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Non-Conventional Fabrication of Ag@ZnO Nanocomposite Core-Shell using Extract of Weed (*Euphorbia hirta*) Leaves

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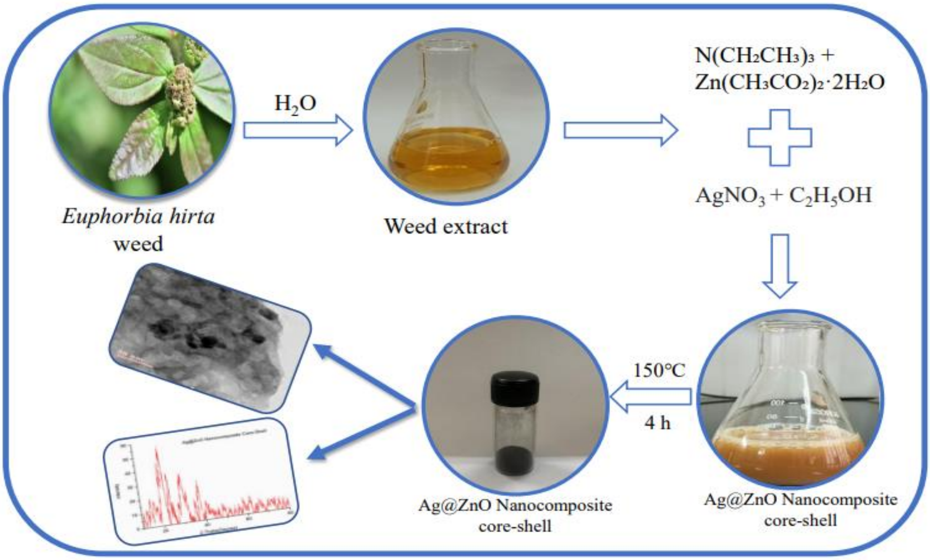
Abstract: Authors introduce a novel, economical and eco-friendly method to fabricate Silver@Zinc oxide nanocomposite core-shell (Ag@ZnO NC core-shell) with significant efficacy in many industrial sectors, medical, environment-remediation, waste-water treatment, etc. Explicitly focused on "*Best out of Waste*", the extract of leaves of Weed (*Euphorbia hirta*) was employed to fabricate Ag@ZnO NC core-shell. *Euphorbia hirta* leaves are richly constituted with the bioactive phytochemicals which are promising reducing, capping and stabilizing agents. Validation of synthesized *green* Ag@ZnO NC core-shell was carried out by using standard characterization tools and techniques, *viz.*, UV-vis spectrophotometer (UV-vis), Fluorescence spectrophotometer (FL), Fourier transform infrared spectroscopy (FTIR), X-ray Diffraction (XRD) and High-resolution transmission electron microscopy (HR-TEM). Data obtained from the characterization tools were analysed. With XRD the size of the *green* Ag@ZnO NC core-shell was found to be below 90 nm; whereas the structure and the polymorphic nature were revealed by HR-TEM and the selected area electron diffraction (SAED) respectively. *Green* Ag@ZnO NC core-shell has a vast range of industrial applications including eco-remediation. In comparison to many conventional synthesis methods for nano-materials, the non-conventional method of fabrication of Ag@ZnO NC core-shell is an economically viable, less time consuming, non-toxic and easy to handle.

Keywords: Euphorbia hirta, Ag@ZnO nanocomposite core-shell, High-resolution transmission electron microscopy, X-ray Diffraction.

Graphical Abstract:



1. Introduction

The fabrication of nanoparticles and nanocomposite core-shell through natural sources become a prevalent and eco-friendly approach[1]. The material at the nanometer scale shows unique properties which improve the optical[2-4], electrical[5,6], and many other physicochemical[7,8] properties of the material. Most popularly synthesized and used silver nanoparticles possess unique electrical, optical, and biological properties[9]. Moreover, silver[10] and zinc oxide[11] nanomaterials are nontoxic material as they are neutral and alkaline. The process of development of core-shell composite materials is the most vigorous area of nanoscience research[12-22]. The most attractive area of a nanocomposite is the core-shell structure because of its applications like heterogeneous catalysts[16], drug delivery[17], energy storage[18], and anticounterfeiting[19]. Nanocomposite materials comprising metals and semiconductors are captivating, due to their composition with enhanced optical, catalytic, and biomedical properties including phenomena such as harvesting, and concentration of electromagnetic radiation[20-23].

