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# EVALUATION OF ANTIBACTERIAL ACTIVITIES OF ZnO, Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>, Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>/ZnO & Ag-ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub> NANOPARTICLE AND NANOCOMPOSITES Gowthami K<sup>1</sup>, Arumugam M<sup>2</sup>, Dinesh Kumar N<sup>1</sup>, Thirunarayanan G<sup>1</sup>, Krishnakumar B<sup>3</sup>,

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# ABSTRACT

Inorganic metal oxide nanoparticles and nanocomposites may serve as effective disinfectants, due to their relatively nontoxic profile, chemical stability and efficient antibacterial activity. ZnO,  $Fe_2V_4O_{13}$ ,  $Fe_2V_4O_{13}/ZnO$  and Ag-ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub> have been prepared by sol-gel, liquid phase precipitation method and photodeposition method. Antibacterial activity was tested using ten microbes of biomedical and agriculturally significance was achieved. Synthesized nanoparticles are potential green remediators of polluted water and perilous pathogens.

**Keywords:** ZnO, Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>, Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>/ZnO and Ag- ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>, Antibacterial activity.

# 1. INTRODUCTION

Antibacterial agents are of great importance in several industries e.g., water disinfection, textiles, packaging, construction, medicine and food [1, 2]. Microbial strains including bacteria have expressed remarkable adaptability towards a wide range of antibiotics exhibiting multidrug resistant (MDR). Such resistance of pathogenic microbes towards inhibitory substance is not only alarming for human beings but also for agricultural sector prone to pathogenic attacks [3]. These inorganic compounds present strong antibacterial activity at low concentrations [4]. Green synthesis technique is a simple cheap, ecological and speedy route for the production of nanoparticles as compared to the conventional physical and chemical methods which are quite expensive and potentially hazardous to the environment [5]. Various advanced nanomaterials-including metals and/or metal oxide, MoS<sub>2</sub>, MXenes, and carbon nanomaterials, among other have demonstrated great potential for managing bacterial infections [6-10]. Recent studies have demonstrated that specially formulated metal oxide nanoparticles have good antibacterial activity [11]. Among metal oxide nanoparticles, ZnO nanoparticles as one of the multifunctional inorganic nanoparticles has many significant features such as chemical and physical stability, high catalytic activity, effective antibacterial activity as well as intensive ultraviolet and infrared adsorption with broad range of applications as semiconductors, sensors, transparent electrodes, solar cells, etc. [12,13]. ZnO and metal-doped ZnO materials have been widely investigated due to the case of production and modulation of their properties, which renders materials with enhanced activity for a specific application [14-17]. Zinc Oxide bactericidal effect has been studied in different Gram positive and Gramnegative bacteria [15, 18]. However, majority of results have expressed the superior activity of doped ZnO nanoparticles [19-21]. In nanometer size metallic nanoparticles, iron has received special attention because of its physical and chemical properties which are determined by its size, shape, composition, crystallinity and structure [22]. Bimetallic iron and silver containing nanoparticles (Fe-Ag NPs) have numerous applications in optical, medical and remediation fields [23]. Silver may have an important advantage over conventional antibiotics as it kills all pathogenic microorganisms and no organisms has ever been reported to readily develop resistance to it furthermore microbes are unlikely to

develop resistance against silver as they do against conventional and narrow-target antibiotics because the metal attacks a broad range of targets in the organisms, which means that they would have to develop a host of mutations simultaneously in order to protect themselves for these reasons silver-based compound have been used extensively in many bactericidal applications [24,25]. Nanoparticles of silver have aptly been investigated for their antimicrobial properties [26]. Literature survey reveals that there is no report available for the evolution of antibacterial study of above nano composites. Hence, in the present study, ZnO, Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>, Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>/ZnO and Ag-ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>, were prepared and their antibacterial activity was investigated Gram positive and Gram negative bacteria such as, Staphylococcus aureus, Proteus vulgaris, Bacillus substilis, Proteus mirabilis, Klebsiella pneumonia, Salmonella typhi-A, Pseudomonas aeruginosa, Salmonallatyphi-B, Escherichia coli, and Shigellaflexneri.

## 2. MATERIAL AND METHODS

#### 2.1. General

Zinc nitrate hexahydrate (Himedia), oxalic acid dihydrate (Merck), Ferric nitrate, ammonium metavanadate (Qualigens), Silver nitrate (SD Fine-chem ltd) were used as received. The experimental solution was prepared using distilled water.

