ISSN 0974 - 9373

Vol. 26 No. 4 (2022) **J. Int. Acad. Phys. Sci.** pp. 457-470

Latent Use of Invasive Plant Species (*Lantana camara*) to Fabricate TiO_2NPs

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(Received November 25, 2022, Accepted December 3, 2022)

Abstract: Environment sustainability is a matter of high concern and the humanity is focused to maintain it at any cost. Thereby, the scientists have also enthused to develop innovative ways to fabricate nanomaterials which are environmentally sustainable, eco-friendly, economic and time-saving. Adopting non-conventional *greener* route, indigenous and abundantly available invasive plant species, *Lantana camara* has been used to fabricate Titanium dioxide nanoparticles successfully. The active phytochemicals of *Lantana camara* potentially act as reducing, stabilizing and capping agents to fabricate TiO_2NPs. Fabricated TiO_2NPs were validated by standard characterization tools and techniques, *viz*, UV-Visible spectroscopy, FTIR, FESEM-EDS, XRD, etc. Due to their enhanced transparency, photocatalytic efficacy, corrosion resistivity, and better UV-blocking efficacy, the fabricated TiO_2NPs have numerous utility in various sectors, *viz.*, sunscreen-cosmetics, dental implants, anti-fogging automobile mirrors, windows, fabrics, pharmaceuticals & medicines, pollutant eradicator, wastewater treatment, environmental remediation and hydrogen creation, etc.

Keywords: *Lantana camara*, TiO_2NP, Non-conventional approach, FESEM/EDS, XRD.

1. Introduction

Anthropogenic activities are majorly responsible for the environmental degradation, ecological imbalances, release of industrial wastes, hazardous & pollutants, etc. A large number of nanomaterials with potency as environment remediation have been synthesized via conventional routes, i.e. chemical and physical methods[1-15], but in view of use of chemicals and release of toxicants enroute their synthesis, these materials are non-eco-friendly and non-biocompatible, thereby damaging the environment and eco-system. Consequently, now-a-days, the scientific communities advocate adopting the non-conventional methods for nanomaterial synthesis[16-24]. A large number of nanomaterials have been synthesized via *greener* route using extract of various plant species as they are richly constituted with many bioactive phytochemicals, *viz.*, terpenoids, alkaloids, flavonoids, polyphenols, steroids, saponins, tannins, vitamins, etc. Individually each of these organic compounds acts as reducing, stabilizing and capping agents thereby producing nano-sized materials[16-41].

Among metal/metal oxide nanoparticles/nanomaterials, titanium based nano-sized particles and nanocomposites have fascinated considerable interest on account of their high refractive index (n = 2.4), photocatalytic, anti-fogging, self-cleaning properties and many other chemical and mechanical qualities. TiO_2NPs are ideal for vivacious applications as anti-microbial, anti-fungal, anti-termite, antioxidant, photo-catalytic, bio-remedial, decontaminant, eco-sanitizer, dental implants, eco-remedial, coatings, inks, plastics, drugs, medicines, food, cosmetics, textiles, windows, anti-fogging automobile mirrors, drug-pharma, agriculture, eco-purification, quantum dots based solar cells, energy reservoirs, biosensors, anticorrosive coating, manufacturing of the durable light weight articles, etc.

A lot of plants/bio sources have been used to fabricate TiO_2NPs[3,22,26,29,33]. However, so far, none have used weeds/invasive plant species as non-conventional synthesis of TiO_2NPs. Weeds and invasive plant species are mostly treated threat to the agriculture lands and thus most of these are either burnt or thrown away. However, they are also natural source of numerous bioactive phytochemicals with potency to generate nanomaterials. Thereby, in the present investigation, with focus on “best out of waste”, *Lantana camara* leaves’ extract-mediated fabrication of TiO_2NPs has been carried out.

