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An Investigation On: Bio-Pesticide, Photo-Catalysis And Anti-Bacterial Activity Of Green Synthesized Seo_2 Nanoparticles

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ABSTRACT

Selenium oxide (SeO_2) nano photo catalyst was prepared by a facile biological method where reduction of Na_2SeO_3 occurs by plumeria pudica leaves extract and characterized by XRD, scanning electron microscope, Energy dispersive X-ray analysis and IR spectroscopy. The XRD showed that SeO_2 is amorphous and has an average crystallite size of 99.20 nm. The biopesticide activity of synthesized was studied against rice and moong dal pests to get 100% mortality by 72 hours in rice and 20% mortality in moong dal. The photocatalytic activity of the sample was evaluated by the degradation of methyl orange and crystal violet in aqueous solutions under ultraviolet radiation. High efficiency was achieved at a very low concentration of 0.003g of SeO_2 nanoparticle. Evidently, the anti-bacterial activity of SeO_2against E. coli is considerable compared with the standard drug.

Keywords: Selenium oxide (SeO_2), plumeria pudica, biopesticide activity, photocatalytic activity, anti-bacterial activity.

1. INTRODUCTION

In agriculture, insects, plants pathogens, and weeds are major problems where significant losses of yield [30% - 40%] can occur. A number of methods have been developed for preventing crop damages from pests, in particular where the most successful outcome has been achieved through pesticides application. It was recently estimated that globally around 3 million tons of pesticides are applied on crop fields every year [1]. To achieve the desired level of pest within a given period, pesticides are applied repeatedly and

indiscriminately. Therefore, in reality the amount of pesticides greatly exceeds the amount actually required [2].

Biopesticides have attracted much interest in the research community and have been recommended as potentially good alternatives to synthetic pesticides. Biogenic sources and the green synthetic route of these nano-particulates further ensure their eco-friendly applications, particularly when the nano-biopesticides are prepared through adsorption, attachment, entrapment, and encapsulation techniques [3,4]. To some extent, nanoparticles themselves can nullify agricultural pests. A biopesticides containing a nanocomposite matrix and an emulsion based, nanodroplet-oriented formulations can increase biopesticides molecule solubility and stability [5,6].

Selenium nanoparticles (SeO_2NPs) have the potential to be used for various applications. Therefore, they have attracted more attention in recent years and several synthesis methods have been exploited [7-9]. Green synthesis using plant extracts has gained popularity because it requires non-toxic solvents and moderate temperatures [10]. Furthermore, it is environmentally friendly and uses a reducing agent that is easily accessible and biodegradable [11]. In this paper, we present the capability of plant materials for the biosynthesis of SeO_2NPs.

Selenium is most highly exploited for its biological activity but its capability as a photocatalyst is very less studied [12,13]. Semiconductor Se nano photocatalyst can use the available source of ultraviolet (UV) radiation from sunlight or artificial light and conduct a chemical reaction to degrade the organic pollutant present in both the liquid and gaseous phases [14-16]. However, the need for UV radiation in photodegradation processes has limited both the practicality and environmental benefits on industrially relevant scales [17]. Hence, the present work focuses on study of photodegradation of Methyl orange and Crystal violet dye, biopesticides and anti-bacterial properties of low-cost biological synthesized SeO_2 nanoparticle.

2. Materials and methodology

2.1 Collection of plant materials

The leaves of plumeria pudica were collected from the wild growing tree in the botanical garden Of Pooja Bhagavat Memorial Mahajana PG Center, Metagalli, Mysore, India. Identification and confirmation were performed a qualified taxonomist [18]. The collected plant material was made thoroughly free from any foreign organic matter. (**Fig.1**)

Fig.1: Plumeria pudica leaves

2.2 Preparation of leaf extract

The collected plumeria pudica leaves were washed with double distilled water and dried in hot air oven at 40° C.Subsequently, leaves are crushed into the fine powder by using pestle and mortar. Weigh out 10g of plumeria pudica leaves powder in a 250 mL round bottom flask added 100mL distilled water and kept for stirring at 80° C for about 60min on heating mantle. After cooling to the room temperature, the homogenate was centrifuged at 10,000 rpm for 15 min and then filtered through Whatmann No. 1 filter paper to remove the debris. The extract was stored in the refrigerator until use[19](**Fig.2**).

