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# Synthesis and Characterization of Palladium Nanoparticles Using Leaf Extracts

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## ABSTRACT

Green synthesized metallic nanoparticles are an evolving environment-friendly technique in recent years. An environmental friendly, biological processing of palladium nanoparticles by employing *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate* leaf extracts were described in our current study. The prepared PdNPs were further underwent characterization via Ultra Violet-visible, Fourier transform infrared, Photo-luminescence and X-ray diffraction spectroscopy and Scanning and Transmission electron microscopy. Furthermore, a validation of PdNPs creation was established through UV–visible spectrophotometer and also a spherical shaped PdNPs within a size between 2-5 nm was perceived in TEM and 500 to 2000nm was achieved in SEM study. XRD study deep-rooted the crystallic nature of Pd NPs and then it was related with the standard. FTIR spectral studies were carried out to recognize the functional groups exist in the Pd NPs. The photoluminescence study confirms that these extracts were action as a stabilizing and capping agent. The analysis exposed that all the three extracts *Basella alba, Allium fistulosum* and *Tabernaemontana divaricate* may have efficient less-toxic reducing substance for the process of Pd NPs and the formulated Pd NPs may be used in the arena of biomedical applications. These Pd NPs mainly intricate in the biological reduction reaction rate for the processing of Pd NPs.

**Keywords:** *Basella alba, Allium fistulosum, Tabernaemontana divaricate*, Green synthesis, Palladium nanoparticles.

## INTRODUCTION

In recent years, Nanomaterial development mostly with necessary quality is one of the most fascinating things in nanoscience and technology. Due to its interesting physical, chemical and thermodynamic properties, metal techniques have gained dramatically increased attention, making them viable candidates for uses in different areas like catalysis [1, 2], optical electronics [3, 4] alongside its biomedical uses [5]. Owing to their fruitful applications in the realm of bioscience, biomedicine, and pharmacy, palladium nanoparticles also known as PdNPs catalysts has attracted considerable interest. Progress in the production of Pd nanoparticles has achieved tremendous importance due to its usage in both homogenous and heterogeneous catalysis, owing to its higher surface-to-volume ratio and huge exterior strength. Normal formulated PdNPs delivery methods comprises electrical and chemical [7, 8] and laser pulse ablation [9] as well as sono-chemical decline techniques [10]. Because the synthetic chemical techniques of producing Pd NPs create a punitive action and decrease the catalyst performance of palladium, novel synthetic procedures need to evolve to meet a wide range of potential uses for the development of PdNPs with regulated size and thickness.

Metal ions may be reduced into NPs by employing methods like physio-chemical, enzymatic, and biological. The techniques of physio-chemical use higher radiation and concentrated reduction substances that pollute the atmosphere and even damage people's health. Nevertheless, the enzymatic method to nanoparticles processes is better but more costly. The usage of biological processes has emerged in the last decade as an innovative and reliable techniques for the development of nanomaterials due to a necessity to establish nature-friendly techniques in the processing of nanomaterials [11]. The biosynthetic pathway of metal NPs, using microscopic organisms [12] and flora [13], has put much effort on. Plant contains the minimizing properties of the metal atom and thus absorb and stored in a synthesis of NPs. Discrepancies in the dimension and thickness of NPs formed by certain floras because of variations in propagation as well as position of metal-based ions reduce their properties and hinder their use because precision and highly developed structure are needed. These issue can be fixed by using leaf extract rather than whole plants to gain versatile influence over their output and purification [14]. The simplest procedure for dealing with processing of environmentally friendly nanoparticles is a one-step biological reduction process utilizing floral extracts. Owing to prosperous biological diversity and simple accessibility the floral-based nanomaterial processing have been widely investigated [15]. These have been established that floral crude aqueous extract innovative 1° and 2° metabolites which show a significant role in reducing ionic metal form into environment friendly nano-sized metallic nanoparticles [16]. Nanoparticles extracted from the leaf extracts were widely used in the clinical, nutrition, waste $H_2O$ therapy as well as beautifying products. Numerous studies have shown that a floral-based NPs has been employed successfully as an antimicrobials [17], larvicides [18], and cytotoxic agents [19]. A perception of these literary works, biosynthetic strategy using plant resources has elevated in recent times a clear and acceptable alternative to the physicochemical method implemented so far. Throughout this analysis, PdNPs by employing floral extract of numerous plants including *Basella alba* [20], *Allium fistulosm* [21] and *Tabernaemontana divaricate* [22] have been described in current time reports, but hitherto prospective of floral resources to be examined. *Basella alba* (family of Basellaceae) is a speedy developing palatable perennial plant, mounting up to 9 m in extent. Moreover, *Tabernaemontana divaricate* and *Allium fistulosm* is a flowering plants belongs to the family of Apocynaceae and Alliaceae, respectively. The extracts of all the three plants exhibited an antioxidant, antibacterial and anti-inflammatory characteristics [23].