Among such nanoparticles and nanocomposite structures, the Ag@ZnO NC core-shell has attracted attention because it has vast applications towards eco-remediation including photocatalysis, and UV screening applications[24]. The synthesis of nanocomposite core shell is well reported via conventional methods[12-22, 24], but now-a-days in view of the environmental fortification, *greener* approach is much in practice. *Green* approach to fabricate nano scale materials has been well adopted and successfully fabricated many nano structured metal/metal oxide materials[25-26]. Development of eco-friendly procedures (*green* approach) to fabricate nanocomposite core-shell has achieved fast momentum. Only a few have carried out *green* synthesis of Ag@ZnO nanocomposite (NC) core-shell using essential oil of wild ginger, *Alstonia Scholaris*, and neem (*Azadirachta indica*) leaf [27-29]. Literature remains silent about the use of weeds/invasive plant-parts to fabricate Ag@ZnO NC core –shell. Thus, adopting *greener* route and also using "*Best out of Waste*" approach, *Euphorbia hirta* (a weed) leaves' extract was explored to synthesize Ag@ZnO NC core-shell.

The largest genus of the family *Euphorbiaceae* is *Euphorbia* with about 1600 species and it is characterized by the presence of white milky latex which is toxic[30-32]. There are many species of *Euphorbia* that are used in traditional medicines and all the species exude a milky juice when broken, which is more or less poisonous and used as an ingredient in arrow poisons[30-32]. Lattices of *E. ingens*, *E. mey*, *E. tirucalli,* and *E. triangularis* are also possible sources of rubber[33].

Phytochemical analysis of *Euphorbia hirta* shows presence of flavanoids, triterpenoids, amino acids, carbohydrates, terpenoids, alkaloids, reducing sugars, steroids, tannins, proteins, fats, oils, mucilages, glycoside, saponin, coumarin, anthroquinones, etc.[30-33]. *E. hirta* possesses antibacterial, anthelmintic, antiasthmatic, sedative, antispasmodic, antifertility, antifungal, and antimalarial properties[30-33]. Moreover, most of these bioactive phytochemicals, co-existing in many other weeds too, has potential efficacy to impede metal-corrosion[34-37].

In the present work, undergoing via a non-conventional synthesis route and furthermore, instead of using any natural resources, a weed (*Euphorbia hirta*) was used to fabricate Ag@ZnO NC core-shell focused with "*best out of waste*" approach and endorsing the product by characterized with the standard tools and techniques. *Euphorbia hirta* is an indigenous weed consisting of many active phytochemicals individually known to act as reducing, capping and stabilizing agents. Fabrication process of Ag@ZnO Nanocomposite core-shell is cost effective, less time consuming, environmentally benign, readily scaled up for large-scale synthesis, and it is always preferable due to its advantages over other conventional approaches.

2. Materials and Methods

2.1 Materials:

All the chemicals used as precursors for the nanocomposite core shell synthesis were of AR grade, *i.e.* Silver nitrate, $AgNO_3$, Zinc acetate dehydrate, $Zn(CH_3COO)_2.H_2O$, and Triethylamine, $(CH_3CH_2)_3N$. All chemicals were used without further purification. Doubly-distilled water was used to prepare all the standard solutions of precursors. Fresh leaves of *Euphorbia hirta* were collected from campus, University of Rajasthan, Jaipur, Rajasthan, India.

2.2 Preparation of weed (*Euphorbia hirta)* leaves extract:

Fresh leaves of *Euphorbia hirta* were washed in running tap water, followed with distilled water to wipe out the dust particles, and then allowed to dry in shade for 1-2 weeks at room temperature. After complete drying of the leaves, they were powdered using a mixer grinder and stored in an airtight container at room temperature. As per the standard extraction procedure[25-26, 34-37], Soxhlet extractor was used. 20 g of *Euphorbia hirta* dried and finely powdered leaves were extracted employing distilled water as a solvent. The aqueous solution obtained after extraction is centrifuged and filtered through Whatman paper No. 1 to remove the plant residues and to get a clear solution. After that, for further analysis extract was stored in a refrigerator at 4^0C.

