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Preparation and Characterization of Zinc Ferrite ($ZnFe_2O_4$) Nanoparticles Using Self-Propagated Combustion Route and Evaluation of Antimicrobial Activity.

R Vidya[1] * and K Venkatesan[2].

[1]School of Bio Sciences and Technology, VIT University, Vellore – 632 014, Tamil Nadu, India.
[2]Advanced Materials Research Centre, School of Advanced Sciences, VIT University, Vellore – 632 014, Tamil Nadu, India.

ABSTRACT

The zinc ferrite nanoparticles were prepared by using self-propagated solution combustion route. The metal nitrates were used as an oxidizer and amino acids were used as the fuels to control the combustion reaction. In combustion reaction by changing the fuel, single phase of zinc ferrite was achieved. The X-ray diffraction pattern confirmed the single phase of zinc ferrite and it has found that the X-ray diffraction pattern matched very well with JCPDS data (# card number – 01 082 1049). The prepared zinc ferrite nanoparticles were investigated by X-ray diffraction technique (XRD) and field-emission scanning electron microscope (FE-SEM). Anti-bacterial activity of zinc ferrite nanoparticles was carried by well diffusion technique. The anti-bacterial assay was done against the *bacillus subtilis* (gram positive) and *klebsiella, salmonella* (gram negative) bacteria. The single phase of prepared $ZnFe_2O_4$ nanoparticles showed better anti-bacterial activity compared with mixed phase. The zone width increased, when the concentration of $ZnFe_2O_4$ nanoparticles was increased.

Keywords: $ZnFe_2O_4$, Amino acids, FE-SEM, Anti-bacterial activity

Corresponding author

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INTRODUCTION

Nowadays research is focused on metal oxide nanoparticles due to their complex structure, chemical stability, surface area and volume fraction of atoms at interfacial regions. Based on the material nature they exhibit optical, electrical and magnetic properties. Ferrites are having various applications such as MEMS, data storage [1,2] and iron-oxide based materials have been used in various biomedical applications related to magnetic phenomena such as hyperthermia, targeted drug delivery system, MRI, etc [3]. $ZnFe_2O_4$ nanoparticles are synthesized by various methods to obtain their desirable size, shape and magnetic properties as reported by researchers in recent years. The oxygen (O^{2-}) ions from closed packing of FCC, zinc (Zn^{2+}) ions and ferrous (Fe^{3+}) ions occupied tetrahedral (A) and octahedral (B) sites [4]. These applications depend on the properties of ferrite nanoparticles that are influenced by their composition and microstructure. The special properties of magnetic nanoparticles required for biomedical applications demand precise control of particle size, shape, chemical stability, composition and dispersion conditions that affect these properties.

The compositional and microstructural properties are sensitive to the preparation methods used in their synthesis. Respective methods are available for preparing the $ZnFe_2O_4$ nanoparticles such as solid state reaction [5], redox process [6], microemulsion [7], hydrolysis [8], co-precipitation [9], Polyol [10] and combustion method [11 – 13]. However, the combustion method offers excellent chemical stability, uniform distribution of the metal ions and nanosized particles in the as prepared condition. Hence, we adopted combustion method to prepare the $ZnFe_2O_4$ nanoparticles. Effect of the fuel for combustion reaction on particle size, surface morphology and anti-bacterial assay were investigated.

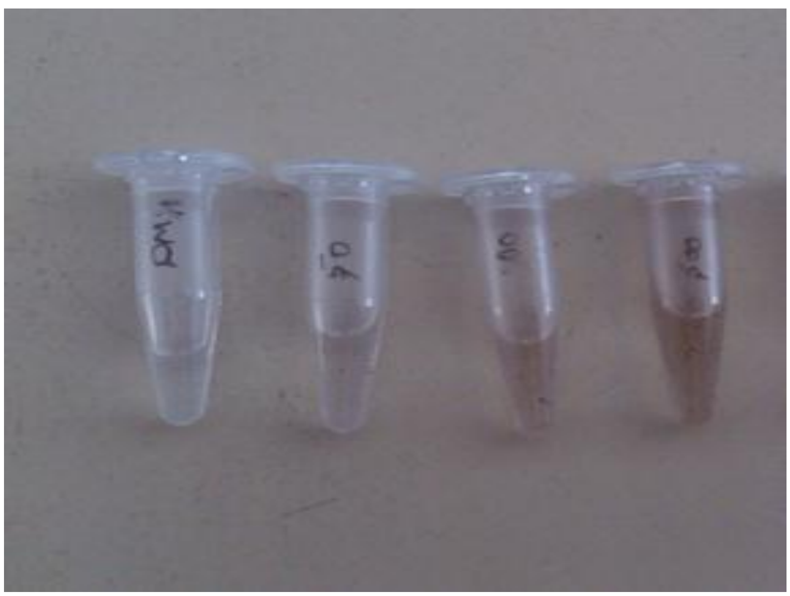

Figure 1: Image of different concentration of $ZnFe_2O_4$ nanoparticles

EXPERIMENTAL

The nanoparticles of $ZnFe_2O_4$ were prepared by self-propagated combustion route. Analytical grade of iron nitrate ($Fe(NO_3)_3 .9H_2O$), zinc nitrate ($Zn(NO_3)_2 .6H_2O$) were taken as oxidants and amino acids such as alanine, glycine and proline were used as the fuel to control the combustion reaction. The metal nitrates and fuel were dissolved in double distilled water to get the precursor solution. The preparation of precursor solution was taken with different fuels. The solution was kept into the preheated furnace for the ignition process. At the end of the combustion process the fluffy foam of our final product was obtained without further calcinations steps.