This paper looks at the biogenic synthesis of PdNPs utilizing various leaf extracts namely, *Basella alba*, *Allium fistulosm* and *Tabernaemontana divaricate*. Furthermore, we have characterized the processed PdNPs via employing UV-vis, FTIR, XRD, TEM and Photoluminescence spectroscopy.

## MATERIALS AND METHODS

### Preparation of plant extract

*Basella alba*, *Allium fistulosm* and *Tabernaemontana divaricate* were collected from in and around Vellore district. The collected leaves were washed thoroughly to remove dust particles and fungal spores and shade dried to remove moisture. The solutions were equipped

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by balancing 10 g of *Basella alba* leaf. Further, the leaves were transferred into 250 ml glass holding 200 ml distilled $H_2O$ and then heated the mixture for 45 minutes on heating mantle. The extract obtained was cooled to room temperature and then filtered. Similarly, the plant extract of the other two plants viz. *Tabernaemontana divaricate* and *Allium fistulosum* were also prepared.

## SYNTHESIS OF PALLADIUM NANOPARTICLE

1 mM Palladium acetate solution was formulated and it was utilized for the process of Palladium nanoparticles. The aqueous solution of *Basella alba, Allium fistulosum* and *Tabernaemontana divaricate* were added separately into test tubes containing 2mM aqueous Palladium acetate solution in different proportions. The variation in color of the solution designated the creation of palladium NPs. To achieve the finest features for the processing of palladium NPs, this investigations were executed at a diverse circumstances with variable temperature and pH. The colour was transformed from mild brown to dark brown when the *Tabernaemontana divaricata* and *Allium fistulosum* leaf extract were added dropwise to the palladium acetate which indicates the formation of palladium nanoparticles. Likewise, in case of *Basella alba*, the color change was noticed from light brown to green which is a characteristic of palladium nanoparticles.

## CHARACTERISATION OF PALLADIUM NANOPARTICLES

### UV- Vis spectral analysis

The reduction of palladium ion to metallic palladium nanoparticles was spectroscopically identified by double beam UV-Visible spectrophotometer at different wavelength ranging between 200-1000nm. The optimum conditions of processing a palladium NPs from the extracts were observed to be 2 mM palladium acetate at a temperature of 60°C after carrying out the experiments at different concentrations of palladium acetate. The intensity of UV-Vis was gradually enhanced to constant as a function of the reaction time. The color changes from light to dark brown and mild brown to green was owing to an excitation of surface plasmon resonance in a PdNPs. These optic-based characteristics of PdNPs were examined by means of UV-Visible absorption spectrophotometer by employing a Varian Cary-50.

### Fourier transform infrared spectroscopy (FTIR)

A functional group of prepared PdNPs were determined using FTIR (Varian 800) and it was compared with aqueous extract of *Basella alba, Allium fistulosum* and *Tabernaemontana divaricate*. The synthesized samples were added to potassium bromide (KBr) in the proportion of 1:99 to attain the pellet and it was scanned in between 400 to 4000 $cm^{-1}$ within a resolution of 2 $cm^{-1}$.