2.3 Fabrication of Silver@Zinc Oxide Nanocomposite Core-shell:

Triethylamine was dispersed into 60 ml of *Euphorbia hirta* leaves’ extract containing 1 mM of Zinc acetate dihydrate with continuous stirring for about 1 h at room temperature. Thereafter, silver nitrate (1 mM) was dispersed in ethanol and the solution was ultrasonically shaken for about 30 min. Silver nitrate solution was introduced into the reaction mixture solution consisting of *Euphorbia hirta* leaves’ extract, zinc acetate and triethylamine. This reaction mixture was ultrasonicated for about 2 h. The product was centrifuged at 10,000 rpm for 10 min and washed with distilled water and ethanol several times. Finally, it was dried in a vacuum oven at 150°C for 4 h to obtain Ag@ZnO NC core-shell via non-conventional approach.

2.4 Characterization:

The product, *green* Ag@ZnO NC core-shell so obtained was endorsed by carrying out characterization using standard tools and techniques. UV-visible spectra of Ag@ZnO NC core-shell were acquired by Shimadzu UV-2450, Japan, UV-vis spectrophotometer over the range of 200 to 800 nm. FT-IR Perkin-Elmer 1725X was used to analyze the vibrations and stretching in the range of 400-4000 cm^{-1}. X-ray diffraction (XRD) pattern of the Ag@ZnO nanocomposite core-shell was obtained using the X-PERT PRO Diffractometer. XRD was performed at 40 Kv voltage with 40 mA current and 2θ range of 10-80° using Cu $K\alpha$ radiation of wavelength (λ= 1.5406 Å) at room temperature. The surface morphology of Ag@ZnO nanocomposite core-shell was observed using Tecnai G2 20 S-TWIN [FET] high-resolution transmission electron microscopy with 200Kv voltage.

3. Results and Discussion

Ag@ZnO NC core-shell was fabricated by *greener* approach and analysed by standard characterization techniques. The primary visual observation was the change colour of the reaction mixture depicting the formation of Ag@ZnO NC core-shell. The pH values of the precursor solutions, viz., $AgNO_3$, $Zn(CH_3COO)_2.H_2O$, $(CH_3CH_2)_3N$, aqueous extract of *Euphorbia hirta* leaves, and the reaction mixture were observed as 4.52, 6.13, 10.62, 5.68 and 6.10 respectively. The product formed was advanced for its justification by obtained UV-visible spectra, FL-spectra, FT-IR spectra, HR-TEM and XRD and thereafter analysing them.

3.1 UV-visible Analysis:

The UV-visible absorption spectra of the Ag@ZnO nanocomposite core-shell (figure-1) revealed an absorption maximum at 260 nm. The band gap energy of Ag@ZnO nanocomposite core-shell was calculated by fitting the absorption data in the equation:

$$\alpha h\vartheta = K(h\vartheta - Eg)\text{^}2$$

With the Tauc plot (figure-2) the band gap energy value for the Ag@ZnO NC core-shell was found to be 3.08 eV. This value was observed to be very low as compared with the individual band gap energies of Ag(NPs) and ZnO nanomaterials (as 2.51eV and 3.63eV respectively) (these have been evaluated during a work carried out in our laboratory for some other purposes).

