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Plant Latex Mediated Solution Combustion Synthesis of $Mg_{1-x}Zn_xAl_2O_4$ Nanoparticles: Structural and Antibacterial Analysis

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Abstract - *Crystalline $Mg_{1-x}Zn_xAl_2O_4$ nanoparticles were synthesized via a simple solution combustion method using E-tirucalli latex as fuel. The obtained samples calcined for 3 hours at 1200˚C. These nanoparticles were characterized by various analytical techniques namely, X-ray diffraction (XRD), scanning electron microscopy (SEM), energy-dispersive X-ray (EDX), transmission electron microscopy (TEM), Fourier transform infrared spectroscopy (FTIR) and UV–Visible spectrum. The XRD results showed that the cubic nanoparticles are formed with a space group of Fd-3m. SEM images show the agglomerated spherical nanoparticles. The antibacterial activities of the calcined $Mg_{1-x}Zn_xAl_2O_4$ nanoparticles are preliminarily studied against Gram-positive (S. aureus) and Gram-negative (E. coli) bacteria by implementing micro plate dilution technique. Prepared nanoparticles have shown significant inhibition against Escherichia coli and Staphylococcus aureus. The results obtained indicate that $Mg_{1-x}Zn_xAl_2O_4$ nanoparticles are fairly a beneficial and operational agent for the control of bacterial pathogens, which will be more precise and economical.*

Key Words: Green combustion, nanoparticles, antibacterial activity.

1. INTRODUCTION

Microbial contagion is a severe trouble in the healthcare and food industry, so that growth of antimicrobial agents and surface coatings has been acquiring consideration in modern years. It has been well recognized that toxicity is more generic in the case of micro sized particles. Hence, antimicrobial properties with nano sized particles are of substantial curiosity. The synthesis of inorganic oxides has recently been broadly explored due to their catalytic, sensors, and solar cells applications [1-8]. In recent studies, magnesium aluminium oxide nanoparticles with dopants have demonstrated antimicrobial efficacy against microbes, which significantly have low toxicity and low cost and are ecofriendly compared to conventional synthesis methods [8-12]. Because of the larger surface to volume ratio in nano sized particles, the antibacterial activity is much stronger than that of micrometre-sized particles. Owing to focus over the perovskite materials, we report the influence of Zn as dopant over antibiosis of pathogenic microorganisms in MgAl2O4 nanoparticles prepared by solution combustion process using novel fuel E. tirucalli plant latex as fuel demonstrating their prospective relevance in medicine and agriculture.

1.1 Materials used and Synthesis

The nitrates of magnesium, zinc, and aluminium are of analar grade procured from Sigma Aldrich. India and used without further purification. The plant latex is collected from E.tirucalli plants located in Pavagada village Tumakur district, Karnataka and kept at 4 °C until use.

A homogeneous aqueous solution containing the stoichiometric amounts of nitrate salts and 40ml latex was taken in 300ml crucible and kept in the muffle furnace maintained at 400°C. After 5-10 min the puffy mass of the nanomaterial will be obtained. It was cooled to room temperature and crushed into fine powder. Then these powders were kept for calcination at 1200°C for 4h. finally the product is crushed and used for further analysis. The Zn concentration was increased by 0.25-1 mol% and samples were named as PR1, PR2, PR3 and PR4 respectively.

1.2 characterization methods

The powder X-ray diffraction (PANalytical X'Pert Pro powder diffractometer) using Cu Kα radiation (λ = 0.15418 nm) with a nickel filter is used to check phase purity of the compounds. The morphological details were examined using Hitachi 3000 scanning electron microscope (SEM) with energy dispersive X-ray (EDX) attachments. The Fourier transform infrared spectroscopy recorded using Perkin Elmer Spectrometer with KBr reference by scanning the dried powder of synthesized nanoparticles the range 350–4000 cm^{-1} at a resolution of 4 cm^{-1}.

For studying the antibacterial activity Invitro disc diffusion technique was carried out using microbial strains Gram-negative bacteria: Escherichia coli (NCIM 2574) and one Gram-positive bacteria: Staphylococcus aureus (NCIM 5021) Potato dextrose agar and nutrient

medium were used to culture microbial strains at 37 °C. All the pure microbial strains obtained from National Chemical Laboratory (NCL), Pune, India. Newly synthesized $MgAl_2O_4$: Zn nanoparticles was tested in invitro disc diffusion technique. Antimicrobial potentialities of these nanoparticles were estimated by pre-sterilized filter paper discs (6mm in diameter). The newly prepared nanomaterials were tested in in-vitro using the agar disc diffusion method. In brief, the antimicrobial potentialities of the $MgAl_2O_4$: Zn nanoparticles were estimated by pre-sterilized filter paper discs (6mm in diameter) impregnated with these nanoparticles dissolved in concentration (100 µgmL−1) was placed on the inoculated agar. The plates were incubated for 24 h at 37 °C in case of bacteria.