### X-Ray Diffraction (XRD)

XRD technique was employed to determine the crystals dimension utilizing X' Pert Pro A Analytical X-ray 107 Diffractionwith Copper potassium (alpha) radiation lies between 10° to 80°.

### Scanning electron microscopy (SEM)

The PdNPs were categorized using Scanning electron microscopy methods. The crystal-like nature and its dimension was further validated by employing SEM configuration for PdNPs exhibited an indications for palladium metal material.

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**Transmission electron microscopy (TEM)**

A shape, dimension and structure of PdNPs were examined by Transmission Electron Microscope (TEM). A one droplet suspension of PdNPs were incorporated on to a carbon covered copper grid. The imageJ platform was utilized to identify the dimension and shape of PdNPs from the obtained TEM pictures.

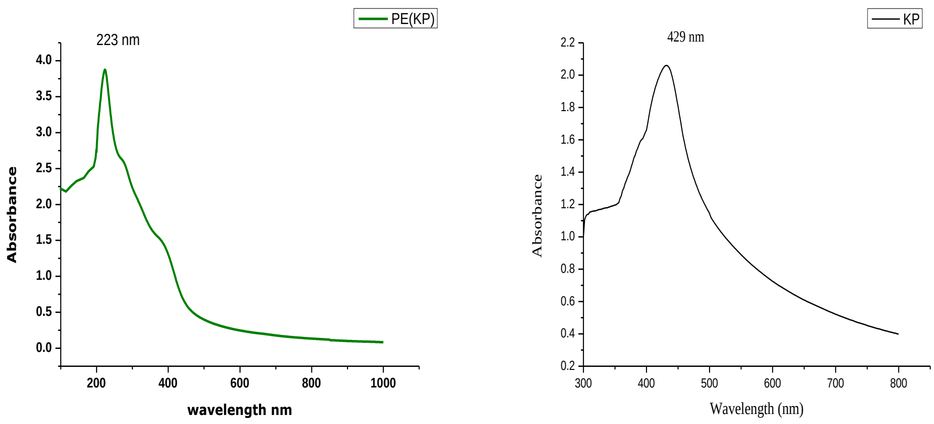
**Photoluminescence spectroscopy (PL)**

PL is a contactless, non-destructive technique of probing the electronic assembly of nanomaterials. This spectral analysis were documented to estimate the photo-luminescence action owing to an organic molecules from the floral solution which steadies the palladium based nanocatalyst [24].

## Results and Discussion

### UV spectral analysis

UV-visible spectral results provides a 1° information regarding the creation of nanoparticles. A three group of plant extracts, namely, *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate* combined with the palladium nanoparticles were perused underneath an UV-visible spectroscopy. Figure 1(a) shows the plant extract of *Basella alba* whereas Figure 1(b) shows the plant extract of *Basella alba* with palladium nanoparticle. It was observed from the Figure 1(a) and (b), that the plant extract of *Basella alba* and palladium nanoparticle ensues at a maximum absorption peak of 223 and 429 nm, respectively. The clear hump indicates formation of palladium nanoparticles. Similarly, Figure 2 and 3 shows the comparative study of the extracts of *Allium fistulosum* and *Tabernaemontana divaricate* along with palladium nanoparticle. The absorption peak intensity of *Allium fistulosum* and *Tabernaemontana divaricate* extract was found to be 225 and 215 nm, whilst the peak intensity of their synthesized PdNPs were found as 275 and 277 nm respectively. The color change occurred owing to the existence of surface plasmon resonance which is an inherent feature of metal-based NPs.




(a) (b)

