A Review on Synthesis of ZnO Nanoparticles and Their Multifunctional Roles.

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***Abstract* -ZnO nanoparticle has been widely studied as photocatalyst. ZnO nanoparticle has greater potential due to their chemical stability, non-toxicity and high reactivity. Nanoparticles can be synthesized in many ways including Chemical Vapour deposition, Co-precipitation, Combustion, Sol-Gel, Hydrothermal, Solvothermal, Microwave Assisted and Green synthesis. In this review, some of the Top-down approach and Bottom-up approach methods for synthesis of nanoparticles and its antimicrobial assays are discussed. The purpose of the paper is to review optimum conditions required for photodegradation of industrial effluent like Dyes using ZnO nanoparticle synthesized by Green synthesis method.**

***Index Terms* – Photo catalysis, Antimicrobial Assays, ZnO Nanoparticles, Dye concentration, Bandgap Energy.**

I. INTRODUCTION

Study on nanoparticles increasing rapidly due to its unique properties like change in surface to volume ratio, optical properties, energy band gap, photocatalytic reaction, particle size, particle shapes and so on. ZnO nanoparticle synthesized by green synthesis route has been extensively using semiconductor nanoparticle because of its non-toxicity and stability property. In this review, different types of nanoparticle synthesis methods are discussed. Study of ZnO nanoparticle synthesized by green synthesis method can act as photocatalysts. The study of optimum conditions is trivial for synthesis methodology as these factors has major impact on the characteristics of nanoparticles. In these review, some of the conditions like energy band gap, pH, Dye concentration, calcinations temperature and antimicrobial assays are discussed.

II. NANOPARTICLE SYNTHESIS METHODOLOGY

1 Top-down approach

1.1 Ball milling method

Ball milling is a mechanical technique to reduce particle size to nanoscale by physical crushing of materials between balls and walls of containers. The selection of material of balls, wet/dry collision (1) , the grinding time, coating material to the container, atmosphere inertness, reactivity between the balls and materials, the tear and wear of balls , rotational speed control are to be in optimal condition. This is cost-effective hence most of the industries use method for industrial Energy storage application (2).

1.2 Lithography

Nanolithography is imprinting and etching pattern on nonoscale to build small structures. Lithography method used in many industrial applications. In-organic Metal oxide based photo resists better due to their high chemical reactivity under Extreme Ultraviolet Lithography (EUL) which intensively used in Microelectronics industries for making precise patterning (3). Acrylin Resin embedded with TiO2 nanoparticle act as Photoresist which shows high transparency of 98% for visible light and low haze of 0.05% which gets solidified and imprinted after get exposed to UV radiation can be used in optical industries (4) . This has similar efficiency to Electron beam Lithography which is expensive in usage. The efficiency of Bioactive Glass (BG) scaffold fabricated by Two Photon Lithography has used in medical fields as to promote new bone formation (5). High quality SHINERS substrate can be obtained by combining Lithography techniques for nanoparticle synthesis and

Atomic layer deposition (ALD) techniques for sensors purpose (6)

1.3 Electro spinning Method

Electrospinning is a nanofibre manufacturing technique where the diameter of the nanofibre produced is in the range of nanoscale. Polymer solution filled in syringe where high voltage is applied between the needle and collecter plate to derive taylor shape droplet (7). Equation set up by experimental data has been used to derive optimal conditions like fiber diameter, surface area of fiber, flow rate, distance between collector plat and needle. The errors can resolve by Machine learning (ML) (8)

Applications: Nanofibre produced by Electrospinning has many applications in industries. This has been utilizing in medical industries for tissue engineering (9), biosensors application, treatments and drug delivery systems. Hybrid nanofibres improve the biomedical applications (10). Noble Metal based nanofibre produced by electrospinning have high efficiency which utilized for water splitting applications (11)

2 Bottom-up approach

2.1 Sol-Gel method

Sol- Gel method is a wet technique that converts the precursor salt to nanoparticle by Hydrolysis and Condensation. Hydrolysis converts the precursor salt to metal alkaxide whereas condensation method converts to M-O-M bond (12). The selection and amount of precursor, the type of solvent, the solubility of precursor, pH, temperature, critical point of temperature- pressure diagram to be optimized in the synthesis process.