2. RESULTS AND DISCUSSIONS

Fig 1 depicts the XRD patterns of nanoparticles. The patterns are in good agreement with the JCPDS 12-267.The XRD studies reveals the cubic Perovskite crystal structure with the space group Fd-3m. The nano particle size was assessed using X-ray line broadening technique from the Debye-Scherer's formula, $D = \frac{0.9\lambda}{\beta cos\theta}$ (1)

The average crystallite size calculated from this method is found to be 35-45 nm.

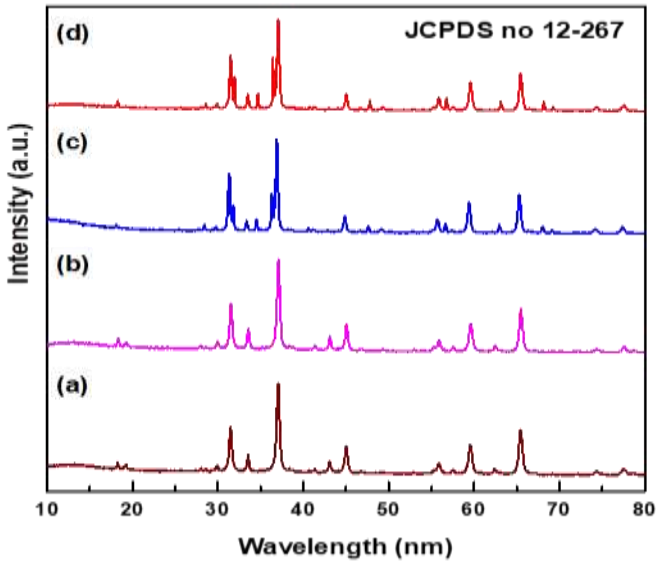

Fig-1: PXRD of $MgAl_2O_4$: Zn nanoparticles

The Fig 2 represents the SEM micrographs of $MgAl_2O_4$: Zn nanoparticles. It is clear from the SEM image that the particles are microgranular and agglomerated by nature. This agglomeration might be due to the liberation of huge amount of gas during the combustion process.

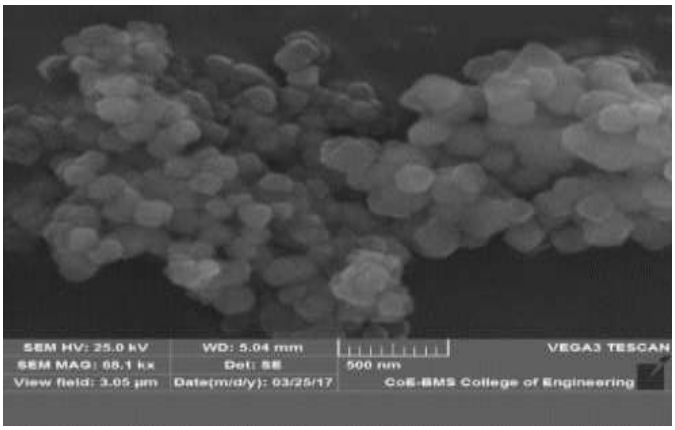

Fig-2: SEM image of $MgAl_2O_4$: Zn nanoparticles

Fig 3 refers to the FTIR spectra. In the 820–400 cm^{-1} region of the FTIR spectrum the observed specific peak at 820, 737 and 573 cm^{-1} may be attributed to the characteristic M–O (possible Mg–O, Zn-O and Al–O stretching frequencies) vibrations for the perovskite structured compound. Also, in the FTIR spectra, the $MgAl_2O_4$ samples exhibited characteristic frequencies around 500 cm^{-1} attributing to the [AlO6] groups and the lattice vibration of Mg–O, Zn-O stretching, and representing the formation of Zn doped $MgAl_2O_4$ nanomaterials [13-18]. The phonon vibrations at 3515 cm^{-1} and 1680 cm^{-1} can be attributed to surface adsorbed water molecules and stretching due to NO_3^- group respectively. The mutual band at around 2380 cm^{-1} can be accounted to the propagation of the IR beam through air [38].