**Figure 1. UV-visible spectral study of (a) plant extract of *Basella alba*, (b) plant extract with PdNPs.**

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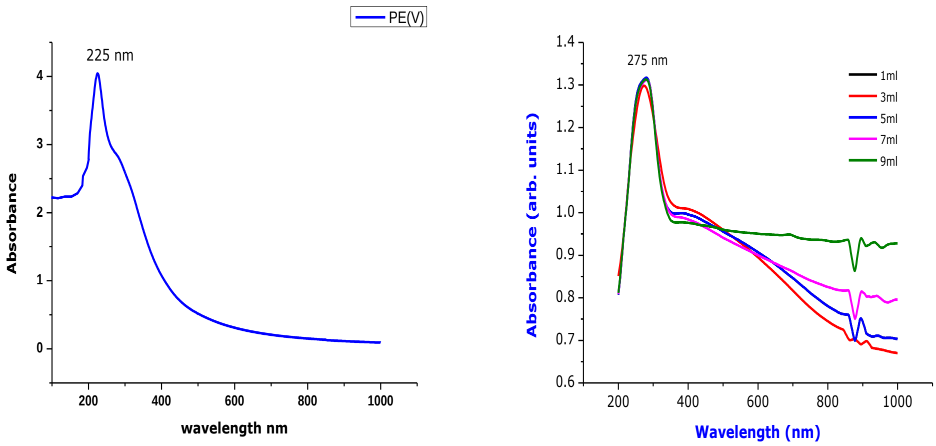
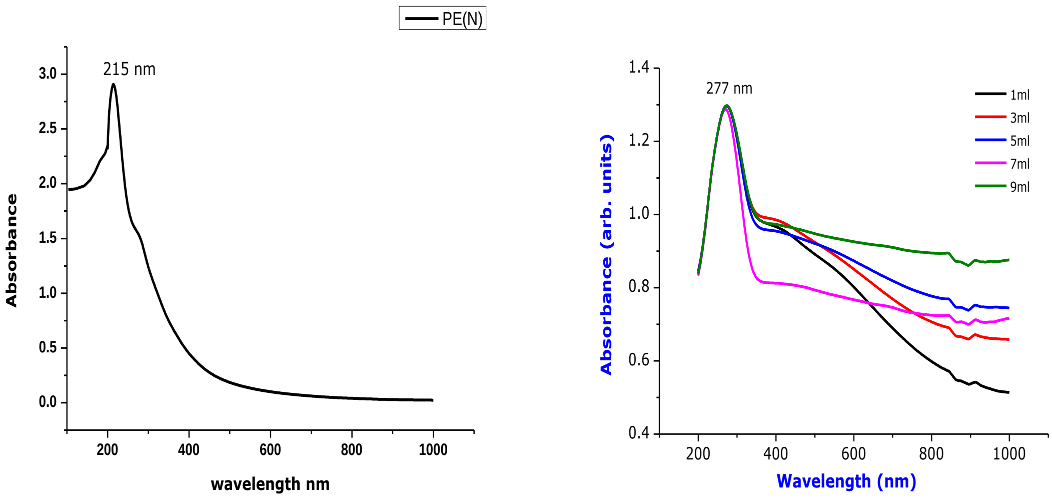



**(a)** **(b)**

**Figure 2. UV-visible spectral study of (a) plant extract of *Allium fistulosum*, (b) plant extract with PdNPs.**




**(a)** **(b)**

**Figure 3. UV-visible spectral study of (a) plant extract of *Tabernaemontana divaricate*, (b) plant extract with PdNPs.**

There is a significant increase of peak in the PdNPs when compared to that of crude plant extracts. Overall, the UV-spectral analysis of the PdNPs describes, increase in the absorption peak represents the greater the formation of palladium nanoparticles.

## FTIR analysis

FTIR analysis was performed to examine both the NPs and floral extracts exhibited absorption peaks: while PdNPs allocated as symmetry and anti-symmetry frequency of broadening. The results for all the three extracts of palladium nanoparticles were explained in Figure 4. The Figure 4(a), a spectrum of *Basella alba* synthesized particle showed a total of 3 peaks in the area of 3500-1500 cm$^{-1}$ and a greater band at 1638 cm$^{-1}$ where a lesser peak at 3516 and 2147 cm$^{-1}$, respectively.