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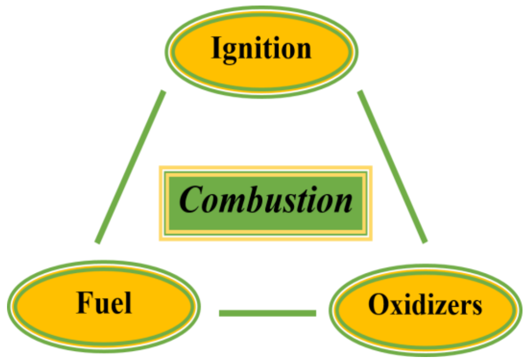

Figure 2: Schematic diagram of Combustion synthesis

Figure 3: As prepared $ZnFe_2O_4$

Bacterial Assay

The bacterial activity for the zinc ferrite nanoparticles was tested on Gram positive *bacillus subtilis*and Gram negative Bacteria *klebsiella, salmonella* by Well diffusion method with three different concentrations (50,100 and 200 µg/L) which was dispersed in the Dimethyl sulfoxide (DMSO) solvent for the easy diffusion into the Agar. Each well was loaded with prepared sample solution. The plates were incubated at 37 $^{\circ}$C for 24 hours. After this period zone of inhibition was observed.

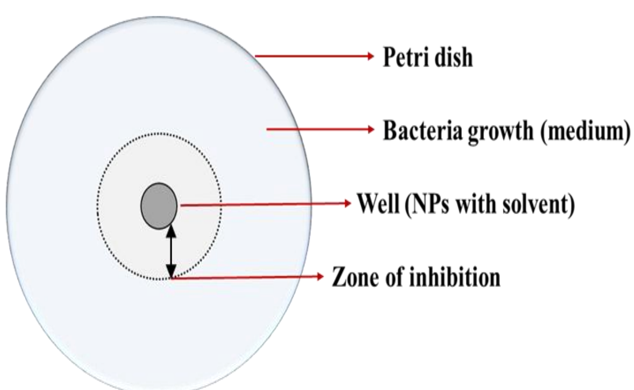

Figure 4: Well diffusion method

RESULTS AND DISCUSSION

The prepared $ZnFe_2O_4$ nanoparticles was easily ground by mortar and pestle for further investigations. The structural parameters were characterized by XRD and FT-IR. The surface morphology was identified by FE-SEM. The anti-bacterial assay was carried by well diffusion method using the as prepared materials against gram positive and negative bacteria. The test confirmed the prepared material can be used as an anti-bacterial agent.

The single phase of $ZnFe_2O_4$ was confirmed by powder X-ray diffraction technique. It was used to calculate the structural parameters of $ZnFe_2O_4$. The prepared $ZnFe_2O_4$ showed the cubic closed spinel structure with $Fd\bar{3}m$ space group. The functional groups were identified by the Fourier-transformation infrared spectroscopy technique. It confirmed the tetrahedral coordination and octahedral coordination using the metal-oxide vibrations. The surface morphology was analysed by field emission scanning electron microscope. It revealed that the prepared material had voids and was porous in nature. The elemental analysis was used to measure the composition of the prepared material - $ZnFe_2O_4$. It confirmed the exact composition of the given molecular formula ($ZnFe_2O_4$).

The bacterial assay was carried out by well diffusion method against zinc ferrite nanoparticles. Bacterial assay claimed that zinc ferrite nanoparticles inhibited the gram positive and gram negative bacteria. The pure cultures were transferred to sterile tryptone broth, which was incubated overnight at 37 °C. The broth culture was used for further determination of Minimum Inhibition Concentration (MIC). The cultures were swabbed on the sterile Muller Hinton (MH) agar plates with four wells made by sterile cork borer. Each well was loaded with 20 µl of the nanoparticle sample solution. The plates were incubated to observe the clear zone of inhibition around the wells. The zone of inhibition for bacterial cultures was measured using the formula given below

$$\textit{Zone of inhibition} = \frac{\textit{Zone of diameter (mm)} - \textit{well diameter (mm)}}{2}$$

