BAJOPMAS JOURNAL 16(1): 160 – 168
Printed in Bayero University, Kano - Nigeria.

MODERN GREEN SYNTHESIS OF GOLD NANOPARTICLES VIA *Cactus Igneous Extract* FOR CATALYTIC REDUCTION OF ORGANIC DYES

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ABSTRACT

A novel green approach for the synthesis and stabilization of gold nanoparticles (AuNPs) using water extract of cactus igneous leaves under ambient conditions is reported in this article. The instant formation of gold nanoparticles (AuNPs) was analyzed by visual observation and UV–visible spectrophotometer. Further the effect of pH on the formation of AuNPs was also studied. The synthesized AuNPs were characterized by X-ray diffraction (XRD),energy-dispersive X-ray (EDX) dynamic light scattering (DLS) with zeta potential. Appearance of purple colour confirmed the formation of AuNPs. In the neutral pH, the stability of AuNPs was found to be high. The stability of AuNPs is due to the high negative values of zeta potential and capping of phytoconstituents present in the cactus igneous extract which is evident from zeta potential studies. The XRD and EDX pattern of synthesised AuNPs showed their crystalline structure, with face centered cubic geometry orientated in [1, 1, 1,] plane. DLS studies revealed that the diameter of stable AuNPs was approximately 25nm. Moreover the catalytic activity of synthesized AuNPs in the reduction of methylene blue was studied by UV–visible spectrophotometer. The synthesized AuNPs are observed to have a good catalytic activity on the reduction of methylene blue by Cactus igneous, which is confirmed by the decrease in absorbance maximum values of methylene blue with respect to time using a UV–visible spectrophotometer and is attributed to the electron relay effect.

Keywords: cactus igneous , catalytic activities, green synthesis, gold nanoparticles, X-RD

1.0 INTRODUCTION

Green synthesis of nanoparticles has emerged as a sustainable and environmentally responsible alternative for producing nanomaterials with diverse physical, chemical, and biological properties within the range of 1-100 nm. [1], [2], [3], [4], [5] [6]. It is simpler and safer to use than chemical and physical methods, which take a lot of time and have complicated steps. Conventional physical and chemical synthesis methods are often associated with limitations such as the generation of toxic by-products, high operational costs, and the use of hazardous reducing and stabilizing agents. Moreover, these approaches frequently require sophisticated equipment and energy-intensive conditions, which hinder their large-scale and eco-friendly application. As a result, there is an increasing need for cleaner, environmentally safer, and cost-effective routes to replace harmfull component ina variety applications [7].

In the recent past, there has been an increasing demand for nanoparticles due to their applications in various areas like medicine, catalysis, energy and materials [8]. The size, shape, and surface morphology of nanoparticles play a key role in controlling the physical, chemical, optical, and electronic properties of these nanomaterials. Metallic nanoparticles are synthesized by various methods such as physical vapour deposition, chemical vapour deposition, the sol–gel method, laser pyrolysis, pyrolysis, microwave-assisted synthesis, ultrasonication, electrochemical synthesis and chemical reduction of metallic ions [9][10]. The chemicals used for these syntheses are often toxic, costly and non-ecofriendly. Nowadays bioreduction methods based on fungi, microorganisms and plant extracts are being attempted due to the ease of synthesis, environmentally benign nature and greater stability of nanoparticles [10]. The applications of AuNPs in the fields of medicine, optoelectronics, optics, catalysis, and sensors are well known. Nanoparticle synthesis (especially gold nanoparticles) is usually carried out by various physical and chemical methods such as chemical vapour deposition, sol–gel technique, aerosol technology, sonochemical method , photochemical reduction and so on. The chemicals used for these syntheses are often toxic, costly and non-ecofriendly. However, these procedures are not regarded as ecologically friendly, limiting use in the food and medical industries; therefore, the green synthesis approach for producing AuNPs is an alternative source of conventional methods and possesses excellent anti-fungal activity. Recently, the synthesis of AuNPs have been reported using extraction of plants such as Penicillium rubens[11], Echinophora platyloba[12] ,

