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Green Synthesis of ZnO Quantum dots from *Azadirachta Indica* leaf and its photocatalytic activity

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ABSTRACT

The main objective of the current study is to look into the synthesis of zinc oxide (ZnO) quantum dots (QDs) using zinc acetate that is precursor and neem leaf extract as a reducing agent. The synthesis of ZnO QDs was monitored by ultraviolet-visible absorption spectroscopy at wavelength 324 nm. The optimal synthesis of ZnO QDs was recorded at a temperature of 20 degrees and 40 degrees, the physical interaction between ZnO QDs and methyl orange was studied, the interaction between them to check the photo catalytic effect of it. ZnO QDs were applied in rheo leave to see the plasmolysis effect of it, the work opens up the application in several commercial and clinical products, preservatives, and fluorescence labeling including the antimicrobial agents. The plant contains numerous compounds in its body. These biomolecules are produced and stored by plants for their survival, physiological processes, energy, reproduction, safety, etc. interestingly most of these plant-based biomolecules exhibit different medicinal properties and many of these compounds or biomolecules are either excellent drugs or precursor of some important drugs. So, our motive is to find the pharmaceutical importance of the plants and their interaction with the Zn for further research, which can thereby be used for the beneficial needs of humans.

Keywords: *Zinc Oxide, quantum dots, methyl orange, plant, environment, nanoparticles*

Introduction

Nanoscience materials have attracted increasing attention during the past few decades due to their marvelous properties and a wide range of applications such as catalysis, electronics, optics, and environmental biotechnology application [1]. The increase in world demand to manipulate nanoscience materials trying to solve technological environmental situations should be accomplished by green chemistry processes to minimize the hazards of the traditional chemical processes. Among the most widely used nanostructured metal oxides, ZnO is considered as one of the most important metal oxides with unique properties as high surface energy, high electron mobility, cheap and environmentally nontoxic, and the most significant property s at wide-bandgap [2-3]. This wide-bandgap makes ZnO a field of many studies in using and improving the applications in electronics light emitters, chemical sensors, and photocatalysts [2]. The nanoparticles have gained significant interest because of their very small size (1- 10nm) with the huge surface to volume ratio that causes both physical and chemical variation in their properties such as catalytic activity, thermal and electrical conductivity, biological and sterical properties, melting point, mechanical properties, and optical absorption compared to the bulk of the same chemical composition [4]. Zinc oxide (ZnO) nanostructure is the forefront of research because of their distinct properties and wide range applications like sensors, transistors, nano piezo electronics, Uv detections, nano optoelectronic devices such as light-emitting diodes, photodetectors, solar cells, biomedical uses like antimicrobial activity [5], nanofertilizers [6], etc.

Green chemistry involved the use of a set of principles, which decreases or eradicates the utilization or production of harmful materials in the design, manufacture, and application of chemistry production. In this approach, the biomolecules such as proteins, metabolites, etc. present in the biological system serve as reducing, capping and stabilizing agents for the synthesis of specific shape and size NPs [7]. The cost-effective cultivation, safety, short production time capability to scale up to allow plant systems as an attractive platform for the synthesis of metal/metal oxide NPs/QDs over another biological system like algae, fungi, bacteria, yeast, diatoms and human cells [8]. Zinc oxide nanoparticles (ZnONps) have been extensively explored for various biological applications because of their characteristic biocompatibility, antimicrobial activity, and wound healing [9]. Green synthesis is an environment-friendly alternate as compared to chemical synthesis, hence ZnONps using *Azadirachta Indica* (Neem) leaf extract have been synthesized in this study. ZnONps were then characterized using different analytical techniques including UV-Visible spectroscopy, FTIR, XRD, SEM, FESEM, and EDAX.