#### 2.2. Fabrication of $Fe_2V_4O_{13}$ / ZnO

The synthesis of Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub> was followed the procedure reported in literature [27]. About 100 mL (0.4M) of zinc nitrate hexahydrate and 100 mL of 0.6 M oxalic acid in deionized water are brought into boil separately, and zinc nitrate solution is added rapidly to the oxalic acid solution and then immediately 300 mg of  $Fe_2V_4O_{13}$  is added to this, and the mixed suspension is stirred for 3 h. The formed precipitate zinc oxalate dihydrate with  $Fe_2V_4O_{13}$  is filtered, washed with DI water several times, and dried in hot air oven at  $80^{\circ}$ C for 6 h. Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>-zinc oxalate dihydrate coupled system is taken in a silica crucible and calcined at 450°C for 12 h in muffle furnace at the rate of rising temperature 20 °C min<sup>-1</sup>. After 12 h, the furnace is allowed to cool down to room temperature. The ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub> catalyst is collected and used. This catalyst has 11 wt% of Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>with respect to ZnO. Similarly, with 3, 7 and 14 wt% of  $Fe_2V_4O_{13}$ coupled ZnO are prepared using appropriate amount of  $Fe_2V_4O_{13}$ . The bare ZnO is prepared using the same procedure without addition of  $Fe_2V_4O_{13}$ .

**2.3.** Fabrication of Ag coated with ZnO/  $Fe_2V_4O_{13}$ About 1.0 g of ZnO/  $Fe_2V_4O_{13}$  coated with AgNO<sub>3</sub> stirring for 2 h in photoreactor. Used solvent ethanol and water 60+40 mL and finally filtered and dried in 100 °C.

#### 2.4. Sources

Materials used for the study of antimicrobial activity of ZnO,  $Fe_2V_4O_{13}$ ,  $Fe_2V_4O_{13}$  / ZnO and Ag- ZnO/ $Fe_2V_4O_{13}$ catalysts are Nutrient broth, 1.3g, Nutrient agar 5.6 g, Agar-agar 0.5g, petri-plates and cotton wabs. The strains are *Staphylococcus aureus*, *Proteus vulgaris*, *Bacillus substilis*, *Proteus mirabilis*, *Klebsiella pneumonia*, *Salmonella typhi-A*, *Pseudomonas aeruginosa*, *Salmonallatyphi-B*, *Escherichia coli*, *Shigellaflexneri*. The antibacterial activity of nanomaterials was demonstrated using disc diffusion method against different Gram positive and Gram-negative bacteria of clinical significance.

#### 2.5. Measurement of antibacterial assay

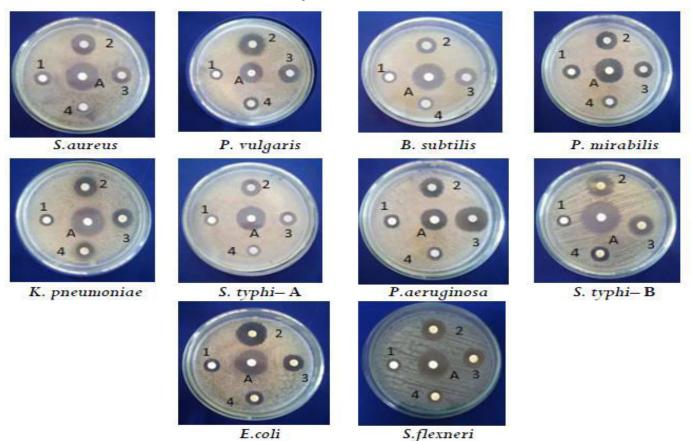
The antibacterial activity of the various nanoparticles was tested using ten bacterial pathogens gram positive and gram negative. Briefly 105 cells/mL in Luria-Bertani broth (LB) were shaken (250 rpm) in light with the various nanoparticles 950 mg for 24 h at 37 °C. The number of viable bacteria was determined by plating serial dilution on LB agar plates and determining the number of colonies forming units (CFU) [28].