Costus igneus Nak is commonly called the spiral flag (*Costaceae* family). It possesses the potential to increase insulin by strengthening b-cells of the pancreas in the human body and is universally recognized as the *insulin* plant in India. As stated by conventional medical professionals, with the diagnosis of complex diseases that include diabetes, the administration of herbal drugs might be far more valuable in comparison to other therapies based on individual medicinal plant utilization [13][14]. Use of traditional medicine has been used from a very long time has a long history. It is the collective knowledge, skill, and practices based on the theories, principles, and experiences indigenous to various cultures, whether explicable or not, used in the maintenance of health as well as in the prevention, diagnosis, improvement, or treatment of physical and mental sickness. The collective influence of herbal therapy formulations leads to an improved restorative result through enhancing their protective impacts while reducing their toxicity. The Costaceae family includes Costus igneus, as shown in Figure 1commonly known as the *Spiral Flag*. It is found in eastern Brazil (Bahia and Espirito Santo states) and South America. It is a newbie to India and is cultivated as an elegant plant in Kerala. It thrives in a tropical environment.

Until now there is no report on the synthesis of gold nanoparticles using *Cactus igneus* extract. In this study, we present a rapid, inexpensive, and one-step green synthesis approach using extracts from cactus-type plants to fabricate stable and multifunctional gold nanoparticles (AuNPs). The synthesized nanoparticles were characterized using various spectroscopic and microscopic techniques to confirm their morphology, crystallinity, and surface chemistry. Furthermore, the biogenic nanoparticles are anticipated to exhibit excellent stability and promising biofunctional properties, highlighting their potential applications in antibacterial and catalytic studies.

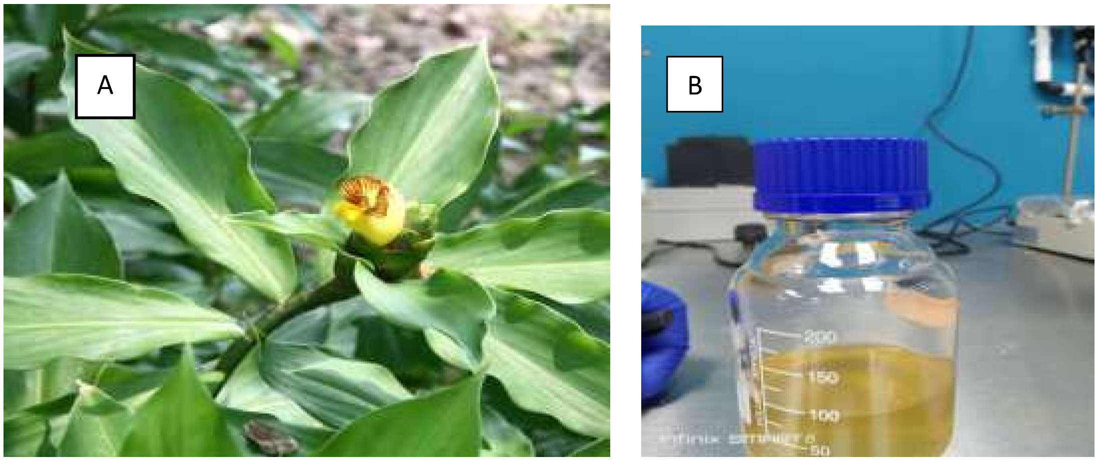

Plate (i). cactus igneous(A) plant and (B)cactus igneous plant Extarct

2.0 MATERIAL AND METHOD

2.1 Chemicals and Reagents

Cactus Igneous was obtained from Pilau Pinang, Malaysia, and used as received. Hydrogen tetrachloroauric acid (III) trihydrate ($HAuCl_4 \cdot 3H_2O$), silver nitrate ($AgNO_3$). Methylene blue(MB), 4 Nitro and sodium bought from Sigma Alderich Co (St Louis, MO U. S. A)

2.2 Preparation of Plant Extract

Cactus igneous leaves were collected from the forest area of Gelugor, Pilau Pinang, Malaysia, and were cleaned with double-distilled water and shade-dried for a week at room temperature and further dried at 25$^{\circ C}$ in the oven.The cleaned plant material was chopped into small pieces and boiled in distilled water (at 60–80$^{\circ C}$) for 15–30 minutes to extract bioactive compounds. The extract was filtered using Whatman filter paper to remove solid residues. The obtained filtrate was stored at 4°C for further studies[15], [16]

2.3 Bio-Fabrication of CI-AuNPs

The temperature, reaction time and concentration of CI extract and metal precursor for biosynthesis of AuNPs was optimized before this work. First min at 80^{0}C. After 15 min, the The change to violet colour corroborated that CI AuNPs had been synthesized.