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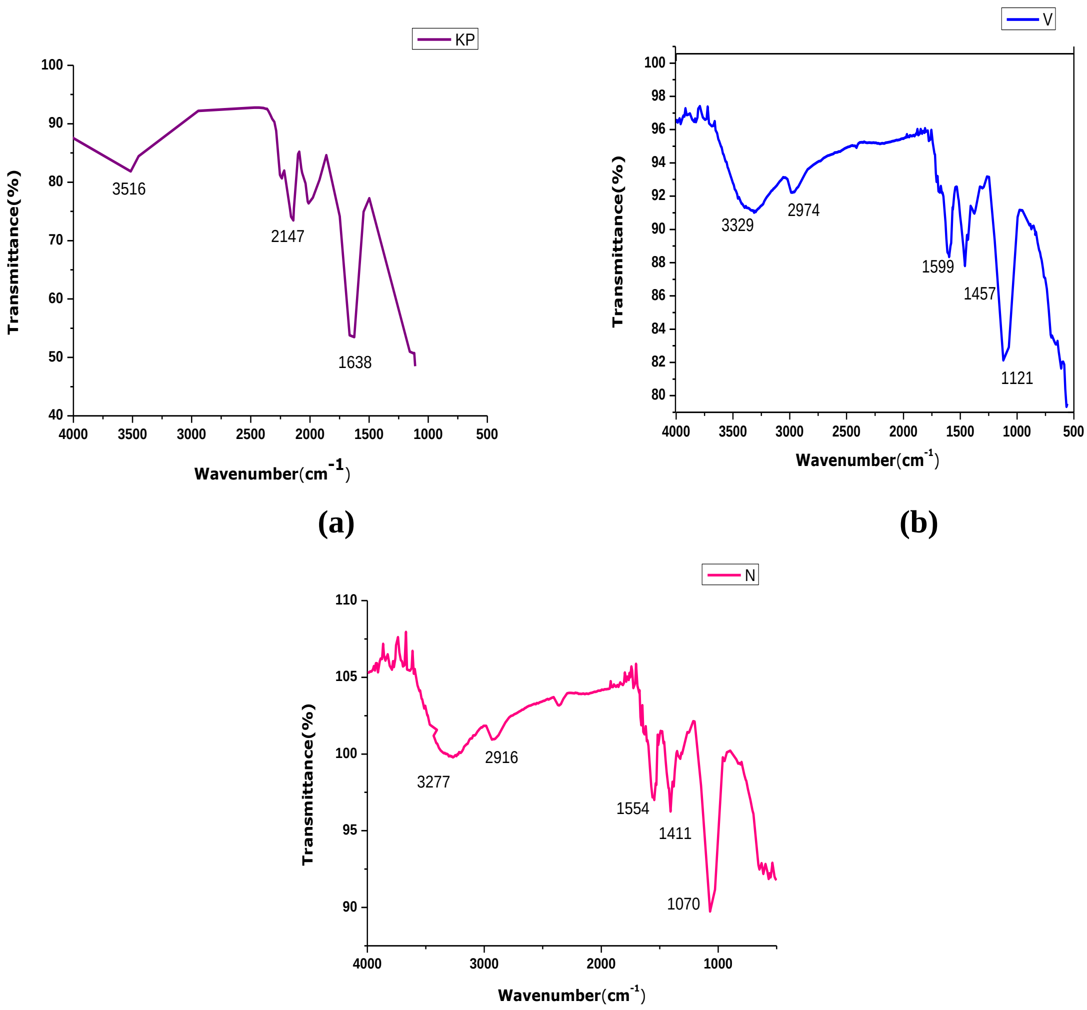


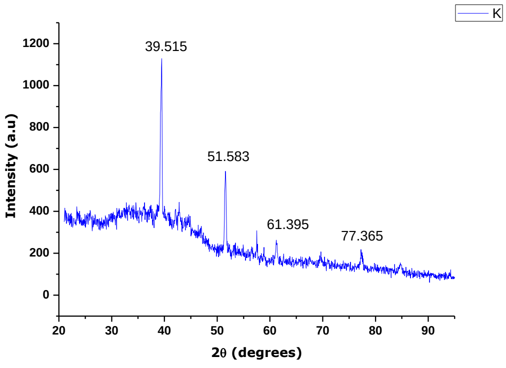

(c)