Crushing Stirring Extraction

Fig.2: Plumeria pudica leaves extract preparation

2.3 Synthesis of Selenium nanoparticles

Take a 100mL of aqueous leaves extract in a beaker, add 0.216g of Na_2SeO_3 for the synthesis of Se-nanoparticles. Kept for the stirring with certain time intervals and observed for color change at 2hours, 4hours, 6hours, 24hours and 48hours. The color change from brown to light green, with reaction mixture, indicates the formation of Se-nanoparticle, which is further confirmed by UV-Visible spectroscopy. (**Fig.3**)

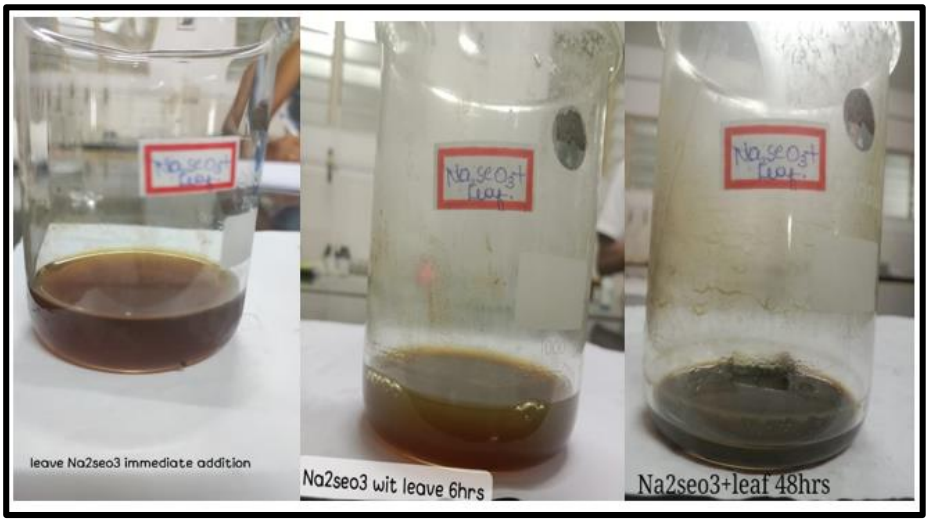


Fig.3: Extraction of selenium nanoparticle: (a) After immediate addition, **(b)** After 6hrs and **(c)**After 48hrs

3.Characterization

FT-IR spectra were recorded using JASCO FT-IR: 460 plus with wave number ranging from 400 to 4000 cm^{-1}. The optical absorption spectra have been observed by UV–Visible spectrophotometer at room temperature with JASCOUV-VIS spectrophotometer. SEM images of the samples were recorded on ESEM Quanta-200FEI-Netherlands, A scanning electron microscope. The reduced SeO_2NPs powder was coated on a glass substrate and the Energy Dispersive X-ray diffraction measurement were carried out by using a powder Energy Dispersive X-ray (PAN analytical BV model) instrument operating at a voltage of 40kV and current of 30mA. The output was recorded in the form of a graph with 2θ on x-axis and then intensity on y-axis. For measuring the absorption characteristics, nanopowder is first dispersed in isopropyl alcohol (IPA) and then taken in a quartz cuvette of path length of 10 mm. The powder X-ray diffraction patterns were recorded using Rigaku Miniflex II desktop X-ray diffractometer (Cu-K radiation = 1.54˚A) employing a scan rate of 0.02˚/s range from0˚to 60˚.

4. Results and Discussions:

Maximum absorbance peak was observed at670nm indicating that the formation of SeO_2NPs as a result of reduction of Se^{4+} ions presents in the aqueous Na_2SeO_3 solution (**Fig 4**).

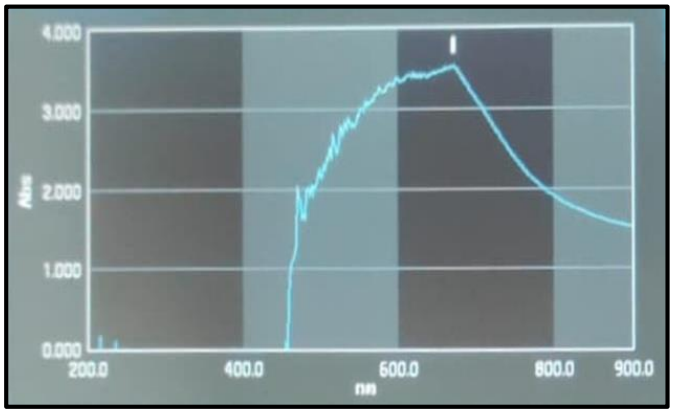


Fig.4: UV-Vis spectra of SeO_2NPs.