2.4 Characterisation. *CI-AuNPs*

UV–visible spectra of NPs green-synthesized using CI were recorded using an Ultrospec 6300 pro UV-Vis spectrophotometer (Amersham Biosciences, Buckinghamshire, UK) in 300–800 nm at wavelengths. The particle size of the dispersions and zeta potential were analyzed by a Zetasizer, Nano-ZS90 (Malvern Panalytical, Malvern, UK). A TALOS F200X(Thermo Scientific, Eugene, OR, USA). The AuNPs were fixed onto a copper grid, Form var/Carbon 200 Mesh (Electron Microscopy Sciences, Hatfield, PA, USA). X-ray diffractometer, X'Pert3 Powder (XRD Empyrean series 2, PANalytical, Almelo, Nederland), was used to reveal the structural information of NPs. The instrument was operated at 40kV with 30 mA in current and Cu Kα radiation (1.540 Å) between 2θ^0 from 200 to 800 for analyzing the XRD pattern and crystal structure.

2.5 Catalytic Study of CI-AuNPs

The catalytic potential for reduction of dyes was studied using the UV-Visible spectrophotometer with ice-cold $NaBH_4$ at room temperature. $NaBH_4$ solution was freshly prepared prior to the experiments. Then, 2 mL aqueous solutions of MB (0.08 mM) and RB (0.05 mM) were added to 1 mL $NaBH_4$ (30 mM) and CI-AuNP (or CI-AgNP) solutions. Next, 2 mL of MB (0.1 mM) aqueous solution was mixed with 1 mL of $NaBH_4$ (30 mM), and AuNP or CI-AgNP solution was added to the mixture at different volumes (150, 10, and 20 μL) of MB, RB, and MO, respectively. Meanwhile, the volume of the CI-AuNP solution added to the mixtures was 10 μL, except for the reduction of MB, where it was 20 μL. The amount of green-synthesized AuNPs and the concentration of dyes and $NaBH_4$ for catalytic activities of CI-AuNPs were optimized before these processes.

3.0 RESULT AND DISCUSSION

3.1 Biofabrication of cactus igneous nanoparticles

Green synthesised Cactus ignous gold nanoparticles (CI-AuNPs) were characterized to investigate their potential (Figure 2.0). We mixed 1 mL aqueous extract of CI (2 mg/mL) 1 mL aqueous extract of CI (2 mg/mL) was added to 1μL $HAuCl_4 \cdot 3H_2O$ (1 M) solution for CI-AuNPs'formation. The color of the colloid became purple within 15 min in the water bath, suggesting CI-AuNPs had formed (Figure1). CI-AuNPs showed a strong LSPR band at 528 nm shown in the spectra. Because of their rich phytochemical makeup, cacti have been studied for their potential in the production of nanoparticles. Bioactive substances such as flavonoids, polyphenols, and alkaloids, which can act as stabilising and reducing agents during the creation of nanoparticles, are plentiful in the cactus igneous extract.