Nanomaterials are controllably synthesized to nano size (less than 100 nm) So that they can show some atom-like properties [8]. They are divided into atomic sizes so that the bandgap between the valence band and conduction band becomes wider and their surface area becomes larger, which results in their higher surface energy [9]. This branch of Nanoscience has attracted ample interest in many fields like biotechnology, physics, chemistry, environmental science, material science, and information technology [10]. Metal oxides have lots of applications in material science. They are widely involved in the preparation of several daily use products, including health care products, and are regarded as safe materials [11]. Nanotechnology has revolutionized its synthetic pathways to nanostructures, changing their properties immensely [12]. Among metal oxide nanoparticles, ZnONps have gained a lot of attention because of their biological applications like antibacterial, anti-fungal, and wound healing [13]. Various chemical and physical methods are available for the synthesis of metal oxide nanoparticles, but the green synthesis approach has now been utilized more often, due to the usage of lesser harmful chemical reagents, especially in the field of pharmaceuticals 3 and biomedicals [14]. Biosynthesis also becomes very important from the economical perspective and hence recent advancements are focused on this cost-effective synthesis [15]. The involvement of plants or plant leaf extracts in the synthesis provides various advantages including the production of the diverse sized and shaped metal oxides nanoparticles [16]. DNA has a major role as a genetic information tool. It guides the molecular machinery in living cells for the synthesis of enzymes and proteins by transcription and translation [17]. The interaction study of DNA with the metallic nanoparticles is of prime interest in nanobiotechnology [18]. Small molecules at the nanoscale can bind covalently or non- covalently with DNA [19]. Enhanced quality of nanoparticles and their similar size to the biological material, can allow these to readily interact with cells [20]. These can easily affect the potential of cellular response selectively and dynamically [21]. Some nucleic acid-based biosensors, called as nano sensors, can act as a reliable drug carrier [22].

Quantum dots (QDs) are tiny semiconductor particles a few nanometers in size, having optical and electronic properties that differ from larger LED particles. They are a central theme in nanotechnology [23]. When the quantum dots are illuminated by UV light, some of the electrons receive enough energy to break free from the atoms. This capabilityallows them to move around the nanoparticle, creating a conductance band in which electrons are free to move through a material and conduct electricity. When these electrons drop back into the outer orbit around the atom (the valence band), as illustrated in the following figure, they emit light. The color of that light depends on the energy difference between the conductance band and the valence band. In the language of materials science, nano scale semiconductor materials tightly confine either electrons or electron holes [24]. Quantum dots are sometimes referred to as artificial atoms, emphasizing their singularity, having bound, discrete electronic states, like naturally occurring atoms or molecules.

Quantum dots have properties intermediate between bulk semiconductors and discrete atoms or molecules. Their optoelectronic properties change as a function of both size and shape. Larger QDs of 5–6 nm diameters emit longer wavelengths, with colors such as orange or red. Smaller QDs (2–3 nm) emit shorter wavelengths, yielding colors like blue and green, although the specific colors and sizes vary depending on the exact composition of the QD [25]. The size of the nanoparticles plays a crucial role in biomedical applications like gene as well as drug delivery. Smaller the size of the nanoparticles, the easier would be its cellular and tissue uptake in cells. The internalization mechanism and uptake efficiency of nanoparticles by cells decrease with an increase in the size of the nanoparticles. The green synthesized nanoparticles are smaller in size and show less toxicity as compared to chemically synthesized ones. In comparison to chemically synthesized nanoparticles, green synthesized ZnO Nps show a higher controlling effect against various pathogens and high 17 antimicrobial and antibacterial activity [26]. Also, the green synthesized nanoparticles are more stable and their rate of synthesis is faster, hence making them more advantageous for the nano biomedical application. We make ZnO quantum dots with neem extract. Zinc and neem both have an antimicrobial effect. *Azadirachta indica*, commonly known as neem, is a tree in the mahogany family Meliaceae. It is one of two species in the genus Azadirachta. Green route synthesis is one of the most promising techniques for the synthesis of nanostructured materials being simple, environmentally safe (mild reaction conditions and no need for toxic chemicals) and inexpensive and can be produced in a large-scale process [23].

In the present work, a traditional and microwave-assisted green synthesis of ZnO using spinach extract was studied and the resulting ZnO nanoparticles were characterized using a scanning electron microscope (SEM) energy, dispersive X-ray spectroscopy (EDX), and X-ray diffraction spectroscopy (XRD), also the catalytic of the obtained ZnO nanoparticles is evaluated. Over the years, visible-light-driven semiconductors have gained a lot of interest because of their efficient photocatalytic performance and higher stability for the photodegradation of organic pollutants [27]. In the recent past, titanium dioxide was the best- known photocatalyst for the degradation of organic pollutants. However, due to its wide bandgap (3.2 eV), its photocatalytic performance is confined under natural sunlight [24– 27]. Until now, various approaches have been employed to enhance the photocatalytic performance of TiO_2 under visible light such as doping with the transition metal or non- metals [28–30], coupling with narrow bandgap semiconductors [31, 32], dye-sensitization [33, 34], etc. However, in certain cases, the functionalization of TiO_2 has resulted in self photodegradation, instability under visible light, and residual toxicity [35].