## 3. RESULT AND DISCUSSION

#### 3.1. Antibacterial activity of AZF

Antibacterial activities of the prepared ZnO,  $Fe_2V_4O_{13}$ ,  $Fe_2V_4O_{13}/ZnO$ , and Ag-ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub>, and catalyst was measured by Baur Kirby disc diffusion method [29]. In this experiment, there are three Gram-positive and seven Gram-negative microbes such as *Staphylococcus aureus*, proteus vulgaris, Bacillus substills, Proteus mirabilis, Klebsiella pneumoniae, Salmonella typhi-A, Pseudomonas aeruginosa, Salmonella typhi–B, Escherichia coli, Shigella flexneri utilized for evaluation of antibacterial activities for 50 mg of catalyst and Amoxicillin used as standard.

In this antibacterial measurement, test pathogens were spread on Muller-Hinton Agar (MHA) plates. A well 6 mm was made sterile cork borer and loaded with required concentration of drug and placed over the agar. The test plates were incubated for 24 h at 37°C. The zone of inhibition (mm in diameter) were read and taken as the activity against the test pathogen. The measured zone of inhibition is expressed in mm.



**Fig. 1: The zone of inhibition for various bacterial strains by AZF**  $1=ZnO; 2=Fe_2V_4O_{13}/ZnO; 3=Ag-ZnO/Fe_2V_4O_{13}; 4=Fe_2V_4O_{13}$  and A=Amoxicillin

Entry	Catalyst	Zone of inhibition (mm)									
		Gram-positive bacteria				Gram-negative bacteria					
		S.	Р.	В.	Р.	К.	S.	Р.	S.	Е.	S.
		aureus	vulgaris	subtilis	mirabilis	pneumoniae	typhi-A	aeruginosa	typhi-B	coli	flexneri
1	Ζ	10	10	_	12	-	10	10	_	15	8
2	ZF	18	22	13	15	10	8	15	12	20	18
3	AZF	12	25	18	17	22	15	30	24	18	22
4	F	10	17	14	15	20	11	13	12	12	16
Amoxicillin		22	18	33	30	18	24	25	22	20	24

# Table 1: Antibacterial activities of AZF

 $1=ZnO; 2=Fe_2V_4O_{13}/ZnO; 3=Ag-ZnO/Fe_2V_4O_{13}; 4=Fe_2V_4O_{13}$  and A=Amoxicillin

The antibacterial effects of the prepared ZnO,  $Fe_2V_4O_{13}$ ,  $Fe_2V_4O_{13}/ZnO$  and Ag-ZnO/ $Fe_2V_4O_{13}$  catalyst is shown in Fig. 1 and the zone of inhibition values are given in Table 1. The catalyst ZF shows good antibacterial activity against *S. aureus* strain. The remaining Z, AZF and F showed satisfactory activity. Excellent antibacterial activities were found to be ZF and AZF catalyst compound with standard. Good antibacterial activity showed the catalyst F and satisfactory activity possess Z against *P. vulgris*. Except Z the other ZF, AZF and F compounds showed satisfactory antibacterial activity against *B. subtilis*.

Prepared all catalysts showed satisfactory antibacterial activity against *P. mirabilis* strain. The compound AZF and F showed excellent antibacterial activity against *K. pneumoniae.* ZF shows satisfactory activity and Z shows least activity. Catalyst except ZF, the others shows satisfactory antibacterial activity against *S. typhi*-A. Excellent antibacterial activity was found for AZF catalyst against *P. aeruginosa.* The remaining Z, ZF and F catalyst

showed satisfactory antibacterial activities. The solid catalyst AZF shows excellent antibacterial activity against *S. typhi*-B. The antibacterial activity of ZF and F are satisfactory and Z had no activity against *S. typhi*-B.

There is an excellent and good antibacterial activities were observed for ZF and Z, AZF and F catalyst against *E. coli* strains. A good antibacterial activity was shown by ZF, AZF and F compounds and Z shown least antibacterial activity against *S. flexneri* strain.

## 4. CONCLUSIONS

The nanoparticles ZnO,  $Fe_2V_4O_{13}$ ,  $Fe_2V_4O_{13}/ZnO$ , and Ag-ZnO/Fe<sub>2</sub>V<sub>4</sub>O<sub>13</sub> had been prepared by sol-gel, liquid phase precipitation method and photodeposition method. In this review, we have discussed only the antibacterial activity was tested using ten microbes. In this experiment, there are three Gram-positive (*S. aureus*, *P. vulgaris* and *B. subtilis*) seven Gram-negative (*P. mirabilis*, *K. pneumoniae*, *S. typhi*-B, *E. coli* and *S. flexneri*) bacterial strains utilized for 50 mg of catalyst. Nanoparticles also showed better, good and satisfactory antibacterial activity.

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