2.2 Biological (Green) Synthesis

Nanoparticles can be synthesized by plant parts like root, stem, leaves, seeds. The nanoparticle can be synthesized from microorganisms like fungi, bacteria, algae and from protein, sugars and enzymes. This method is non-toxic as it doesn’t involve any hazardous chemicals for the manufacturing of nanoparticles.

ZnO nanoparticle for Photocatalysis

Photocatalysis is a process which accelerates the rate of reaction. ZnO nanoparticles can acts like capping or stabilizing agent in photocatalysis process. The energy band gap of ZnO Nanoparticle is 3.37ev which corresponds to the UV light energy, therefore ZnO nanoparticles can act as photocatalyst under UV radiation. But to work on visible light region, the energy band gap of ZnO nanoparticle should be reduced. This can be possible with doping it with organic or inorganic or metallic compound such that the energy band gap should associate with visible light region. Therefore many scientific researchers were carried to decrease energy band gap by choosing appropriate condition.

The aim is to produce large number of electron hole pair by the absorption of visible light .This can be derived by doping process. The more concentration of free charge particle can undergo redox reaction to produce free radicals like OH+ and O+. These free radicals interact with dyes / industrial effluents and degrade them to H2O and CO2 as in the figure 1. The procedure should involve a delay in the recombination of electron hole pair so that more concentration of free radicals is available to degrade dyes present in the aqueous solution.

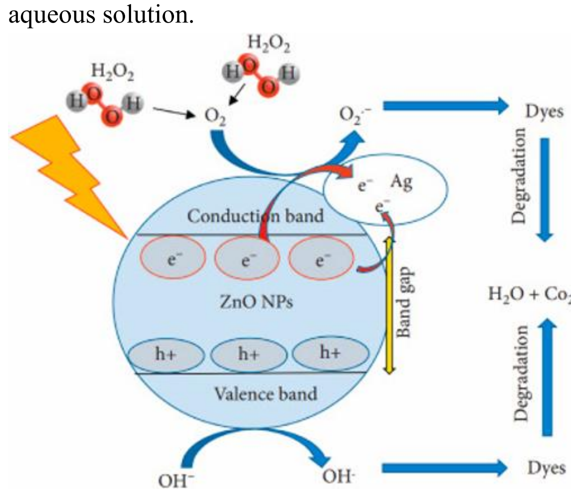

Fig.1.Schematic representation of Photocatalysis (13)

III. FACTORS AFFECTING ZNO NANOPARTICLE PHOTOCATALYTIC PROCESS

Size

ZnO nanoparticles can act as photocatalyst under sun light exposure (14). Calcination temperature is one of the factors which alter the crystal size. Calcination temperature varied as 500 °C, 600 °C, 700 °C, 800 °C, 900 °C and 1000 °C (15). Result shown that the crystalline size increases with increasing the calcinations temperature from 500 °C to 700 °C because of coalescence process. The shift in crystal size from 700 °C to 1000 °C calcinations temperature is not so incremental due to agglomeration of particles. Figure 2 shows the XRD pattern and study of crystallinity variation with respect to calcinations temperation (15). Saad Algarni, Tahani, et al. (16) studied X-ray diffraction pattern as in figure (3) and used Debye Scherer's formula to determine the crystal size of the biosynthesized ZnO nanoparticle from Rosmarinus officinalis leaf extract at different synthesizing temperature. Temperature at 80 °C shows smaller crystallite size compared to synthesizing temperature of 180°C.

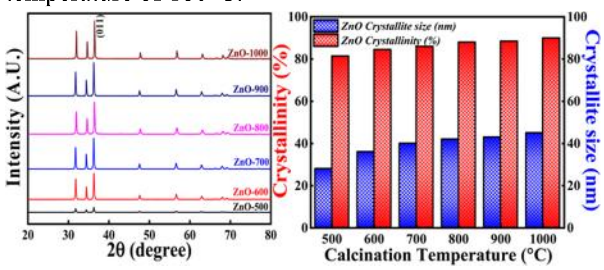

Fig.2. XRD Pattern and study of Crystallinity versus Calcination Temperature.