Materials and Method

The *Azadirachta Indica* (Neem) fresh leaves were collected and zinc acetate dehydrate $Zn(CH_3COO)_2.2H_2O$ was used in these studies. The clean and dried glassware were used in these studies for the synthesis of ZnO QDs. Fresh leaves of *Azadirachta Indica* (Neem) were washed thoroughly several times with distilled water. The extraction was prepared by boiling 10g leaves with 100ml of distilled water. The extract was boiled at different temperatures 20-degree, 40-degree, 60 degree, 80 degrees 100 degree. The resulting extract was then cooled at room temperature(20 degrees, 40-degree, 60 degree, 80 degrees 100degree) followed by filtration using 11μm filter paper. The obtained filtrate (5ml) was treated with an aqueous solution of freshly prepared 5ml of 1mM zinc acetate,3mM zinc acetate,5mM zinc acetate. The concentration of zinc acetate was optimized for the synthesis of ZnO QDs by varying to different values (1, 3 and 5 Mm) using 5ml *Azadirachta Indica* (Neem) leaf extract. To optimize the quantity of leaf extract for the synthesis of ZnO QDs, the leaf extract was varied from 1 ml to 8ml. As the temperature is also an important factor for the synthesis of ZnO QDs, the effect of the temperature was analyzed with a range from 20 to 100 degrees.

The photocatalytic degradation of the methyl orange was performed by using ZnO QDs, which is synthesized with neem extract. ZnO nanoparticles is a photocatalyst used UV illumination for various time interval and sunlight at 48 degrees MO dye (10ppm) solution was prepared in 100ml DI water and it was mixed with 0.3g of different kinds of synthesized ZnO. The suspension was equilibrated by stirring for 30 min to stabilize the absorption of MO dye over the surface of the photocatalyst, which is ZnO nanoparticles, before exposure to the light. The photocatalytic decomposition of MO was examined by measuring the absorbance regular time intervals by using the ultraviolet and visible (UV- Vis) spectrophotometer wavelength at 465 nm. Analytical samples were taken from the reaction suspension at regular time intervals for 10 minutes and were then analyzed for their absorption using a UV-Vis spectrophotometer.

Result and Discussion

Green synthesis of ZnO QDs was associated with the visual observation, the specified color transformation of the reaction mixture of zinc acetate, and the leaf extract of neem. Zinc acetate quantum dots were formed with color variation from dark brown to yellow. The characteristic color variation is due to the excitation of the localized SPR in the metal nanoparticles [36]. The mechanism for the green synthesis of ZnO QDs using the reaction mixture of neem extract and zinc acetate could be explained in the light where the activation stage involves the reduction of zinc ions ($Zn^{2+)}$ to zero-valent states using the reducing agents like flavones, quinines present in neem extract. The reduced zero-valent states atoms then converted to ZnO as a result of reaction with the dissolved oxygen (O_2) content of the reaction mixture followed by nucleation of the ZnO. In the second growth stage, the small adjacent ZnO QDs spontaneously unite into larger size particles with an enhancement in the thermodynamic stability of QDs. Finally, the third stage associated with the final shape of the ZnO QDs in which QDs attained the spherical shape with utmost energetically favorable confirmation, which is strongly governed by the ability of various phytochemicals of neem leaf extract to stabilize ZnO QDs by acting as capping agent. The capping agent stabilizes the QDs and prevents them from aggregation. The probable mechanism of neem leaf extract mediated ZnO QDs formed. UV-Vis absorption spectroscopy is used to characterize the quantum dots and as well as to see the stability and synthesis of nanoparticles in an aqueous suspension, where the nano ZnO reveals absorption in the range of 320-400 nm [37]. The present synthesized ZnO QDs showed a broad absorption band ranging from 315 to 350 nm with maximum peak appeared at 324nm, which corresponds to their SPR [38]. A characteristic peak of ZnO QDs that appeared at 324 nm could be responsible for the intrinsic bandgap absorption, where electron transitions occur from the valence band to the conduction band [39] [fig:1]