Fourier transform-Infrared (FT-IR) analysis was performed to identify the possible biomolecules responsible for the reduction of the Se^{4+} ions and capping of the reduced SeO_2NPs synthesized using Plumeria leaves extract [20]. The strong IR bonds were observed at 3373, 1638, and 650 cm^{-1}. The bands which appeared at 3373cm^{-1} corresponds to N-H, –OH stretching and aliphatic C-H stretching, respectively. The IR bands observed at 1638cm^{-1} may be ascribed to –C-O and –C-O-C stretching modes, respectively [21,22]. The strong band recorded at 650cm^{-1} in the spectra evident the presence of Se-O bond. (**Fig.5**)

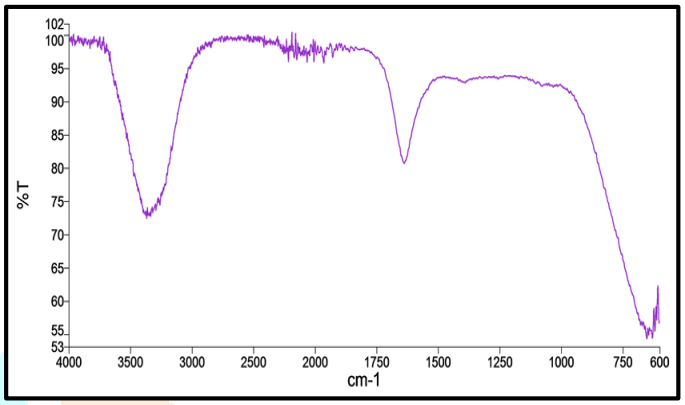


Fig.5: Fourier Transform-Infrared spectra of SeO_2NPs

SEM analysis shows the uniformly distributed SeO_2NPs on the surface of the cells. However, it does not indicate that all the NPs are bound to the surface of the cells, because those dispersing in the solution may also deposit onto the surface of the cells. The SEM image has shown separate SeO_2NPs as well as particle agglomeration. The SEM study indicates that the particle size is irregular and shape of the particles is spherical in morphology of size ranging with an average size of 99.20 nm. SEM images of synthesized SeO_2NPs from Plumeria leaves extracts are shown in (**Fig.6)**

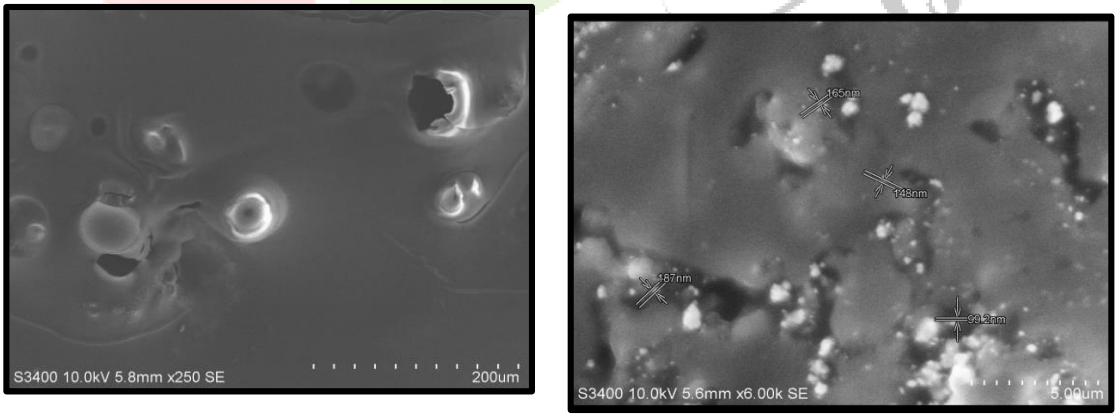


Fig.6: Scanning SEM image of nano particles

In the EDX spectrum (**Fig.7**) of SeO_2NPs, in addition to oxygen and selenium, carbon and sodium elements were also observed, which indicates the presence of some impurities due to the biological synthesis of nanoparticles.