**Figure 4. FTIR spectral study of biosynthesized palladium nanoparticles of (a) *Basella alba*, (b) *Allium fistulosum* and (c) *Tabernaemontana divaricate* extract.**

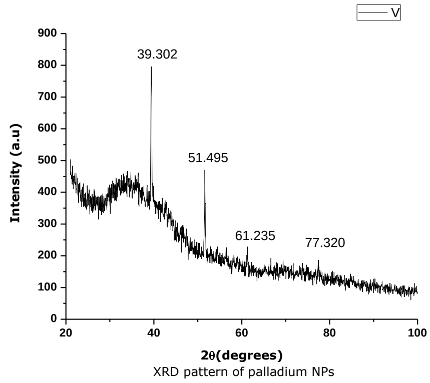
Similarly, in case of *Allium fistulosum* synthesized particle exhibited numerous bands within a wide peak in an arena of 1000-1500 cm$^{-1}$ and the strongest absorption band at 1121 cm$^{-1}$ along with a should band at 1457 cm$^{-1}$, respectively (Figure 4(b)), whereas for the and *Tabernaemontana divaricate* biosynthetic particle also showed several peaks with a broad peak in an arena of 1000-1500 cm$^{-1}$ and a strongest band at 1070 cm$^{-1}$ along with a should band at 1411 cm$^{-1}$, respectively (Figure 4(c)).

**X-Ray Diffraction analysis**

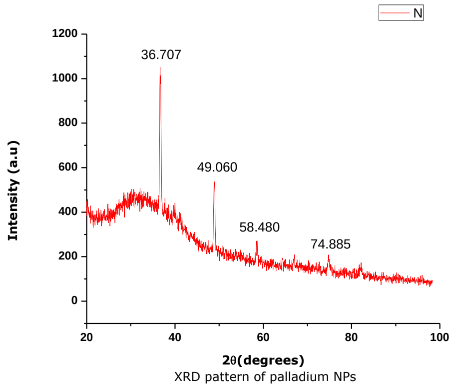
A XRD spectral analysis of the biologically synthesized palladium nanoparticles exhibits both the crystallinity and its stages were shown in Figure 5 (a), (b) and (c).




(a)




(b)




(c)

**Figure 5. XRD diffraction pattern of synthesized palladium nanoparticles of (a) *Basella alba*, (b) *Allium fistulosum* and (c) *Tabernaemontana divaricate* extract.**

From the extract of *Basella alba* PdNPs (Figure 5 (a)), the spectrum possesses four peaks at 2θ values of 40°, 50.2°, 60.3° and 70.8 that confirms the presence of 39.515, 51.583, 61.395 and 77.365 group of lattical planes of Bragg's reflection, respectively. Likewise, an arrangement of PdNPs synthesized by employing *Allium fistulosum* (2θ values of 40°, 50.1°, 60.1° and 70.8° that confirms the presence of 39.302, 51.495, 61.235 and 77.320 group) and *Tabernaemontana divaricate* (2θ values of 30.8°, 50°, 59.9° and 78.7° that confirms the presence of 36.707, 49.060, 58.480 and 74.885 group) extract was also observed as shown in Figure 5 (b) and (c).

**SEM analysis**

The Figure 6 (a), (b) and (c) clearly exemplifies the SEM pictures of palladium nanoparticles synthesized using *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate*.