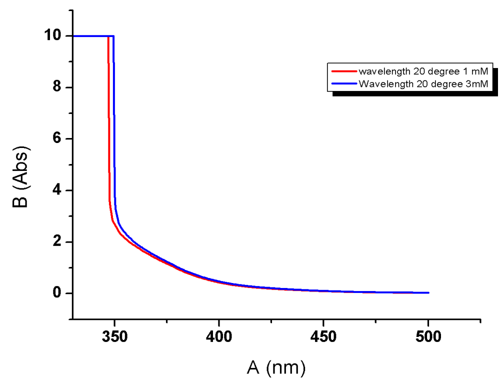


Fig:1 Detection of wavelength

The impact of zinc acetate concentration as a precursor for the synthesis of ZnO QDs was studied. Among varying concentrations of zinc acetate(1, 3, 5 mM), the maximum synthesis of ZnO QDs was recorded under a reaction mixture of 5ml of 5mM zinc acetate and 5ml of neem leaf extract. There was an increase in absorption in the SPR band at 324 nm as the concentration enhanced from 1 to 5 Mm that could be correlated with the increasing number density of ZnO QDs. In contrast, supplementation of zinc acetate at a concentration of 1 and 3 mM accompanied the broadening of absorption peak that signifying ZnO QDs synthesis with relatively larger particle size.[40]. The effect of temperature on the synthesis of ZnO QDs is another significant parameter influencing the synthesis of ZnO QDs in the reaction mixture of neem leaf extract and zinc acetate. Normally, an increase in temperature enhances the rate of response as well as the effectiveness of NPS production. It is presumed that increasing the temperature enhances the rate of nucleation[41]. The effect of temperature(ranging from 20 to 100 degree at regular interval of 20 degrees) on the synthesis of ZnO QDs was an investigation with the optimized concentration of 7ml leaf extract and 5 ml precursor and it was observed that as the temperature increase from 20 degrees to 80 degrees the intensity of absorbance band increases progressively, which point towards enhanced rate of reduction of zinc ions into ZnO QDs, where the increased rate of reduction was resulting at a higher temperature because of the expenditure of zinc ions in the configuration of nuclei while the secondary reduction was blocked up on the surface performed nuclei [fig 2- 5]

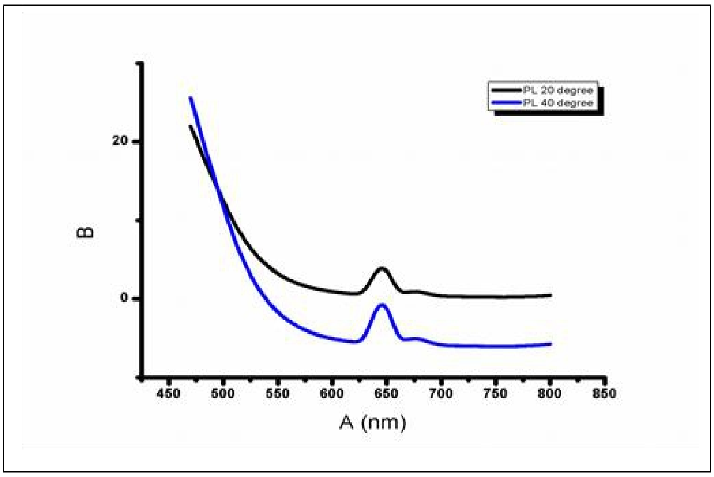


Fig:2 Measurement of PL

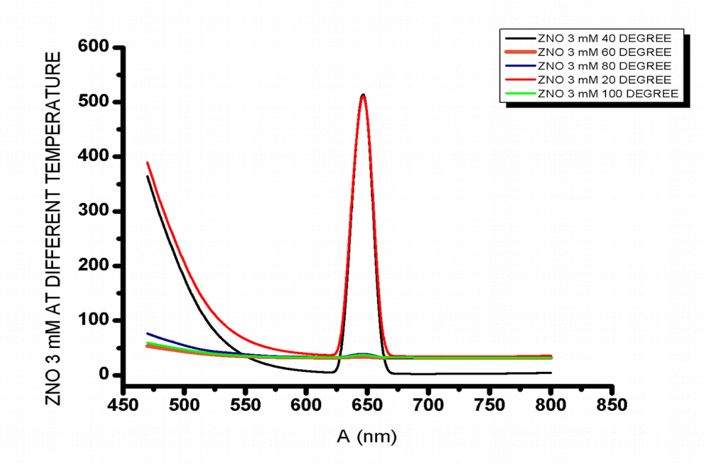


Fig: 3 At different temperature concentration (3Mm)