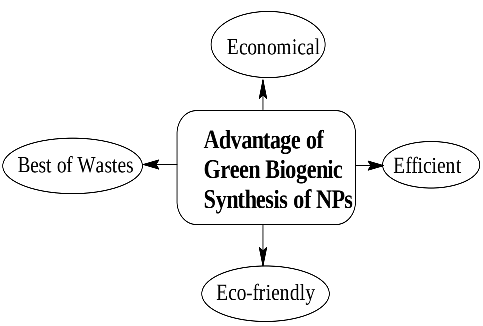

Figure 1. *Lantana camara* shrub

Lantana camara, one of the indigenous and abundantly available invasive species (Figure 1), is richly constituted with many such bioactive phytochemicals Figure 2), *viz.*, polyphenols, flavonoids, alkaloids, tannin, saponin, glycosides, reducing sugars, glycosides, steroids, tri-terpenes, coumarins, catechins, lignins, essential oil, etc.[42-48]. As per literature, the leaves' extract of *Lantana camara* has various metabolites constituted with active functional groups, *viz.*, N-H, C-H, OH, C=O. Furthermore, it has also been revealed that it has 66 bioactive compounds and 19 of these has high potency as anti-microbial, anti-tumor, anti-inflammatory, antiandrogenic, antioxidant and free radical scavenging activities[47-48]. Consequently, the aqueous extract of *Lantana camara* leaves has been probed to generate desired metal oxide nanoparticles, *i.e.* TiO_2NPs.

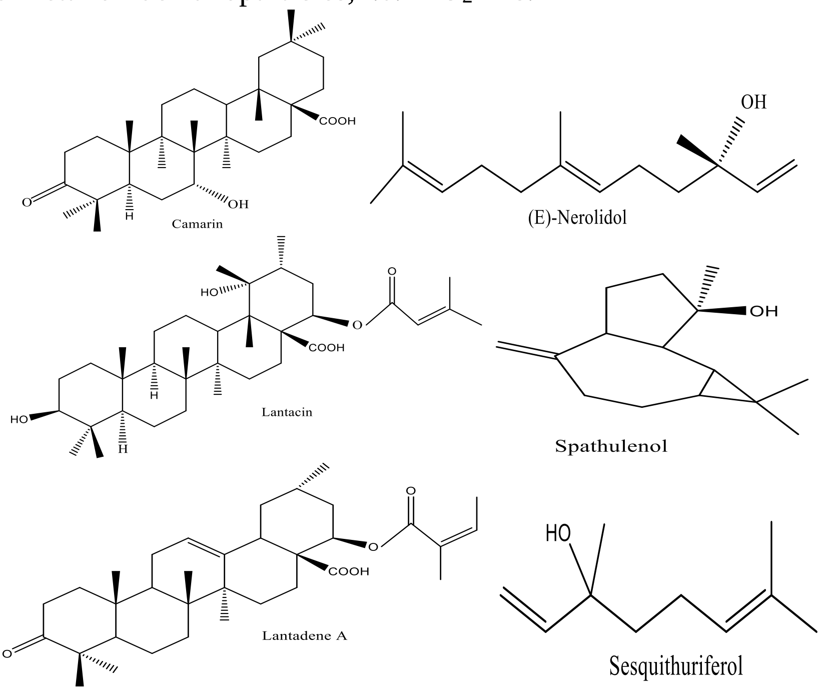

Figure 2. Some major bioactive photochemical of *Lantana camara* leaves

2. Materials and Method

2.1 Materials

AnalR grade Titanium dioxide (TiO_2) was used as standard metal salt precursor. Fresh leaves of *Lantana camara* were collected from the campus of University of Rajasthan, Jaipur, India. Triply distilled water was used to prepare experimentation solutions. Digital pH meter (Remi), heating-cum-magnetic stirrer (Remi), Soxhlet extractor (Borosil), heating mantle, highspeed centrifuge (Eltek), and sophisticated standard instruments for characterization, *viz.*, Ocean optic HR 4000 High-Resolution UV-Visible spectrophotometer, FTIR Spectrometer (PerkinElmer), FE-SEM/EDS 450 (FEI) ZEISS, XPERT-PRO diffractometer system etc. were employed to carryout present investigation.

2.2 Preparation of Aqueous Extract of *Lantana camara* Leaves

Aqueous extract of *Lantana camara* leaves was prepared as per the standard procedure[34,40-41]. Fresh leaves of *Lantana camara* were collected from the campus of University. They were washed to remove any dirt or dust or unwanted materials adhered to their surface and shade dried for two weeks. With the help of mixer grinder the dried leaves were crushed to fine powder. *Lantana camara* leaves' fine powder (20g) was refluxed with 300ml doubly distilled water using Soxhlet extractor for many cycles. Fine colloidal extract was centrifuged for half an hour and then filtered using Whatmann No. 1 filter paper to remove particulate matter. The dark brown extract was stored as stock solution for experimentation.