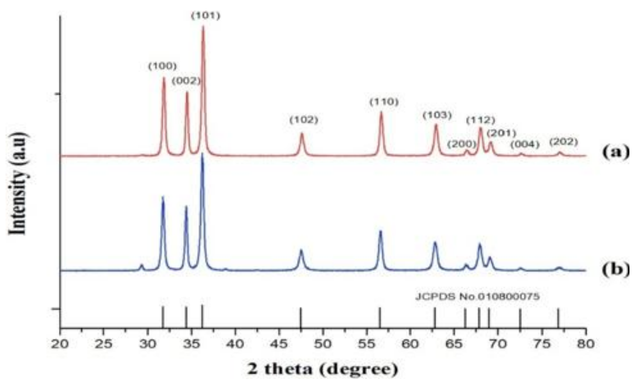

Fig.3. X-ray diffraction pattern of ZnO nanoparticle prepared at 80 °C (a) and 180 °C (b)

IV. BAND ENERGY

A.K.M. Ahsanul Habib et al (17) investigated the photocatalytic activity of (Ni–Mn) co-doped ZnO nanopaticle synthesized using green route using orange peel extract. The UV-Visible Spectroscopy analysis as in Figure (4) revealed the wurtzite structure and energy band gap has reduced by increasing the doping concentration. Deniz Ulukuş et al. (18) investigated the photocatalytic degradation of dyes by green synthesis using Se@Ag/AgO, Se@ZnO and Se@Ag/AgO-ZnO nanocomposite . Investigator used Pinus nigra pollen extract for reduction. UV–Vis Spectroscopy analysis as in Figure (5) revealed that Se doped ZnO showed energy band gap 2.62ev. The energy band gap of Se doped ZnO is less than Se doped Ag/AgO and Se doped Ag/AgO-ZnO.

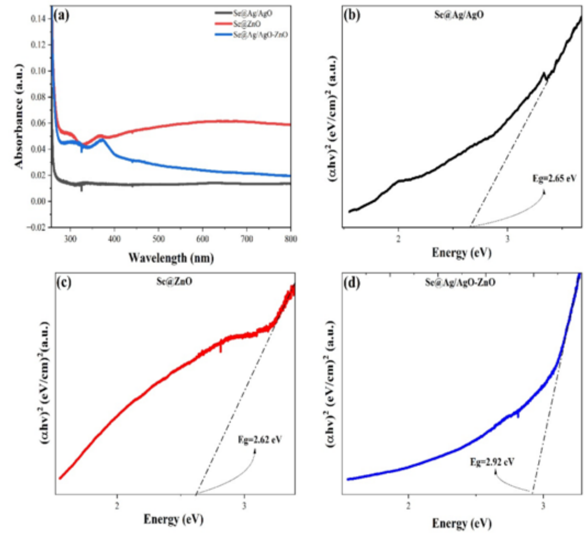

Fig.4. UV-Visible Plot and Tauc plot of pure, 1%, 2% and 3% Ni-Mn to determine energy band gap (17)

