraction pattern using a Bruker Advance D8 XRD machine (Cu α source with 1.5406 Å wavelengths). The FTIR spectrum of lyophilized composite and plant extract were recorded on a Perkin Elmer-Spectrum one, FTIR spectrometer, in the range 4000–400 cm^{-1}after mixing with KBr to make palette. In order to check the toxicity, the synthesized gold nanoparticles were added in to plant growth medium i.e. MS [8] basal medium having no plant growth regulators and the six months old *in vitro* shoots of *Nicotiana tobaccum* were cultured in the medium and kept in the plant tissue culture room to observe its growth and development with response to NPs.

3. RESULTS

The synthesis of Au NPs was carried out by incubating $HAuCl_4$ solution at ambient temperature (25 °C) in presence of flower extract of *Tagetes erecta.* After 10 mins of constant starring, the color of the solution was turned from black to red which indicated the formation of gold nanoparticles. The UV-Vis spectra of the composite exhibited absorption maxima at 525 nm in physiological pH (pH=7) but when the pH increased to 8 the peak became flattened and at pH 9 no peak was observed, which was due to SPR band of the gold nanoparticles (Figure 1A). It was found that temperature and pH have significant effect on synthesis of Au-NPs, the increase in temperature resulted in coagulation of the particles at Physiological pH. But at low pH (pH<7) and at low temperature (<27 °C) particle synthesis gradually decreased along with the time period required for synthesis was also increased. The NPs were remained stable for more than a week as measured by UV-Vis spectroscopy. TEM analysis was performed to find out the size and shape of the NPs. The TEM images indicated that most of the NPs were spherical in shape and monodispersed in nature (Figure 1B). The average particle size was 6.5 ± 0.5 nm (Figure 1C). In addition, the high resolution transmission electron microscope (HRTEM) images showed the individual lattice planes of the NPs. The characteristics Scherrerring patterns in SAED indicated that the NPs were polycrystalline (Figure 1D). Elemental analysis of TEM image showed that the NPs were exclusively formed with metallic gold. It should be mentioned here that the additional cupper and carbon signal was due to the carbon coated grid.

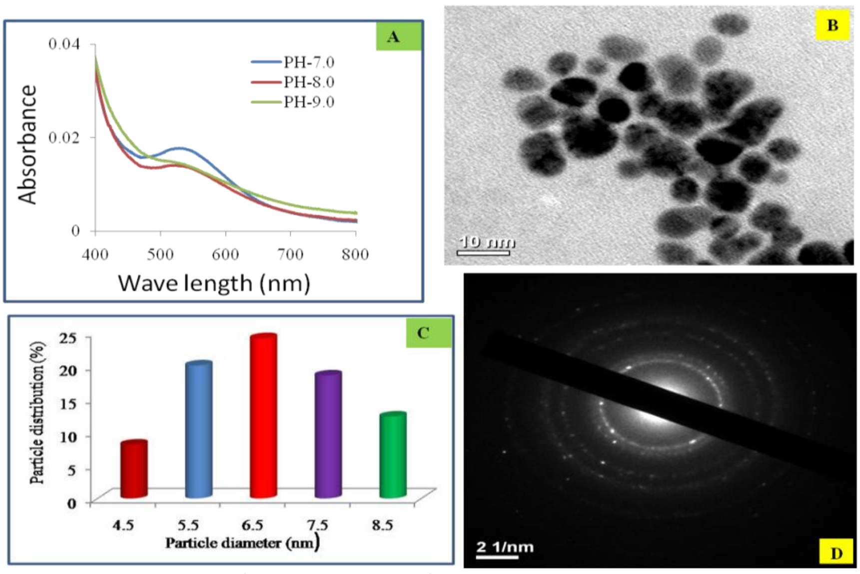

Fig. 1 UV-vis absorption spectra recorded from Marry gold flower extract reduced gold nanoparticles at different pH (A) Representative HTEM micrograph of *Tagetes* flower extract – reduced gold nanoparticles (B), Distribution of different sizes of gold nanoparticles (C), SAED image of *Tagetes* flower extract reduced gold nanoparticles (D)

The powder XRD of the lyophilized composite (Au NPs-extract) assigned the peaks for 2θ at 26.125°, 38.150° and 44.364 °C corresponds to the lattice planes at (111), (200) and (220) (Figure 2A). These are the characteristics Bragg's reflection planes of fcc structure of the gold nanoparticles (Au^0). The FTIR analysis was carried out to identify the functional groups which were involved in the reduction of gold ions and stabilization of the biogenic NPs. The FTIR peak were observed at 3444 cm^{-1}, 2924 cm^{-1} and 1628 cm^{-1} after synthesis of NPs which were originally assigned at 3389 cm^{-1}, 2928 cm^{-1} and 1615 cm^{-1} respectively in case of plant extract (Figure-2B). While studying the toxicity of the gold nanoparticles, it was observed that Au-NPs added in the MS basal medium without any growth regulators has no noticeable inhibition or retardation in the growth and development of in vitro grown shoots after one month of culture (figure-2C).