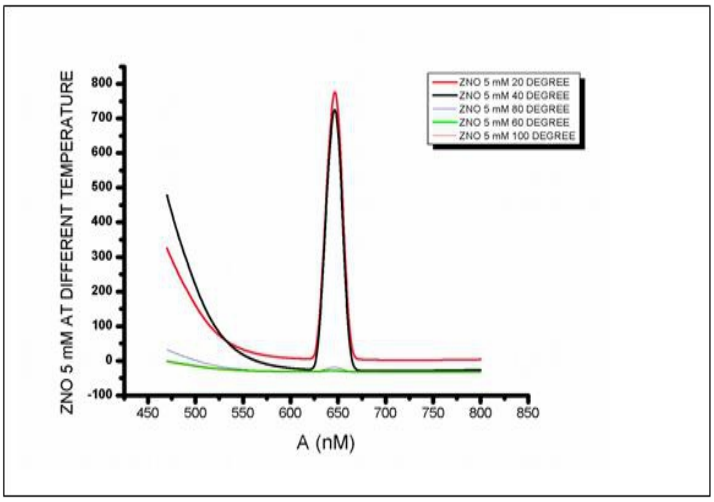


Fig 4: At different temperature concentration (5Mm)

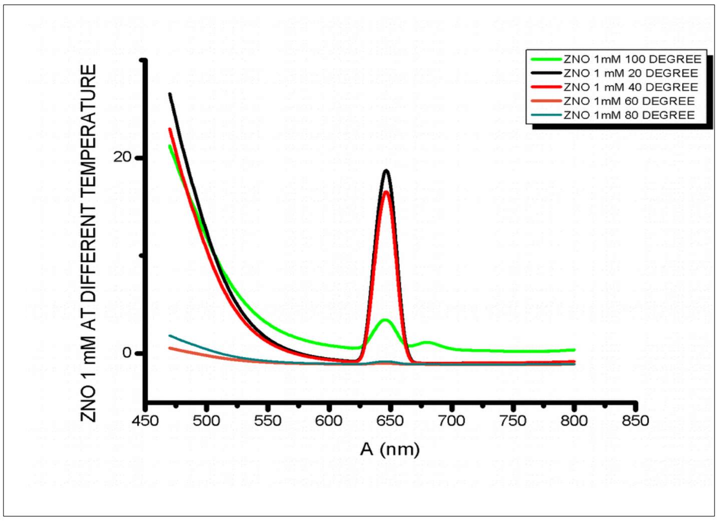


Fig: 5 At different temperature concentration (1Mm)

A time scan was performed with MO and ZnO QDs to visualized the photo catalytic degradation of methyl orange. It was observed that with increasing the catalyst amount, the photo degradation increases. However, when the amount of catalysis exceeds the optimum amount (0.3g), photo degradation efficiency decreases. This could be attributed to the fact that the number of active sites increased as the amount of the catalyst increased [Fig: 6].

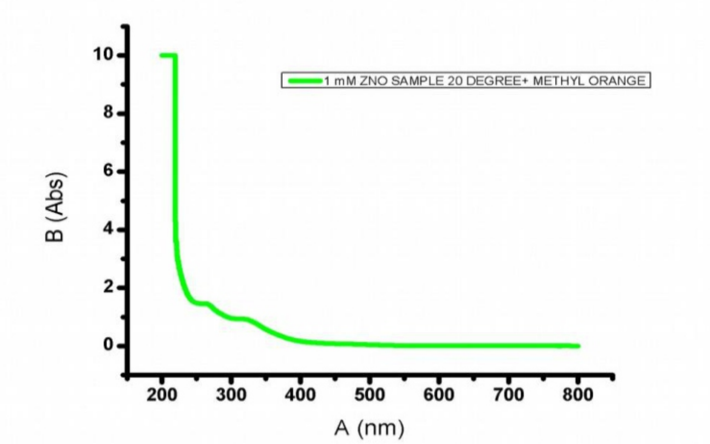
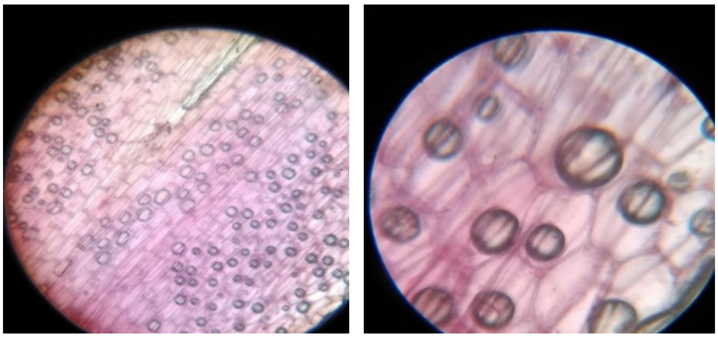