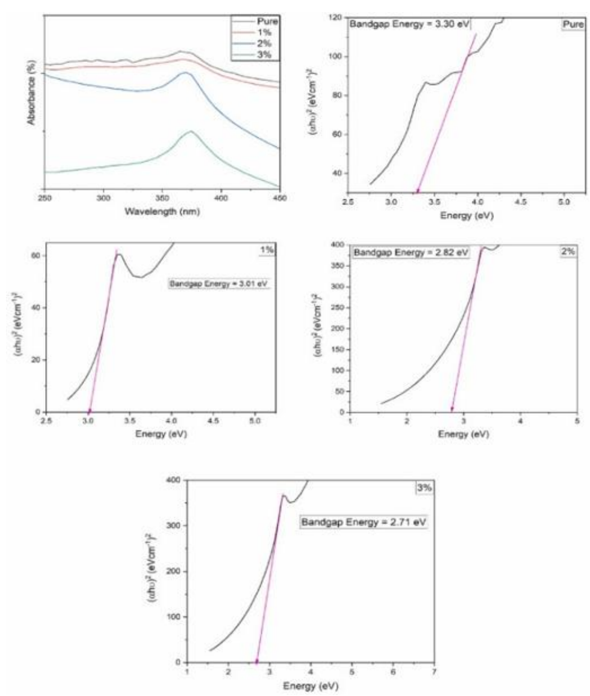

Fig.5 (a) UV-Visible plot of Se@Ag/AgO, Se@ZnO and Se@Ag/AgO-ZnO nanoparticles (b) energy band gap of Se@Ag/AgO nanocomposites (c) energy band gap of Se@ZnO nanocomposites (d) energy band gap of Se@Ag/AgO-ZnO

V. PH - POINT OF ZERO CHARGE (PZC)

The selection of pH of the Dye solution controls the surface charge polarity of photocatalyst. The Point of zero charge is the pH level below which the surface of photocatalyst become positively charged and above acts as negatively charge. This is the point in which the charge on the photocatalyst is neutral. Therefore, based on the charge of Dye, the pH of the solution should vary. Amad Nori Abdulqodus et al. (19) investigated the SEM analysis as in Figure 6 in which ZnO nanoparticle synthesized by aqueous leaf extract of Allium Caliphalum Wendelbow at different pH values of pH 7, pH 8, pH 9, pH 10, pH 11 and pH 12. Study revealed that the size of the Nanoparticle is smaller in size for pH 8. They specify that regulated Zn^{2+} and OH^- ions are at pH 8. pH higher than 8 results in excess of oH- ions.

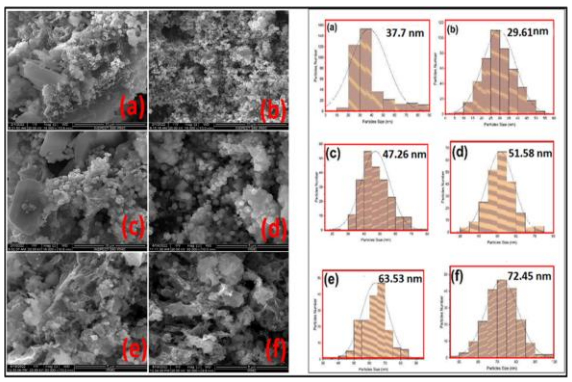

Fig.6. SEM analyses at (a) pH 7, (b) pH 8, (c) pH 9, (d) pH 10, (e) pH 11, (f) pH 12 of green synthesized ZnO nanoparticle. Frequency histogram at different pH values.

VI. DYE CONCENTRATION

Optimum Dye concentration is needed for effective dye degradation. Ansuman Nayak et al. (20) investigated by varying different concentration of Congo Red dye on ZnO-T and ZnO –C. Both adsorbent reported 40mg/L is optimum concentration as increase in concentration results in decrease in degradation efficiency due to lesser active sites for adsorption.

VII. PHOTOCATALYSIS REACTION TIME

The photocatalysis reaction time was investigated by Dhatwalia, J.et al. (21) as Methylene Blue Dye was degraded by utilizing Rubus ellipticus fruit extract. 17.5% of dye has been degraded within 60 minutes and 72.7% by 180 minutes as shown in figure 7. Along with that the solution has been stirred in the dark for 30 minutes to maintain equilibrium between ZnO nanoparticle and dye for adsorption and desorption (21). Photocatalysis time also depends on the Dye used for degradation. Babita Tripathi et al. (22) investigated three dyes namely methylene blue (MB), rhodamine B (RhB), and crystal violet (CV) for degradation using ZnO nanoparticles by two Saffron Flower extract as shown in figure 8. Degradation rate of CV is greater than MB and RhB .RhB involves stepwise N-deethylation which results in slower degradation (22).