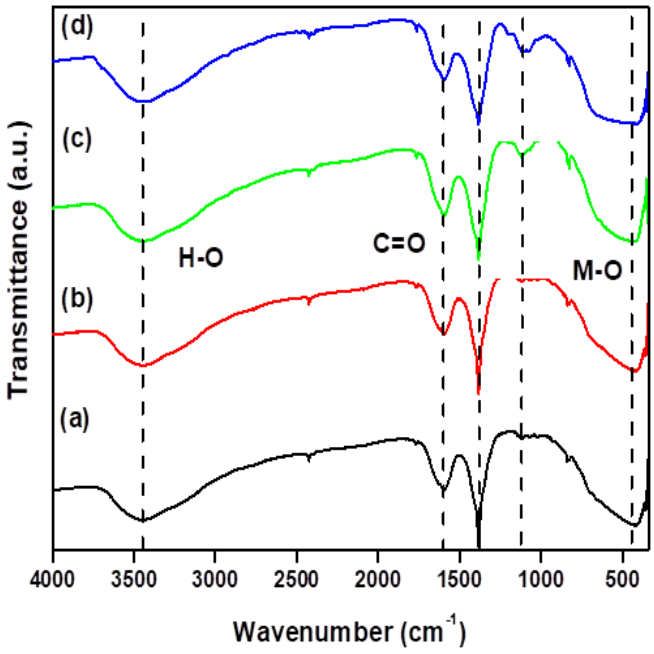

Fig-3: FTIR spectra of $MgAl_2O_4$: Zn nanoparticles

Antimicrobial activity of the $MgAl_2O_4$: Zn nanoparticle was evaluated by means of disc diffusion technique and MIC concentration was evaluated by dilution method as shown in Fig. 4 The materials were screened against Gram positive (S. aureus) and Gram-negative bacteria (Escherichia coli,). Ampicillin was used as the standard and the minimum inhibitory concentration (MIC) was determined [15]. The nanoparticle with various amount were tried to explore both the concentration and composite component impacts on the antimicrobial activity[15].

Fig -4: Petridishes streaked with bacterial cultures

showing MIC and MBC values of $MgAl_2O_4$: Zn nanoparticles synthesized using different combination of Zn dopant with e-tirucalli latex.

Growth of microbial strains inhibited by increase in the concentration of the $MgAl_2O_4$: Zn nanoparticles. The Gram-negative bacteria interaction is more compared to Gram-positive bacteria due to difference in cell walls, physiology, cell structure, metabolism or degree of contact of organisms with nanoparticles [19]. Furthermore, the antibacterial effect against bacterial strains is induced by $MgAl_2O_4$: Zn nanoparticle may be due to the interaction between the positively charged nanoparticle and negatively charged cell wall. Therefore, it appears that the activity of $MgAl_2O_4$: Zn nanoparticle against bacterial strains is multi-factorial [20]. Fig 5 and Fig 6 represents the Graphical representation of the zone of inhibition and mechanism of antibacterial activity respectively. In relation to MBC test, two microorganisms; S. aureus showed a higher susceptibility to ZnAl2O4 NPs at 0.25 and 0.0025 µg/mL and E. coli (2.5 µg/mL) with MgAl2O4 nanoparticles. For PR2 sample where both Mg and Zn were in same molar ratio only E-coli shows higher zone of inhibition where as for PR4 sample the Mg components gets nullified and hence the s. aureus shows more susceptibility.

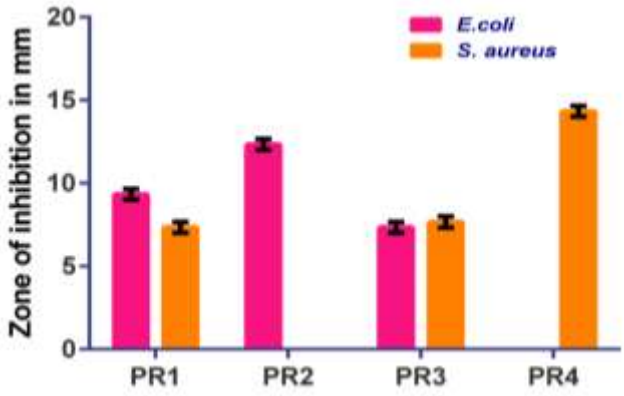

Fig-5: Graphical representation of Zone of inhibition

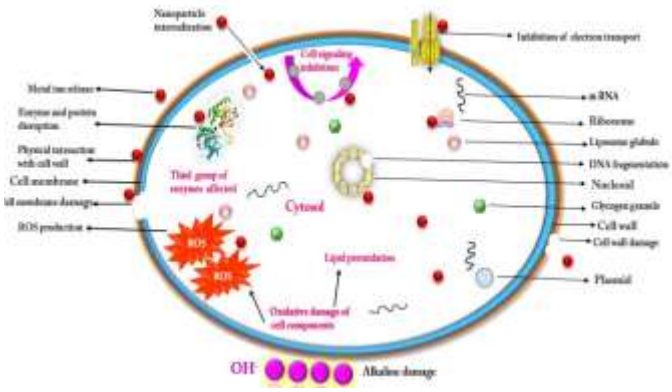

Fig-6: Schematic representation of mechanism of the antibacterial activity.

3. CONCLUSION

We successfully synthesized $Mg_{1-x}Zn_xAl_2O_4$ nanoparticles via nontoxic and eco-friendly fuel approach using E-Tirucalli latex. Also studied the structural and antibacterial activity of the synthesized nanoparticles. There is no structural or phase changes were observed, hence nano aluminate prepared by E- Tirucalli latex (natural fuel) is an alternative method to produce large nano aluminate compound with low cost. In relation to MBC test, two microorganisms; S. aureus showed a higher susceptibility to ZnAl2O4 NPs at 0.25 and 0.0025 µg/mL and E. coli (2.5 µg/mL) with MgAl2O4 nanoparticles. When both Mg and Zn are in combined in different molar ratio both the bacteria have shown significant susceptibility. The Nanoparticles derived fungicides can be achieved in a simple cost-effective scheme and are appropriate for pronouncing new categories of nanobiotics ingredients would be used as an innovative, ecofriendly, Nano antimicrobial for various phytopathogenic organisms.

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