2.3 *Lantana camara* Leaves' Extract Aided Fabrication of TiO_2NPs

TiO_2NPs were fabricated as per standard procedure[34,40-41] followed by their characterization employing standard tools and techniques. Aqueous extract of *Lantana camara* leaves and 0.01M TiO_2 solution were mixed in 2:1 ratio in a conical flask and subjected to the magnetic stirrer for stipulated time period at room temperature. A change in the color of the reaction mixture (brown to creamish white) was observed which could be prelim indication of reduction of metal ions to nano size particles. Reaction mixture was centrifuged @5500 rpm for 10 min and then filtered.

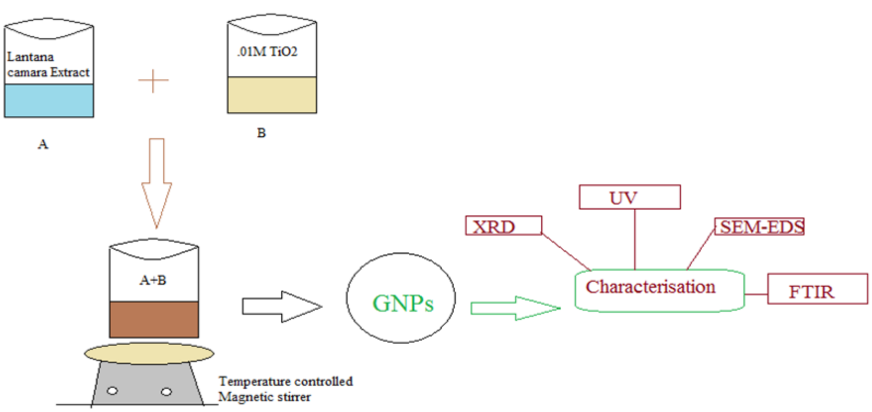

Figure 3: Schematic sketch of *L. camara* leaves' extract aided fabrication of TiO_2NPs.

The above process was repeated thrice. The whole process of fabrication has been illustrated by Figure 3. Finally, the sediments were collected, vacuum dried in an oven and stored in a clean pre-sterilized glass vial for characterization and thereafter usages. Fabrication of TiO_2NPs was validated by carrying out characterization using standard tools and techniques, viz., UV-vis spectroscopy, FTIR, SEM-EDS/Mapping, XRD etc.

2.4 Characterization of Eco-Fabricated TiO_2NPs

The absorption spectra of fabricated TiO_2 nanoparticles were monitored by UV-visible spectrometer (Ocean optic HR-4000 High-Resolution) at the wavelength range 200-800 nm. Further the Fourier transform infrared spectroscopy of plant extract and fabricated TiO_2 nanoparticles were analyzed to find out the possible functional groups for reduction and formation of nanoparticles using PerkinElmer, FTIR spectrometer. Crystal structure of TiO_2 nanoparticles was analyzed by X- ray diffraction (PANalytical, X'Pert Pro Diffractometer). Surface Morphology of the nanoparticles has been examined by field emission electron microscopy (FE-SEM Nova Nano 450) and EDS for elemental mapping were conducted through same instrument.

3. Results and Discussion

3.1 Preliminary Visual Parameter

As a preliminary parameter of the formation of the TiO_2 nanoparticles, a change in colour of the reaction mixture (dark brown to creamish white) was observed (Figure 4). This may be due to the excitation of surface plasmon vibrations of TiO_2 nanoparticles.

Figure 4. Preliminary visual parameter showing change in colour of reaction mixture consisting fabricated TiO_2NPs
[Flask A: *L. camara* extract; Flask B: 0.01M TiO_2 solution; Flask C: Reaction mixture]

3.2 UV-Visible Spectroscopic Analysis

Fabricated TiO_2NPs were characterized through UV-Vis spectrophotometer with range of absorbance from 200-800 nm. The UV-Visible spectral analysis is a convenient, preliminary and indirect method for characterization of TiO_2NPs, based on optical properties called surface Plasmon resonance (SPR). A broad absorbance peak at λ_{max} 461 nm ($\varepsilon = 0.45$) (Figure 5) has been observed in the UV-Visible absorption spectra, endorsing the formation of TiO_2NPs.