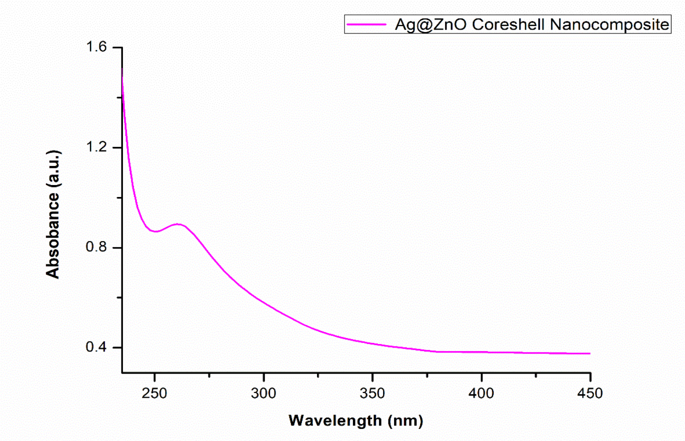


Figure-1. UV-vis spectra of Ag@ZnO NC core-shell

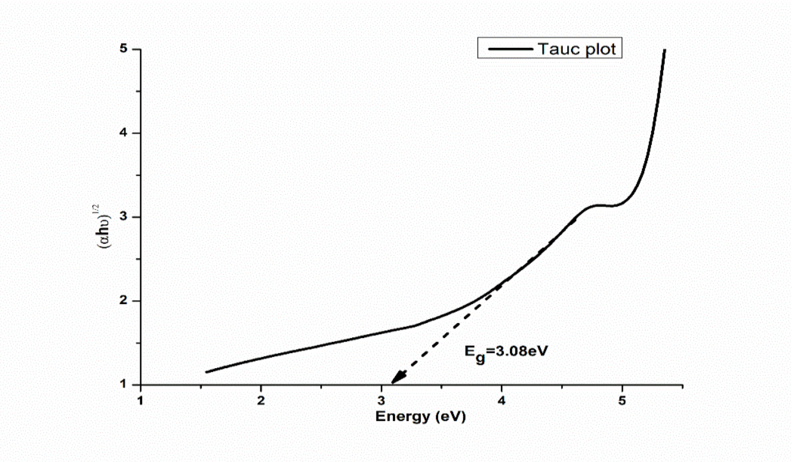


Figure-2. Tauc plot of Ag@ZnO NC core-shell

3.2 Fluorescence Analysis (FL):

The broad visible range emission of Ag@ZnO NC core-shell was observed at 420 nm (figure-3) which attributed to deep-level or trap state emission. The increased local electric field, the increased recombination rate, and the electron transfer, possibly these three mechanisms has contributions to the observed increased photoluminescence[38].

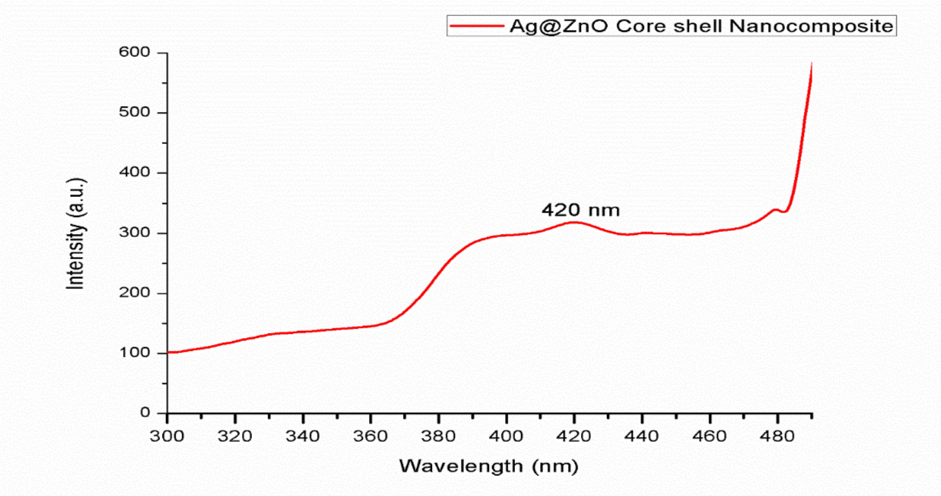


Figure-3. Fluorescence spectra of Ag@ZnO Core-shell Nanocomposite

3.3 Fourier-Transform Infrared Analysis (FT - IR):

The strong peak after 700 cm^{-1} was due to Ag@ZnO NC core-shell, and peaks at 3465 cm^{-1} and 1600 cm^{-1} attributed to –OH vibration probably originating from the residual water. The C-O stretching vibration at 1376 cm^{-1}, the stretching vibration of C=C groups at 1453 cm^{-1}, and the peaks between 1330 cm^{-1} and 740 cm^{-1} resulted from CH bending. The appearance of absorbance band at 1062 cm^{-1} in the spectrum of Ag@ZnO NC core-shell may related to van der Waals forces of interaction between Ag and C-C bonds.