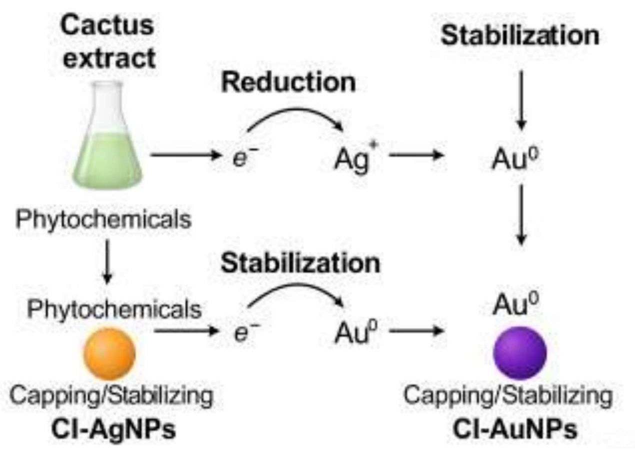

Figure 1. Schematic Meachanism of Formation Gold Nanoparticles

3.2 UV–visible analysis

The key approach for confirming the production of Cactus igneous AuNPs was UV–Vis analysis. The solution changed colour from light yellow to purple due to the synthesis of Cactus igneous AuNPs. 2 depicts the UV–vis absorption spectra as a function of time. The Cactus igneous AuNPs were produced in 120 min, and the NPs development was tracked and retrieved every 30 min. At nanometre dimensions, the electron cloud can oscillate on the particle surface and absorb electromagnetic radiation at a particular energy. This resonance, known as surface plasmon resonance (SPR) [17] or plasmon absorbance of nanoparticles, is a consequence of their small size, but it can be influenced by numerous factors; in particular, solvent and surface functionalization are important contributors to the exact frequency and intensity of the band. This dependence on surface effects makes the surface plasmon an ideal monitor of the absorption, which allows nanoparticle assembly to be used as a sensing device. [2], [18] The formation of cactus igneous-AuNPs is shown by surface plasmon resonance (SPR). This may occur if the electrons in the ground state of the nanoparticles are excited. It is common knowledge that the SPR peak depends only on the form of the size of the nanoparticle.