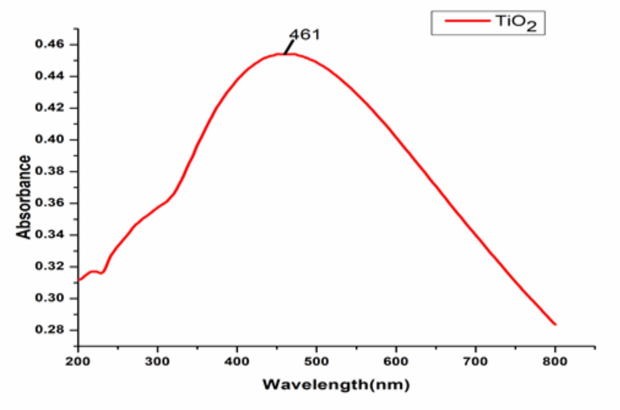

Figure 5. UV-visible spectra of TiO_2NPs synthesized by *Lantana camara* leaves' extract

3.3 FTIR Spectroscopic Analysis

FTIR spectral analysis was carried out (in spectral range of 400- 4000 cm^{-1}) to validate the active phytochemicals present in the *Lantana camara* leaves' extract which are responsible for the TiO_2NPs formation and thereafter its stabilization. The oven dried samples were grounded with KBr to form pellet and then analyzed their individual spectra (Figures 6 and 7) using FTIR Spectrometer (PerkinElmer)[49-50]. Various absorption peaks were observed in the FTIR spectrum of *Lantana camara* leaves extract (Figure 6). Bbroad absorption peak at 3500 cm^{-1} corresponds to the stretching frequency of hydroxyl (-OH) group, C-H stretching frequency at 3050 cm^{-1}, =C-H stretching frequency at 3100-3010 cm^{-1} (m), -C=C stretching frequency at 1600 cm^{-1} (s), C=O (ester) stretching frequency at 1750-1725 cm^{-1}, C-O stretching frequency at 1180 cm^{-1}, =C-H bending vibration at 1000-900 cm^{-1} (s), and -C-H bending (alkane) at 1375-1400 cm^{-1}. Comparative analyses of FTIR spectra of the extract and the fabricated TiO_2NPs (Figures 6 and 7) reveal some shifts in the peaks and appearance of some new bands too. The non-bonded surface hydroxyl (-OH) modes appeared between 3650-3600 cm^{-1}, and the peak at 600-595 cm^{-1} due to Ti-O stretch vibrations. The active phytochemicals present in *L. camara* leaves bounded with the metal and serve as reducing, stabilizing and capping agents to avoid agglomeration, thereby stabilize formed TiO_2NPs.

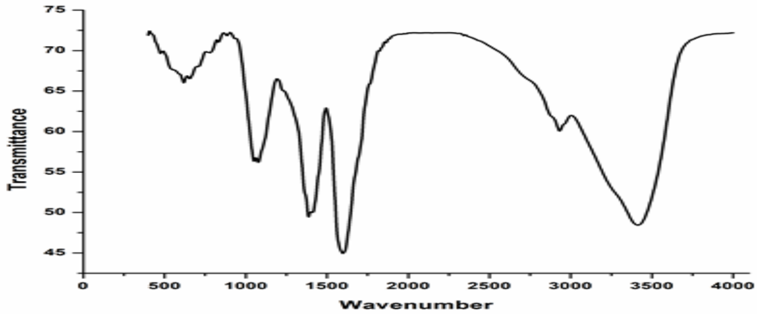

Figure 6. FTIR spectra of *L. camara* leaves' extract

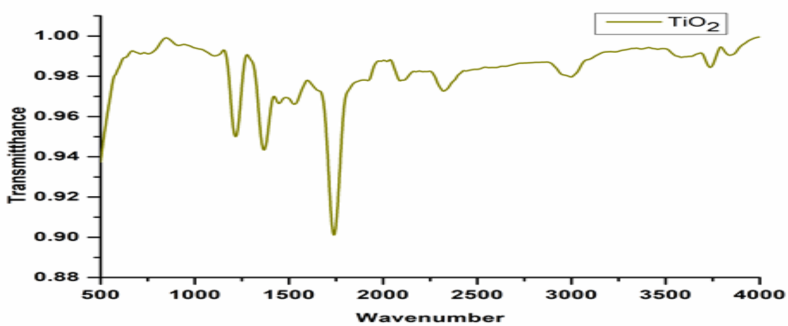

Figure 7. FTIR spectra of fabricated TiO_2NPs using *L. camara* leaves' extract

3.4 FE-SEM/ EDS Analysis

The FE-SEM analysis was used to investigate the surface morphology (shape) as well as the size of the synthesized TiO_2NPs. FESEM micrographs of *L. Camara* leaves extract mediated synthesized TiO_2NPs (Figure 8) clearly illustrated the spherical shaped clusters of nano sized particles in the range of 50-98 nm.