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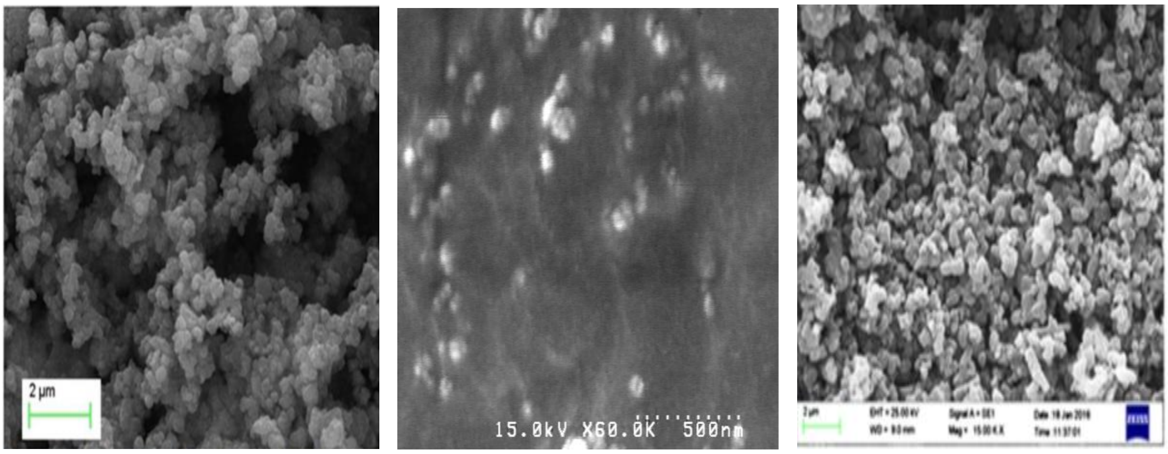


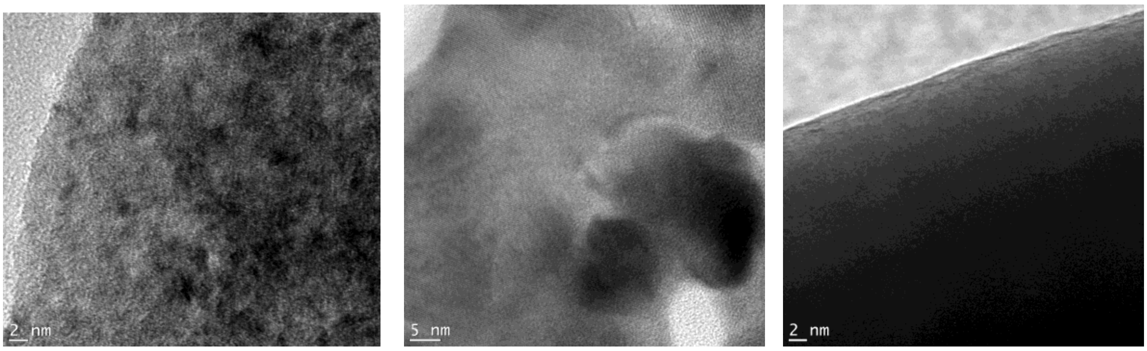

**(a) (b) (c)**

**Figure 6. SEM image of palladium nanoparticles using (a) *Basella alba*, (b) *Allium fistulosum* and (c) *Tabernaemontana divaricate* extract.**

The results from our analysis represented spherical morphology with a dimension of 2 µm, 500 nm and 2 µm, respectively for the extracts of *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate* synthesized palladium nanoparticles. The palladium nanoparticle which is synthesized from the extract of *Allium fistulosum* was found to be better when compared to other two extracts of processes nanoparticles.

**TEM analysis**

TEM images of all the three extracts designate a synthesized PdNPs were moderately unvarying in diameter as well as its figure. PdNPs were exhibited as a range of 2 to 5 nm (Figure 6).




**(a) (b) (c)**

**Figure 7. TEM image of palladium nanoparticles using (a) *Basella alba*, (b) *Allium fistulosum* and (c) *Tabernaemontana divaricate* extract.**

TEM studies confirmed that the *Tabernaemontana divaricata* extract was the suitable and effective material for the formation of highly stabilized PdNPs. The particle size, morphology and crystallinity were studied using TEM.

**Photoluminescence spectroscopy analysis**

The photoluminescence spectral analysis of synthesized palladium nanoparticles were exemplified in Figure 7(a), (b) and (c) for the plant extract of *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate*, respectively. The PL study showed that the excitation

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wavelength peaks were at 481 nm (*Basella alba*), 492 nm (*Allium fistulosum*) and 481 nm (*Tabernaemontana divaricate*), respectively.