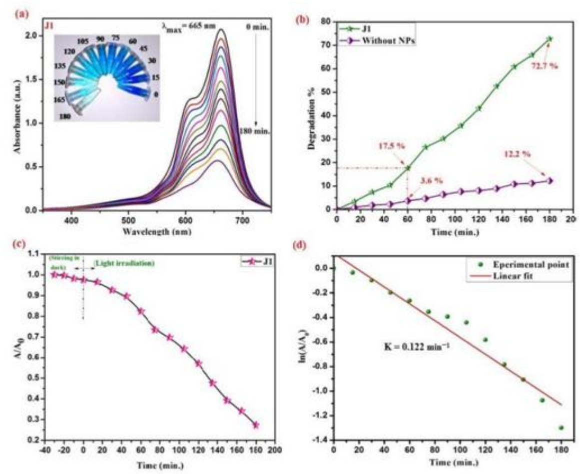

Fig.7. Photocatalytic dye degradation analysis (a) Absorption spectrum of MB dye with decoloration analysis; (b) Degradation percentage of dye at different time scale (c) Absorption analysis in dark and sunlight (d) Kinetic study of dye degradation reaction.

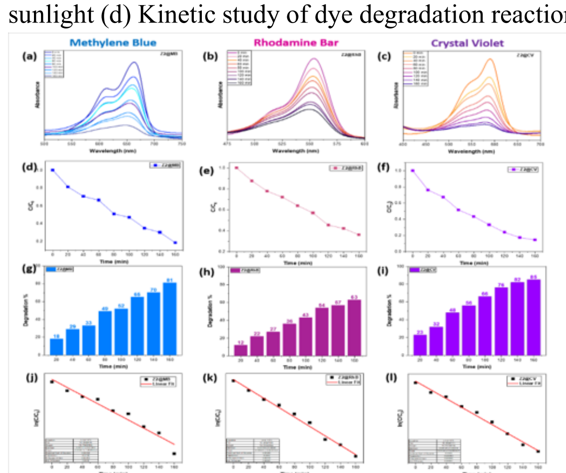

Fig.8. (a-c) UV Visible Absorption spectra of Methylene Blue, Rhodamine Bar and Crystal Violet; (d-f) plot of C/C 0 vs. time of MB, RhB and CV; (g-i) corresponding plot of percentage of degradation vs. time; (j-l) plot of pseudo-first order kinetics.

VIII. PRECURSORS

Selection of Precursor is one of the criteria in the study of Photocatalytic degradation. Verma, Lahur Mani, et al investigated the synthesis of ZnO nanoparticle by Sugarcane press mud by using three different precursors namely Zinc Nitrite, Zinc Sulphate and Zinc Acetate (23). The study reveals that the morphology of the ZnO nanoparticle produced depends on the precursor used as indicated in Figure (9). The degradation efficiency also varies as surface to volume ratio and active sites available for sphere shaped and sheet like structured nanoparticles.

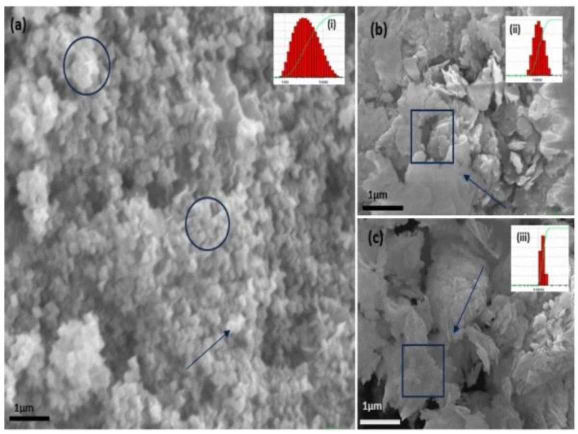

Fig.9. SEM images of three different salt precursors (a) Zinc acetate (b) Zinc nitrate (c) Zinc Sulphate (23)

IX. REUSABILITY OF NANOPARTICLE PHOTOCATALYSTS

Purushotham, D. et. al.(24) investigated that ZnO nanoparticles synthesized from Aqueous Extract of Pavonia zeylanica confirms reusability for photodegradation for five cycles by heating in 100°C in oven after every cycle which results in slight decrease in the degradation efficiency from 89.32% to 82.82%.No changes in the peak position indicates no phase transformation as shown in Figure (10). Akanda et al. (25) suggested different methods for reusability of Nanoparticles which is needed in large scale industrial application. The methods include pH or salt concentration variation, self cleaning and electrochemical regeneration and so on.