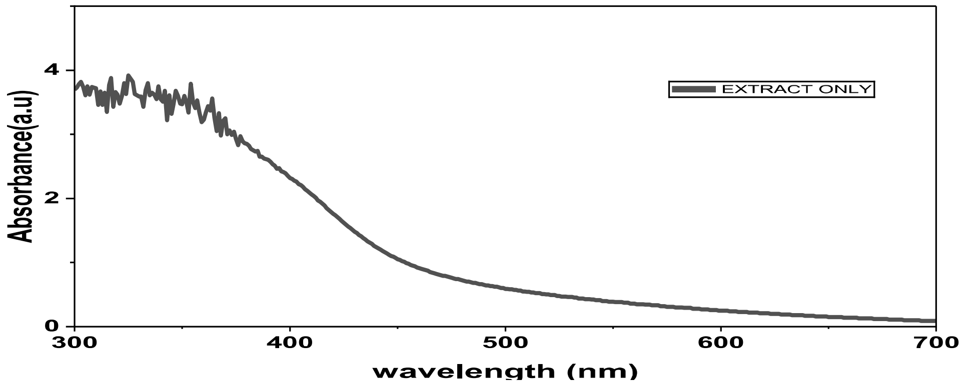

Fig. 2(a). The UV-Vis absorption spectra of cactus igneous plant extract

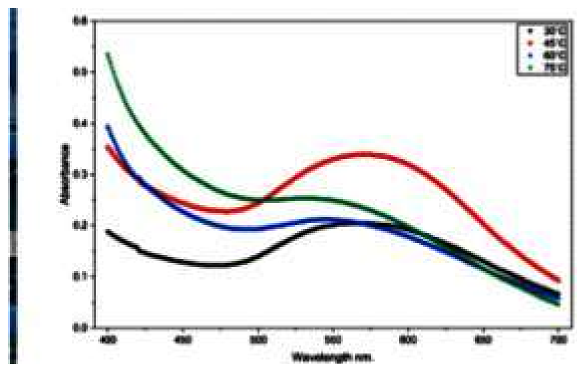

Fig. 2(b). The UV-Vis absorption spectra of synthesized gold nanoparticles

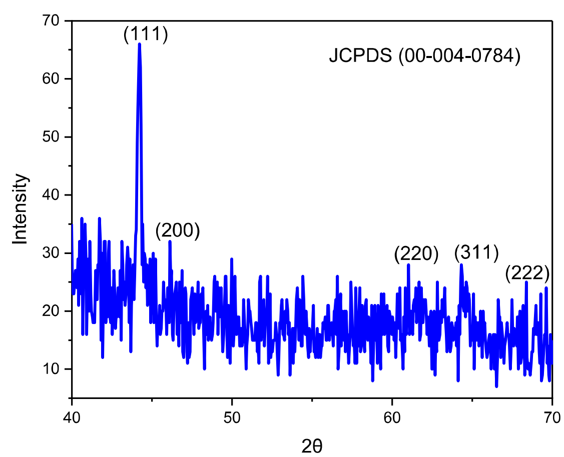

Fig. (2c) XRD spectrum of green synthesized gold nanoparticle

3.3 XRD

XRD is primarily used to identify crystallographic phases and characterize the crystalline nature at room temperature of CI-AuNPs. Figure 2b displayed the XRD pattern of AuNPs .The diffraction peaks were detected in the 2θ angles in the range of 20-800, which can be indexed (1, 1, 1), (2, 0, 0), (2, 2, 0) and (3, 1, 1) and (222). As a result, it confirms the synthesized AuNPs planes have face-centred-cubic (FCC) crystalline structure geometry of AuNPs, which is in agreement with the JCPDS file No. 00-004-0784 for gold. .[19] The average crystalline structure size of synthesized AuNPs was estimated by Scherrer's formula.

$$D = \frac{k\lambda}{\beta cos\theta} \quad (1)$$

Where D is the mean crystall size K is constant (0.94). λ is the wavelength, theta is the Bragg's angle, β is the full with half maximum of the diffraction peak.This result was cofirmed with calculted mean diameter of 19.26nm

3.3 Dynamic Light Scattering (DLS) and zeta potential

Figure 4.0 shows the DLS analysis to find out the hydrodynamic distribution of size for -AuNPs. The average size of CI-AuNPs was 17.47 ±0.13 nm at 25°C. The polydispersity index was 0.484. The zeta potential measurements that identify the stability of nanoparticles were −33.2 as shown in Figure. These large negative values of zeta potential indicated a good stability of the colloidal solutions of C-AuNP.

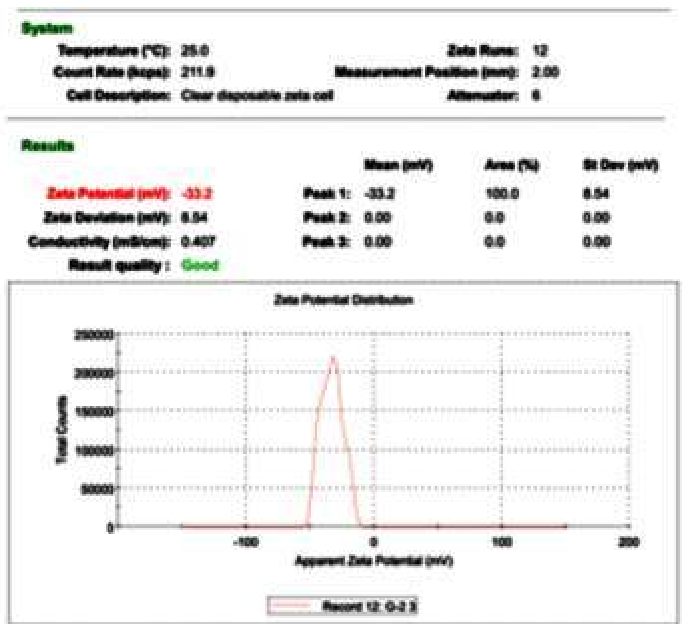

Figure 3.0. zeta potential of gold nanoparticle

3.4 SEM

Scanning electron microscopy (SEM) is used for morphological characterization at the nanometre to micrometre scale [8], [20]. SEM analysis shows uniformly distributed sgold nanoparticles, indicating the stabilization of nanoparticles by capping agents. The gold nanoparticles were spherical in shape with a particle size range from 19.62 to 31.3 nm. The larger gold particles may be due to the aggregation of the smaller ones, according to the SEM measurements Fig4(A). In EDAX strong signals were observed from the gold atoms in the nanoparticles and weaker signals for carbon, oxygen,and potassium were provenients from biomolecules of Cactus igneous Fig.4(C). Elemental gold can be seen in the graph