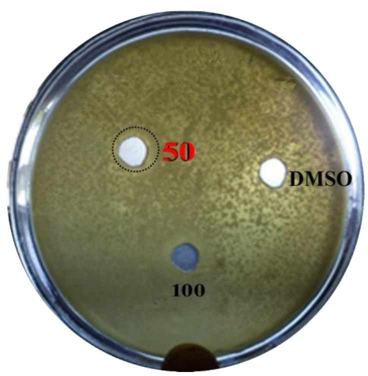

Figure 5: Image of anti-bacterial activity using *klebsiella* against $ZnFe_2O_4$

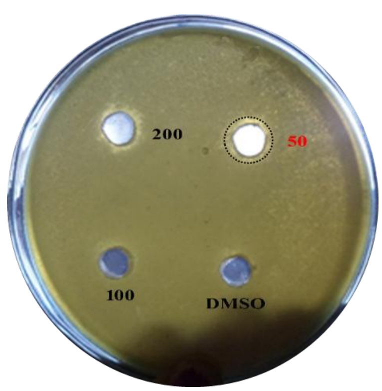

Figure 6: Image of anti-bacterial activityusing *salmonella* against $ZnFe_2O_4$

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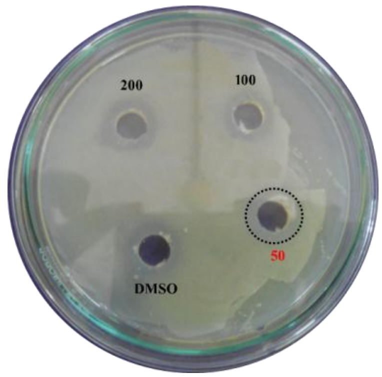

Figure 7: Image of anti-bacterial activity using *bacillus* against $ZnFe_2O_4$ nanoparticles

Table 1: Values of inhibition zone of $ZnFe_2O_4$ against different bacterial agent

Concentrations (mg/L)	*klebsiella*	*salmonella*	*bacillus*
50	3.5 mm	2.5 mm	4 mm
100	4 mm	2.7 mm	4.5 mm
200	4.5 mm	4 mm	5 mm
DMSO	0.2 mm	0.2 mm	0.3 mm

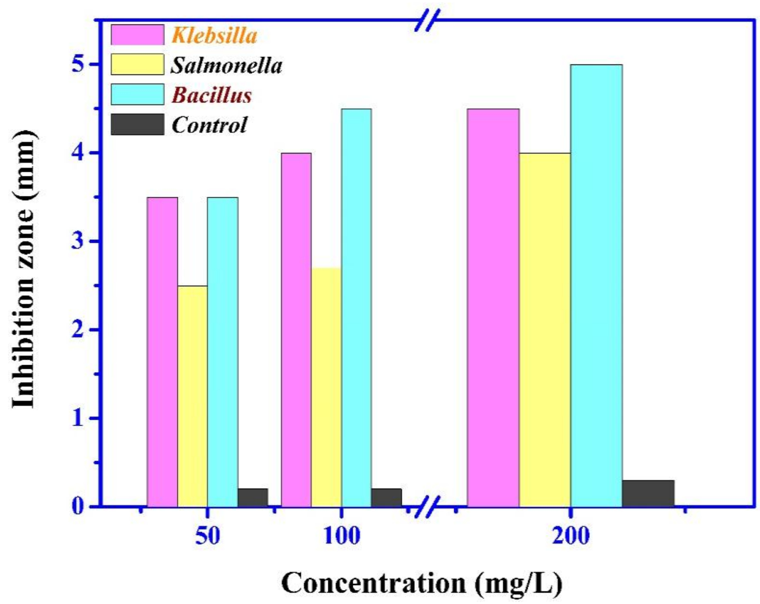

Figure 8: Anti-bacterial activity against $ZnFe_2O_4$ nanoparticles

There are several possible mechanisms for the antibacterial action to ceramic nanopowders. It has been suggested that ceramic nanopowders bind to the membranes of microorganisms, similar to mammalian cells, prolonging the lag phase of the growth cycle and increasing the generation time of the organisms so that it takes each organism more time to complete cell division (14 - 17). The first reason is that the killing rate is intimately associated with the surface area of nanopowders dispersed into the *bacillus*, *klebsiella* and*salmonella* suspension media. The second effect may arise from the structural characteristics of the zinc ferrite nanopowders. The penetration rate of an active oxide through the bacteria cell wall may play a part in the killing rate of ceramic nanopowders against bacteria.

CONCLUSION

The single phase of $ZnFe_2O_4$ nanoparticles were prepared by solution combustion technique. The structural parameters were measured by powder X-ray diffraction technique and functional groups were confirmed by Fourier-transform infrared spectroscopy technique. The porous nature was confirmed by the field-emission scanning electron microscope. The anti-bacterial assay of the prepared material of $ZnFe_2O_4$ nanoparticles was determined through well diffusion method and it confirmed that the prepared material can be used as an anti-bacterial agent. It can be concluded that $ZnFe_2O_4$ nanoparticles showed the maximum inhibition zone for*bacillus* at a minimum inhibitory concentration (MIC) of 50 *ppm*.

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