Fig: 6 photo catalytic activity of ZnO with MO

Plasmolysis was performed with ZnO QDs over rheo leaf. It was noticed that with different concentrations of ZnO QDs the effect of plasmolysis varies. With 1 Mm of ZnO QDs, plasmolysis for 3mM we see less plasmolysis, and for 5mM we saw less plasmolysis. Plasmolysis was performed with ZnO QDs over rheo leaf. It was noticed that with different concentration of ZnO QDs the effect of plasmolysis varies with 1Mm of ZnO QDs, see plasmolysis , for 3mM we see less plasmolysis and for 5mM we saw more less plasmolysis.



10x 45x

Fig: 7 Plasmolysis control

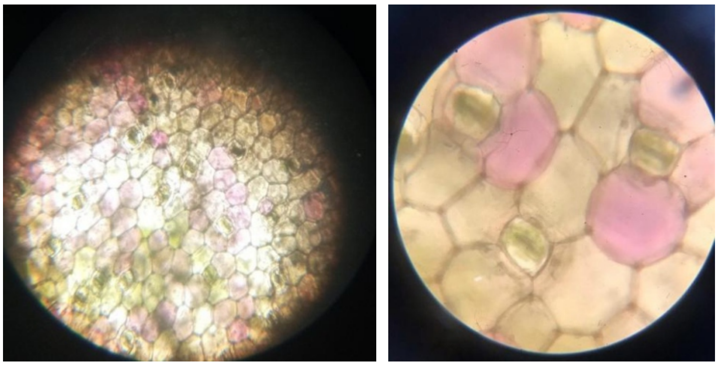


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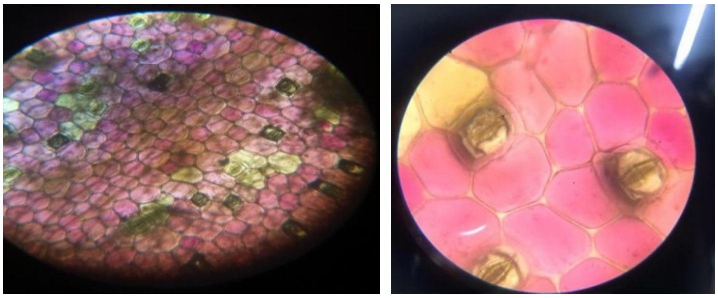
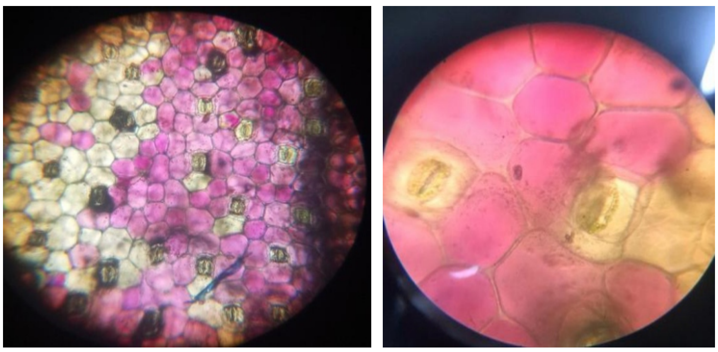
Fig:8 Plasmolysis Treated with Neem Quantum Dots (1mM)



10x 45x

Fig :9 Plasmolysis treated with quantum dots (3mm)



10x 45x

Fig: 10 Plasmolysis treated with neem quantum dots (5mm)

Conclusion

The present biogenic approach for the synthesis of ZnO QDs using neem leaf extract is fast and cost-effective. No chemical reagent was used in this approach that consequently allows the bioprocess with the benefit of being environmentally friendly. The possible mechanism for the formation of ZnO QDs from zinc acetate involves the phytochemicals of neem leaf extract that might be reacting with Zn^{2+} ions and acting as reducing, capping, and stabilizing agents. An important potential advantage of the current technique of synthesizing QDs is that they are quite stable with uniform spherical shape and size in the range of 3-9 nm. These ZnO QDs have significant antimicrobial activity. The green synthesis of ZnO QDs is still in its infancy and intensifies research required to be stressed on the mechanism of QDs production that may result in fine-tuning of the process eventually leading to the formation of QDs with stringent control over the shape and size factors.

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