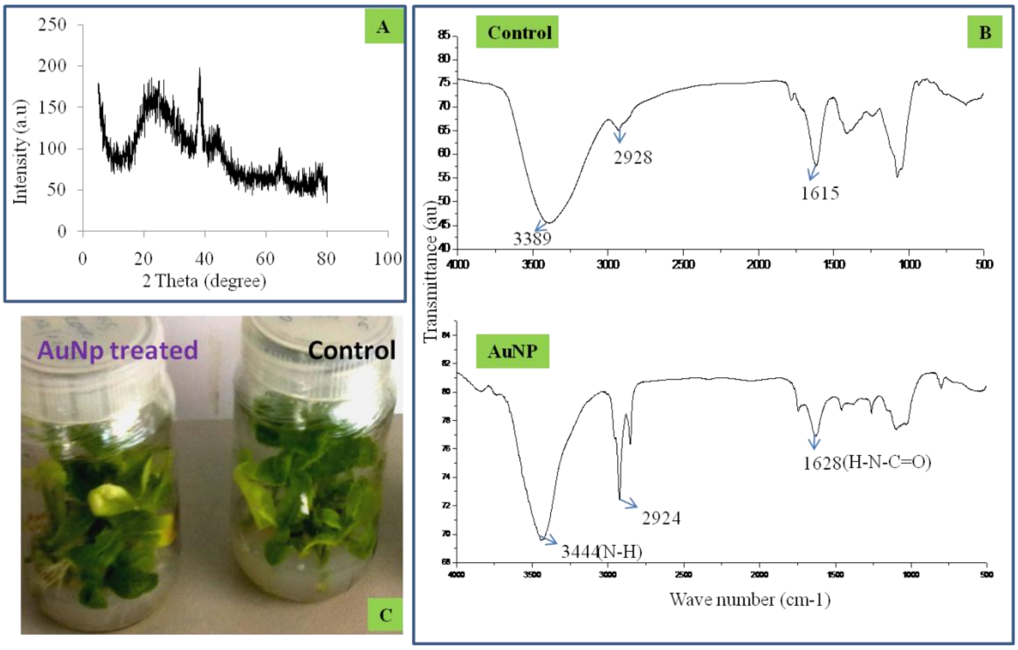

Fig. 2 XRD spectrum of gold nanoparticles synthesized by *Tagetes erecta* flower extract (A), FTIR spectrum of *Tagetes* flower extract and flower extract reduced gold nanoparticles (B), Growth of both treated and control shoots of *Nicotiana tobaccum* after 4 weeks of culture (C)

4. DISCUSSION

The SPR band observed in this report is 525nm which indicated the synthesis of AU-NPs. Similar reports were also reported by different peoples during the synthesis of Au-NPS [1, 9-11]. The NPs synthesized are about 6.5 ± 0.5 nm, comparatively smaller than those particles synthesized earlier [12-14]. The NPs were remained stable for more than a week. The particles were stabilized in solution by a capping agent, it may be due to the compounds like Lutein, Thiophene etc. present in the flower extract. In the FTIR analysis no significant difference between the two spectra of the plant extract before and after synthesis of NPs was observed, which indicated that the chemical constituents of the plant extract was remained unaltered after synthesis of the NPs. The only differences were at 3444 cm^{-1}, 2924 cm^{-1} and 1628 cm^{-1} after synthesis of NPs which were originally assigned at 3389 cm^{-1}, 2928 cm^{-1} and 1615 cm^{-1} respectively in case of plant extract. The peak at 3389 cm^{-1} corresponds to the stretching vibration of the free –OH was shifted at 3444 cm^{-1} suggested that the polyphenolic groups of the plant extract, which was extensively present in the plant extract was participated in the stabilization of NPs. In addition, the peck at 2928 cm^{-1} assigned for –NH stretching vibration was shifted at 2924 cm^{-1} due to the interaction with the Au NPs. It was reported that the lone pair electrons available on the amide group can stabilize the NPs and cause the shifting of its absorption band. Our FTIR data further supported this proposal. Shifting of peak from 1615 cm^{-1} to 1628 cm^{-1} corresponds to stretching vibration of C-N and C=O in the presence of gold nanoparticles suggested that the aromatic compounds like thiophene analogs of the plant extract might be involved in the synthesis and stabilization of NPs. The nontoxic and eco-friendly nature of the synthesized Au-NPs was demonstrated as the growth of the *in vitro* raised shoots was not retarded or inhibited by the addition of Au-NPs.

5. CONCLUSION

In conclusion, we have proposed an eco-friendly method for high yield synthesis of Au NPs using flower extract of *Tagetes erecta* performed at room temperature (at 25 ^{0}C) and physiological pH. The reduction of Au^{3+} to Au^0 and its stabilization believed to occur by the various acids, hydrolysates and aromatic compounds. No significant retardation of plant growth was observed when the nano composite (Au NPs- extract) was employed in the plant growth media. The low temperature, high yield and eco-friendly synthesis of Au NPs demonstrates the advantage of this method over the conventional methods. This green chemistry approach may find potential use in biomedical and pharmaceutical applications. The present investigation also leads to the development of a rational biosynthetic procedure for other metal nano materials.

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