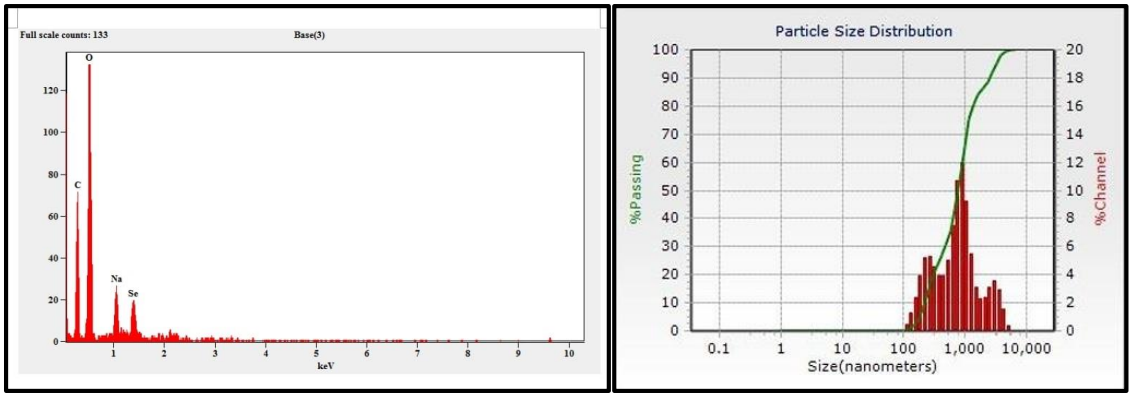


Fig.7: (a) Energy Dispersive X-ray Analysis Spectrum and **(b)** Size distribution of dispersed SeO_2NPs using DLS

The average size of the SeO_2NPs was observed as 306nm and -29.4mv of zeta potential, it is recommended that the outer area of the nanoparticles is negatively charged and diffused in the solution. The negative value indicates the repulsion between the nanoparticles and also it represents that the nanoparticles were stable.

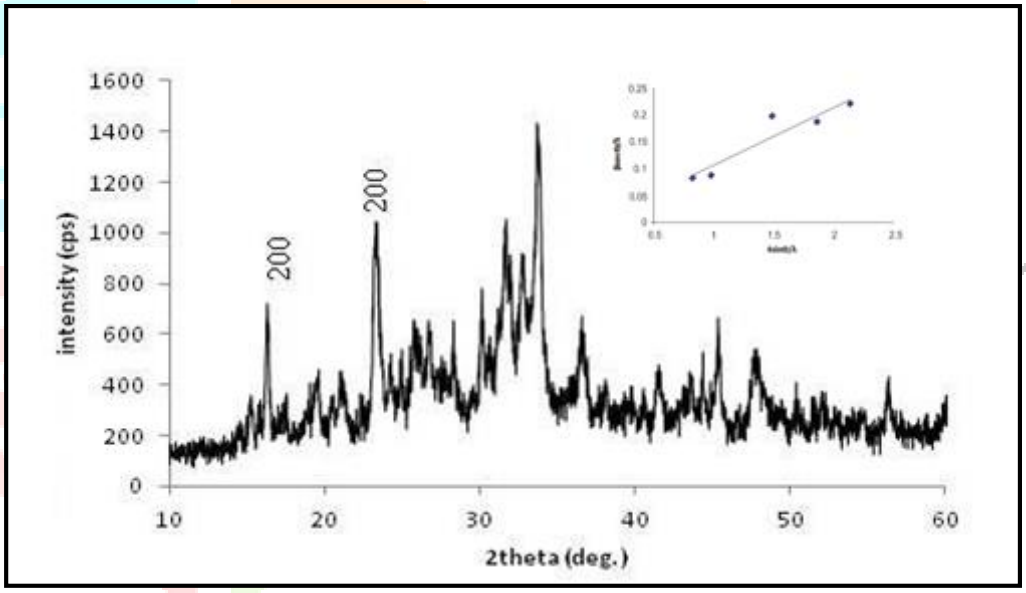
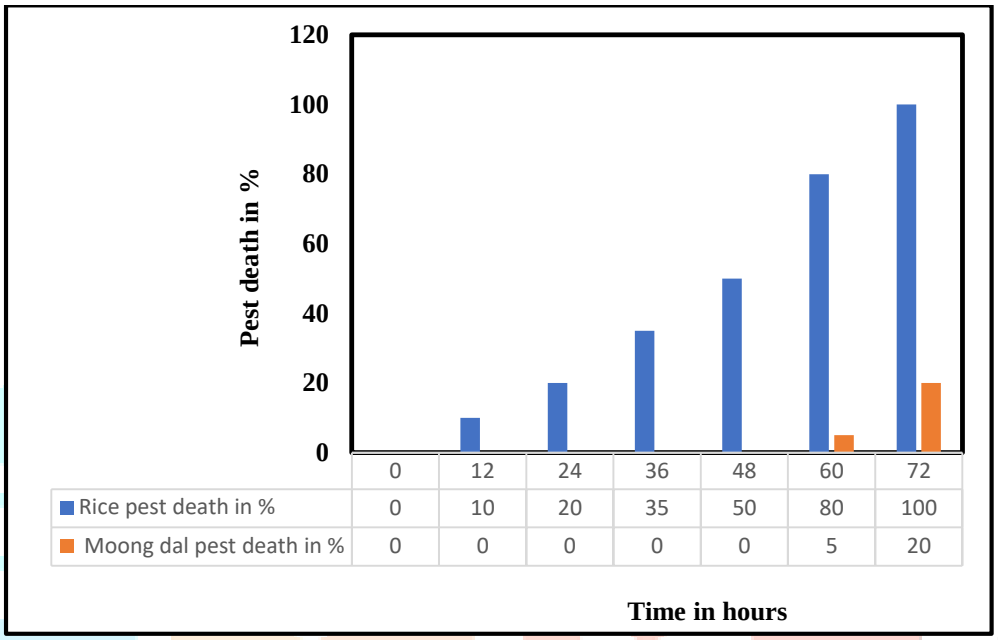