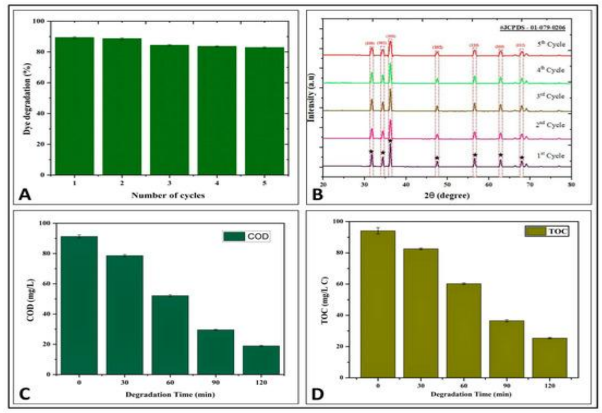

Fig.10. (A) Reusability analysis conductivity over five cycles (B) XRD study; (C) Evaluation of Chemical Oxygen Demand (COD) study using Pz-ZnO-NPs; (D) TOC analysis of Pz-ZnO-NPs (24)

X. ZNO FOR ANTIMICROBIAL ASSAYS

Zone of Inhibition (ZOI), Minimal Inhibitory concentration (MIC) and Minimum Bactericidal Concentration (MBC) were estimated with different concentration of ZnO nanoparticles.

ZOI, MIC and MBC were performed using ASM manual as per CLSI guidelines (33).

Zone of Inhibition (ZOI):

ZOI was performed using disc diffusion method, for which Staphylococcus aureus and E.coli microbes were used. The synthesized ZnO nanoparticles exhibited significant antibacterial activity by inhibiting the growth of microbes. There was increase in ZOI with increase in concentration of ZnO nanoparticles. These results were compared with positive control drug ampicillin. Staphylococcus aureus and E. coli exhibited 22mm and 17mm of ZOI respectively (34).

Minimal Inhibitory concentration (MIC):

1mL of nutrient broth with bacterial culture (E.coli and Klebsiella pneumoniae) of concentration (1.5 x 108 CFU/mL) were incubated for 16 hours, for which 40μL of Indonitrotetrazolium (INT) and incubated for 30 minutes at room temperature. As an end point, the colour changes from yellow to pink and later to white. Colour changes signifies the positivity for inhibiting the bacterial growth.

	ZnO nanoparticles efficacy	Ampicillin
E.coli	125 µg/mL	50 µg/mL
Klebsiella pneumoniae	175 µg/mL	50 µg/mL

Minimum Bactericidal Concentration (MBC)

The cultures which signified negative for MIC test and showed no colour change, 10µL of same sample were spread over nutrient agar plates to measure the MBC.

	ZnO nanoparticles efficacy	Ampicillin
E.coli	150 µg/mL	175 µg/mL
Klebsiella pneumoniae	75 µg/mL	75 µg/mL

These results attributes significant in research field, as several criteria are important in industries like pharmaceuticals and cosmetics.

ZnO application in industries:

ZnO nanoparticle utilized for organic Dye degradation (26) like Methylene blue(27), Congo red(28), Eriochrome Black T (EBT) (29) and for removal of Pharmaceuticals and personal care products (PPCPs) waste in aqueous system (30) . ZnO nanoparticle used in textile industry for odor resistance. In rubber industries for vulcanization of rubber which improves its properties like strength, elasticity and durability. Also used in Tire industries. In cosmetic industries in sunscreen and in skin care.Used in gas sensors (31), transistors (32) and LED. High exciton binding energy improves performance. Antibacterial and antifungal agent. Wound healing ointment, drug delivery systems, Anti corrosion coatings, UV Protection layer. Food spoiling. Solar cell and Hydrogen production.

XI. CONCLUSION

ZnO nanoparticles exhibit great potential in environmental, biomedical, pharmaceutical, and food safety applications because of their effective photocatalytic and antimicrobial activities.

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