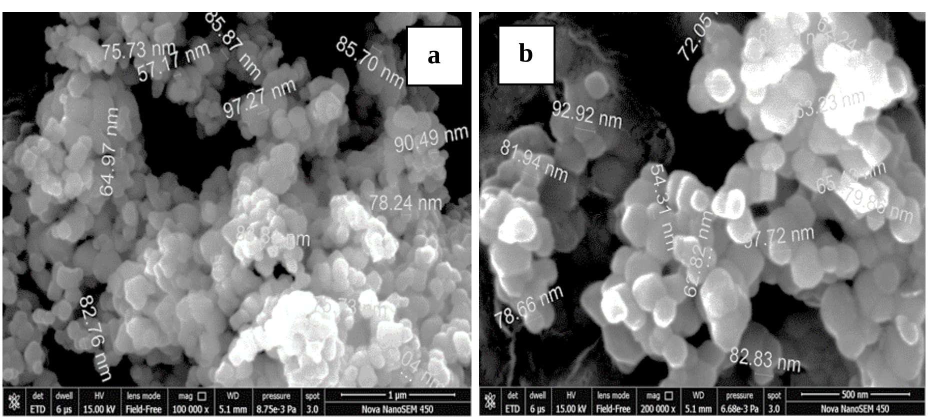

Figure 8. SEM Micrographs of fabricated TiO_2NPs at (a) 1µm and (b) 500nm

The elemental composition of the synthesized TiO_2NPs as well as the normalized concentration of elements was clearly depicted in EDS. The normalized concentration of Ti and O element found 65.74 and 38.29 wt. % respectively. EDS/mapping spectra of TiO_2 (Figure 9) revealed the presence of Ti, C, O and N elements. The elemental signals, C, O and N might be due to the phytochemicals of *L. camara* extract supporting extract's capping potency towards fabricated TiO_2NPs.

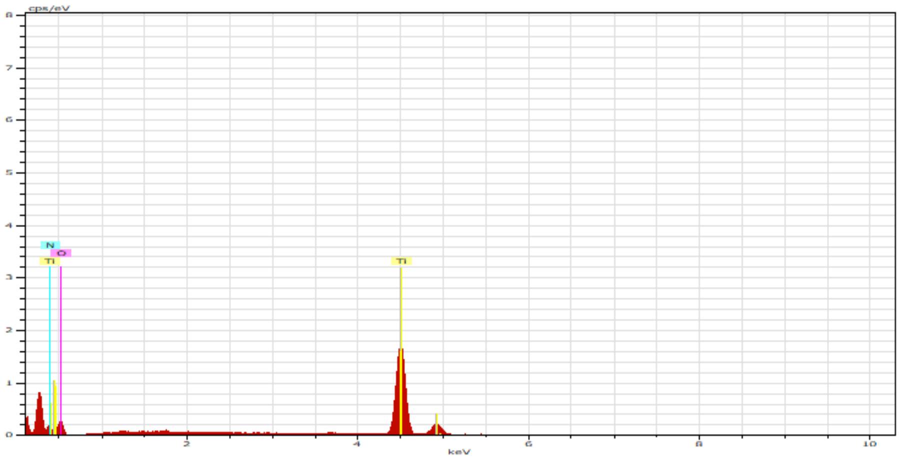

Figure 9. EDS with elemental mapping of TiO_2NPs.

3.5 XRD Analysis

The size and structural elucidation of fabricated TiO_2NPs was carried out by XRD analysis using XPERT-PRO Diffractometer system operating in the reflection mode with Cu-Kα radiations (40 kV, 40 mA). Data was taken for the 2θ range of 10° to 90° with a step size (2θ) of 0.30. Morphology index (MI) derived from FWHM of XRD data elucidate the interrelationship of particle size and specific surface area. The observed data revealed that MI has direct relationship with particle size and an inverse relationship with specific surface area.