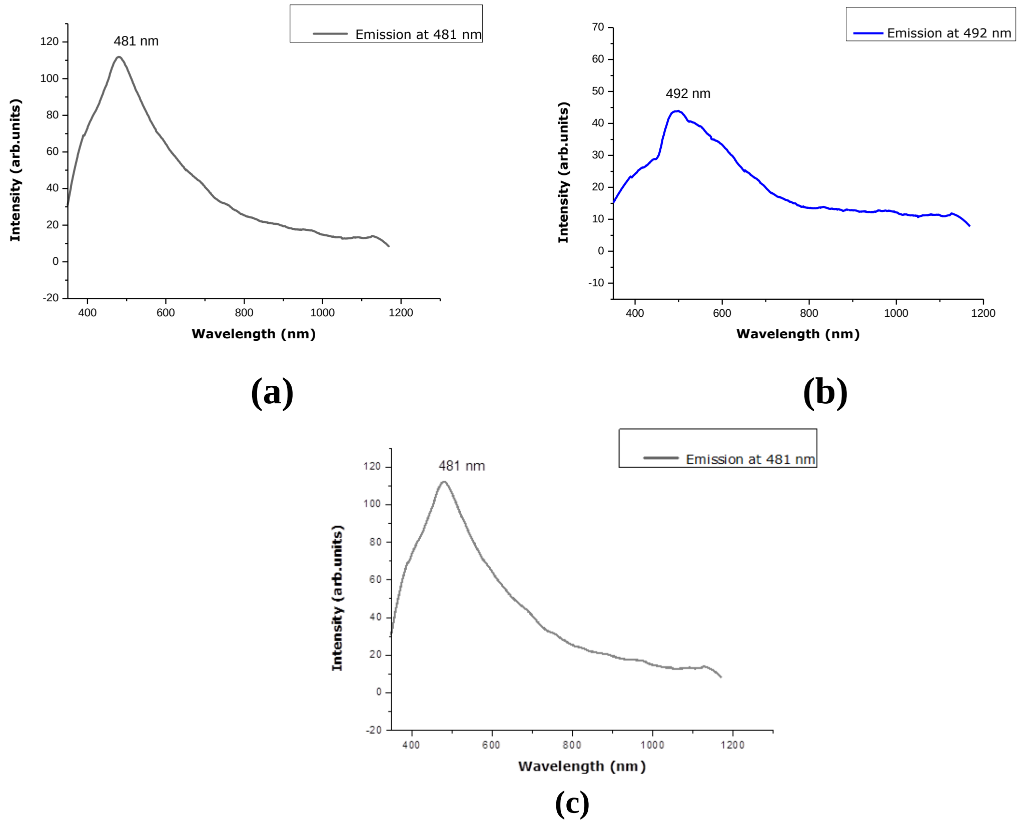


**Figure 8. Photoluminescence spectroscopy of palladium nanoparticles of (a) *Basella alba*, (b) *Allium fistulosum* and (c) *Tabernaemontana divaricate* extract.**

The better photoluminescence of palladium NPs can be measured depends on the behavior of an electron from that of organic compounds which is attained from the plant extracts. Moreover, after the biosynthetic process, these extracts were acts as a stabilizing and capping material.

## Conclusion

A green chemistry based strategy have been recognized for palladium nano-based particle synthesis by employing *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate* leaf extracts. The development of Pd particles were deep-rooted by a color modification and Ultra Violet-Visible spectral analysis. The spherical figure of Pd particles within an extent dissemination of 500 nm to 2000 (that is 2 µm) nm was attained in SEM studies and 2 to 5 nm was perceived in TEM studies. XRD pattern discovered the crystal-like nature of PdNPs materials. FTIR results exposed that the existence of carboxyl, amine and carbonyl groups in a leaf extracts, which is accountable for the change of $Pd^{2+}$ ions to PdNPs reduction. The photoluminescence study confirms that these extracts were action like a stabilized and capping material. The results from our analysis revealed that *Basella alba*, *Allium fistulosum* and *Tabernaemontana divaricate* leaf extracts were found to be a cost-effective, environment-friendly and effectual source for the production of PdNPs and also formulated PdNPs can be utilized in biological applications.

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