Fig. 8: XRD of SeO_2. Inset: W-H plot

The X-ray diffractogram (Fig.8) of the synthesized SeO_2 revealed characteristic diffraction peaks at 2Θ values 16.58 and 24.36 appearing due to reflection from (200) and (200) planes corresponding to the stable amorphous, hexogonal phase (JCPDS no. 65-1876). The average crystallite size of the nanoparticles as calculated using Debye Scherrer equation is 99.20nm. The angle strain is measured to be 2.046×10^{-3}. It is further confirmed by Willianson Haal plot which is a plot of $4\sin\Theta/\lambda$ vs. $\beta\cos\Theta/\lambda$ where intercept gives the crystallite size and slope gives the angle strain.

4.1Biopesticide activity of selenium nanoparticle:

The pesticide activity of SeO_2NPs was analyzed by mixing 20 number pests to each 20g of moong dal and 20g of rice. To each cereal add 2 mL of SeO_2NPs solution. The container is kept for observation for about 72 hours; the death rate was monitor for every 12 hours. After 72 hours of time period, 100 % pests were dead in rice whereas only 20% pests were dead in moong dal, which indicating that SeO_2NPs shows pesticide activity and which is more effective for the rice pest than moong dal pests.



	0	12	24	36	48	60	72
Rice pest death in %	0	10	20	35	50	80	100
Moong dal pest death in %	0	0	0	0	0	5	20

Fig.9: Biopesticide activity of SeO_2NPs on rice and moong dal pests

4.2 Photo degradation of dye using SeO_2NPs:

To assess the photocatalytic efficiency of the prepared nano particles, photodegradation experiments were carried out using Methyl orange and Crystal violet dye as substrate SeO_2NPs as catalyst. A calculated quantity of the catalyst was added to the dye solution, stirred in the dark for 1 min to establish adsorption/desorption equilibrium between the dye and nano particle molecules and then illuminated under 8W UV source to induce a photochemical reaction [23, 24]. Aliquots were taken at an interval of 5 min and absorbance read at 520nm for methyl orange and 590mn for crystal violet which is determined by Elico SL 171 mini spectrometer.