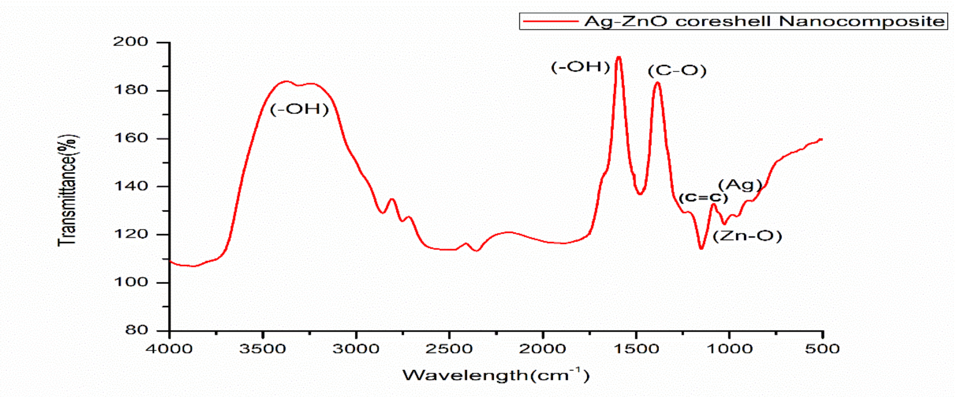


Figure 4. FT-IR spectra of Ag@ZnO NC core-shell

3.4 High-Resolution Transmission Electron Microscopy Analysis:

HR-TEM images (Figures 5a and b) illustrated the morphology and structure of fabricated *green* Ag@ZnO NC core-shell. HR-TEM samples were prepared by dipping the TEM grid in an aqueous dispersion of nanomaterials followed by freeze-drying for 12 h for clear observation. The approximate size of the Ag@ZnO NC core-shell is 30~35 nm. The images reveal that the Ag cores encapsulate inside a single ZnO shell. The scale of HR-TEM is in 50 and 100 nm range. The polymorphic structure of Ag@ZnO NC core-shell was very well depicted in Figure 6.

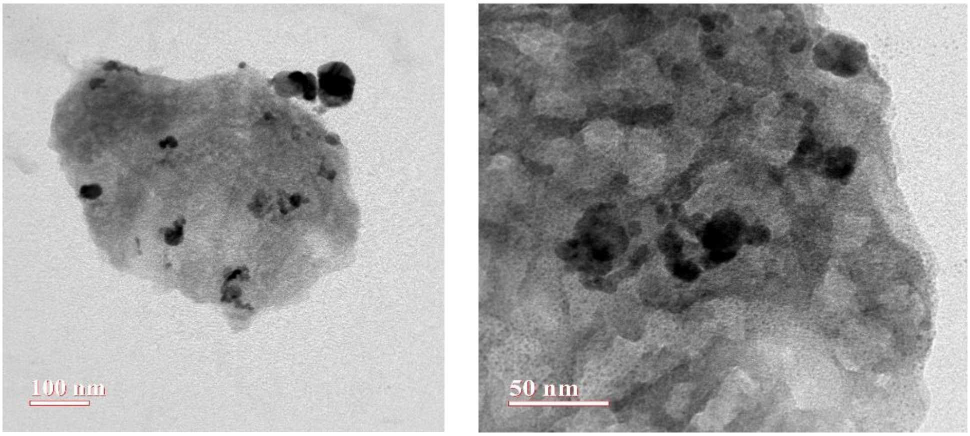


Figure-5 (a and b) High-resolution TEM images of Ag@ZnO NC core-shell

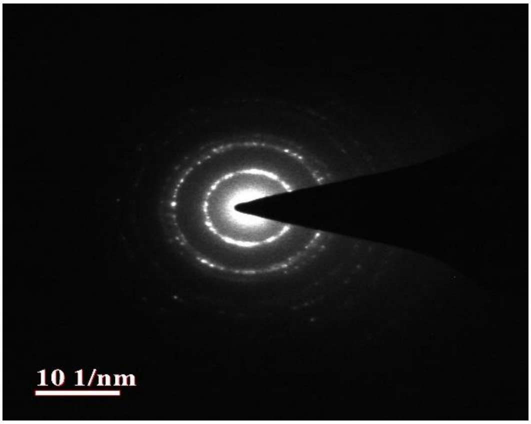


Figure-6: Polymorphic structure of Ag@ZnO NC core-shell

3.5 X-ray diffraction (XRD) analysis:

The XRD pattern of synthesized Ag@ZnO NC core-shell was recorded in the range from 20° to 80° of 2θ values (figure-7). The diffraction peak was found at various 2θ values 41.00°, 59.38°, 74.70° and 88.94° corresponding to the planes (011), (002), (112), and (002) respectively. The indices of these planes correspond to the cubic fluorite type structure of Ag@ZnO NC core-shell.