The XRD pattern of eco-friendly synthesized TiO_2NPs (Figure 10) reveal the formation of well crystalline TiO_2NPs with Anatase phase with diffraction angles 25.36, 36.98, 37.83, 38.61, 48.07, 53.92, 55.10, 62.18, 62.72, 68.80, 70.34, 75.07, 76.08, 82.71 attributed to (101), (103), (003), (112), (200), (105), (211), (213), (204), (116), (220), (215), (301) and (224) planes respectively. The major peak at 28.36 associated with (101) crystalline plane of TiO_2NPs confirms the formation of Anatase form only. The results reveal that the structure of fabricated TiO_2NPs was tetragonal with space group 141/amd and results were in good agreement with reference code 98-000-9852.

Average particle size was estimated using the Debye-Scherrer equation[49-50]:

$$D=0.9\, \lambda / \beta \cos \theta$$

where D is the average diameter of the NPs, λ is the wavelength of X-Ray (1.5406 Å), and β is FWHM (angular full width at half maximum) of the peaks, and θ is diffraction angle.

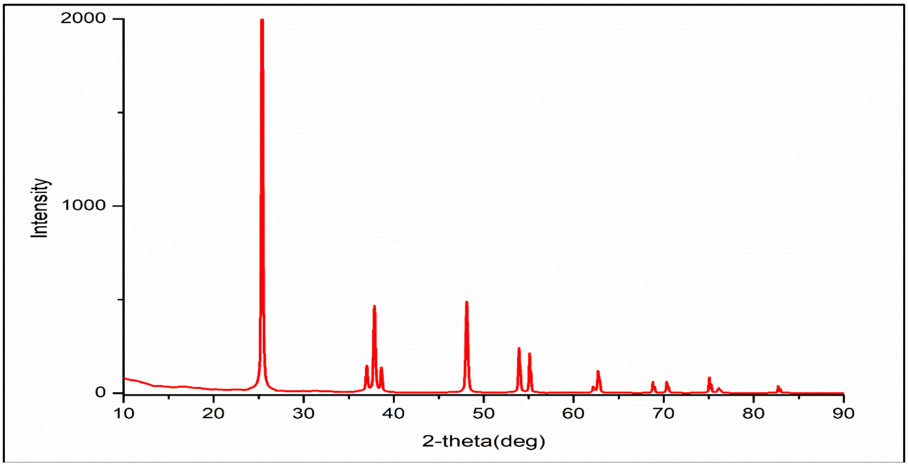

Figure 10: XRD pattern of fabricated TiO_2NPs.

Table 1 Average value of XRD Data of TiO_2NPs

Particle Size (nm)	Surface Area (nm^2)	Volume (nm^3)	SA/V Ratio
61	12644	207316	0.06

Average crystalline size of the fabricated TiO_2NPs as per calculation was found 61 nm (Table 1), thereby confirming the formation of nano sized TiO_2 particles, while the 2θ value at 25.3695 and at 48.0769 established the Anatase structure of TiO_2. The intensity of XRD peaks of the sample depict that the TiO_2NPs produced has crystalline nature and absence of spurious peaks illustrated the purity of the TiO_2NPs.

4. Conclusions

With the focus on, 'Bests of Wastes', the invasive plant (*Lantana camara*) was used for the eco-fabrication of TiO_2NPs. Fabrication of TiO_2NPs was successfully accomplished and the product formed was validated by characterizing through various standard tools and techniques.

The characterization data of UV-Vis spectrophotometer, FTIR spectrometer, SEM-EDS/mapping, and XRD reveal efficacious formation of well crystalline tetragonal 61 nm sized TiO_2NPs.The active phytochemical constituents of the *Lantana camara* leaves extract stabilized the synthesized TiO_2 NPs and prevent conversion of anatase structure into rutile structure and behaved as capping agent to prevent the agglomeration of NPs. The eco-fabricated TiO_2NPs has significant potency to act as eco-remedial, pollutant-eradicator, and can be a pivotal tool for environment sustainability. It can be very clearly advocated that the non-conventional approach is much efficient, effective, less time consuming, sustainable and environmentally benign.

Acknowledgements: With a deep sense of gratitude, one of the authors, Prof. Alka Sharma, wishes to dedicate this research article to the profound memory of (Late) Prof. P. N. Pandey, the founder General Secretary of IAPS, who, as a pathfinder always motivated to keep moving on and excel in research and academic ventures.

The authors expresses thanks to the Head, Department of Chemistry, University of Rajasthan, Jaipur for providing necessary research facility and the authors also acknowledge MRC, MNIT, Jaipur for providing access to necessary characterization facilities.

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