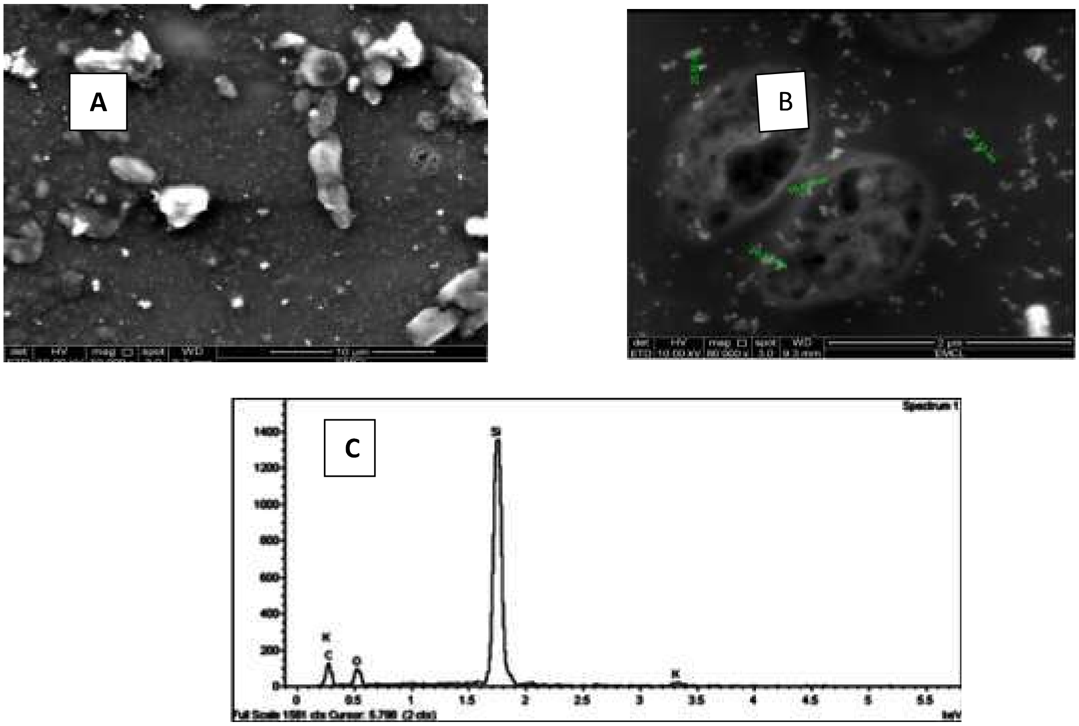

Figure 4.0 (A) & (B) SEM and (C) EDX images showing the presence of gold nanoparticles and the biogenic component of the cactus igneous plant

The strong signal of the Au atoms indicates the crystalline property. The presence of O peaks along with the Au signals suggests that the AuNPs are capped by phytoconstituents through oxygen atoms. The Cu peak comes from the grid. The size of the synthesized AuNPs is approximately 25 nm.

4.0 CONCLUSION

The study has demonstrated that AuNPs could be prepared instantly by making use of aqueous extract of the Cactus igneus plant extract. The phytoconstituents, such as hydrolysable tannins, gallic acid, chebulic acid, chebulic ellagitannins and gallate esters, act as reducing agents for the preparation of AuNPs, and the capping of AuNPs by the phytoconstituents provides stability to AuNPs, as evident from EDX studies. [21] The synthesized AuNPs were found to have a crystalline structure with face-centred cubic geometry, as studied by the XRD method. The DLS studies had shown that the synthesized AuNPs are having a size around 32 nm. The synthesized AuNPs act through the electron relay effect and influence the degradation of methylene blue by myrobalan extract.

5.0 ACKNOWLEDGEMENT

The authors gratefully thank the School of Physics, Universiti Sains Malaysia, for extending the XRD facility and also thank the Department of Physics, Bayero University, Kano, for providing good research guidance.

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