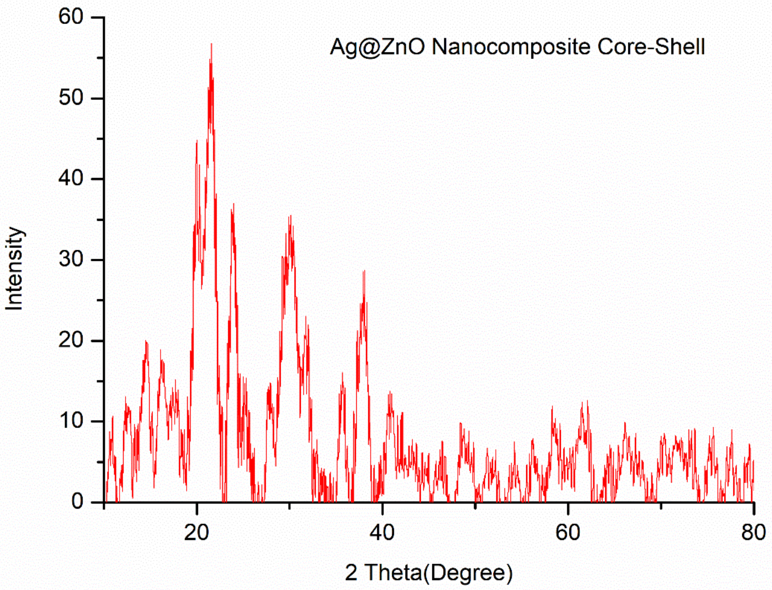


Figure-7: XRD pattern of Ag@ZnO NC core-shell

Rietveld refinement of the XRD patterns was performed using PANalytical high score software and the resulting pattern was found matched with JCPDS reference code 98-005-8385 of Ag@ZnO NC core-shell. The phase structure of the Ag@ZnO NC core-shell was cubic with Im-3m as a space group with the lattice constant value (a) is 3.1100 Å. The crystallite size of prepared nano-sized material was estimated by using Debye-Scherrer's formula:

$$\text{Average crystallite size (D)} = 0.9\ \lambda\ \beta\ \cos\theta$$

where A is the wavelength of the X-ray source used for analysis, β is angular peak width at half maxima in radians and θ represents Bragg's angle of diffraction. The average crystallite size, using the above Scherrer formula, was found to be 90 nm.

4. Conclusions

Keeping in view of environmental legislations, authors have adopted an eco-friendly approach to fabricate Ag@ZnO NC core-shell via non-conventional route. To achieve this target, authors have initiated novelty with focus on espousing *"best out of waste"*, and using the indigenous weeds/invasive plant-parts (i.e. *Euphorbia hirta* (weed) leaves' extract), instead of any medicinally useful natural raw materials to produce nanomaterials.

The morphological, compositional, and optical properties of the product, Ag@ZnO NC core-shell, were validated by various standard characterization tool & techniques, *viz.*, HR-TEM, XRD, FT-IR, FL, and UV-vis. FTIR spectra confirm the presence of the Zn-O and Ag bond and justify the role of the bioactive phytochemicals present in the extract of *E. hirta* leaves to act as reducing, stabilizing and capping agents. The sharp peaks of XRD indicated crystalline nature and the indices of planes analogous to these peaks illustrated the cubic fluorite type structure of Ag@ZnO NC core-shell. The HR-TEM images reveal that the Ag cores encapsulate inside a single ZnO shell. A polymorphic structure of Ag@ZnO NC core-shell of approximate size 30~35 nm was obtained.

The photocatalytic activity efficacy of Ag@ZnO NC core-shell can be endorsed by comparing its band gap energy with the standard reported individual band gap energies of ZnO and Ag(NPs). A shift in band gap energy was observed owing to the encapsulation of Ag(NPs) in ZnO shell, thereby enhancing the photocatalytic activity of Ag@ZnO NC core-shell.

The application worth of Ag@ZnO NC core-shell, is not covered in the present manuscript, it has been communicated separately for the publication, wherein authors have successfully investigated its photocatalytic degradation activity towards organic dyes and industrial waste-water dye effluents and found to have very encouraging efficacy.

As a conclusive remark, with a successful synthesis of Ag@ZnO NC core-shell via non-conventional method, authors wish to advocate for adopting the *green* synthesis process focused with "*best out of waste",* employing indigenous weed/invasive plants, as a very novel, striking and operative approach to fabricate nano-sized materials.

Moreover, it is an environmentally benign, faster, simpler and economically viable approach yielding products of high purity and significant utility in many sectors, industries including eco-remediation.

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