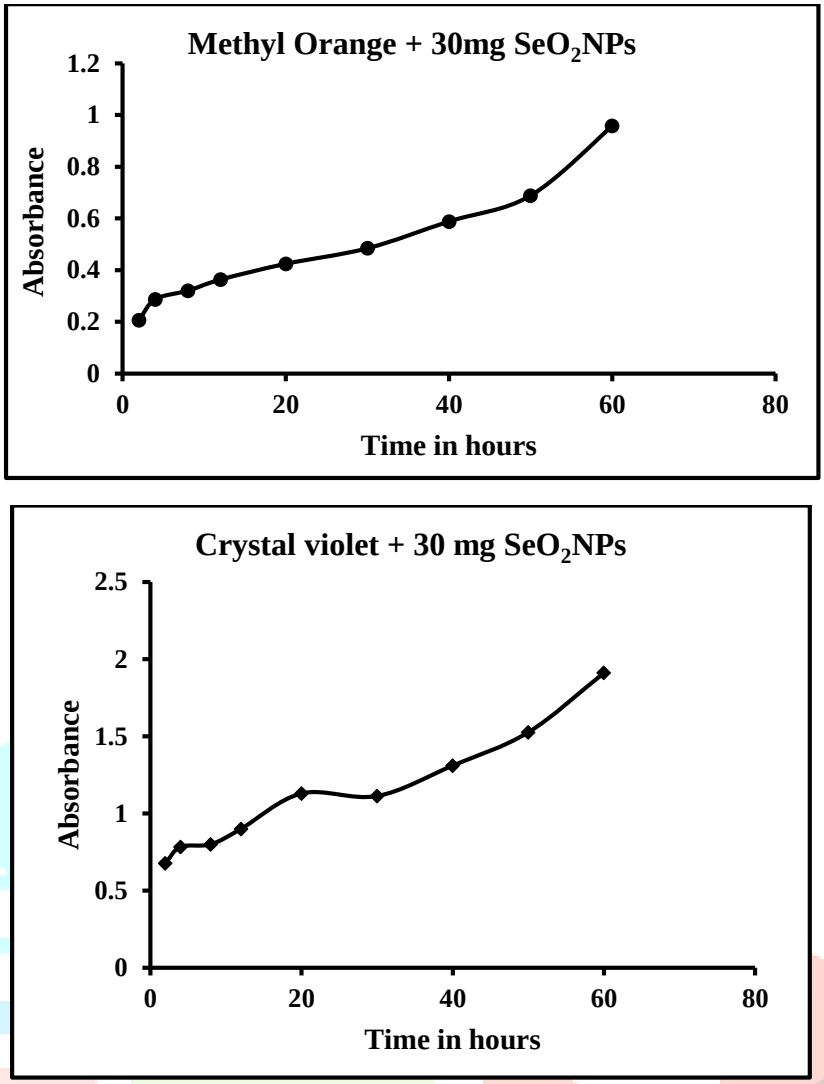


Fig.10: Plot of absorbance v/s time for photodegradation of dye by SeO_2NPs

4.3 Anti-bacterial activities:

The antibacterial activities of SeO_2 NPs against E. coli were determined by using the National Committee for Laboratory Standards (CSLI) approved for both macro and microdilution methods [25]. Paper discs (Whatman filter paper, diameter: 4mm, thickness: 2mm) were soaked in different dilutions of the nanoparticle solutions, kept aside for 1 minute on a sterile micro slide for avoiding the dripping. Nutrient agar plates were inoculated with E. coli culture in an aseptic condition, the filter discs were placed accordingly, and plates were labelled accordingly. Sterile water treated as control. The Selenium nanoparticles showed significant anti-bacterial property as evidenced with an inhibition zone with diameter 3.5 cm.

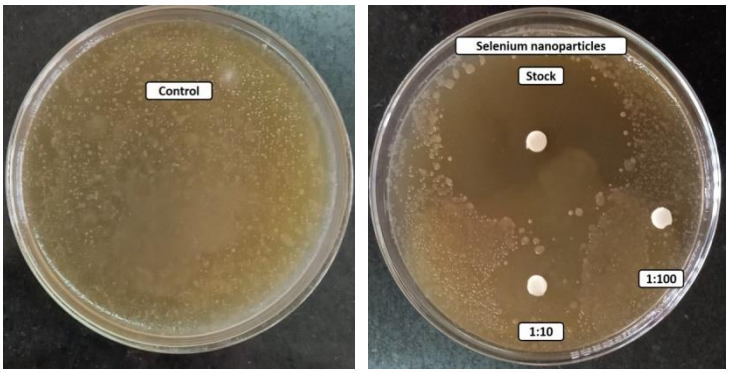


Fig.11: Anti-bacterial activity of SeO_2NPs against different bacterial strains in comparison with the standard E. coli (+ve control).

5. Conclusion:

SeO_2NPs were successively synthesized by reduction with plumeria pudica leaves, an environmentally friendly biological method. Structural studies have confirmed the spherical morphology of size ranging with an average size of 99.20 nm. 100% bio pesticidal activity was achieved in mere 72h making it a promising candidate for commercial studies. Photodegradation by these semiconductors offers a green technology for the removal of hazardous dyes present in the waste waters. Also, the synthesized nanoparticles are capable of entering into the bacterial cell, therefore inhibit the cell growth and hence confirm its role as a potent antibacterial agent. Therefore, the simple, cost-effective, and eco-friendly synthetic method allows for its application in large scale production.

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