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# Preparation, Characterization and Antibacteral Applications of MgO-Zro$_2$ Mixed Oxide Nanoparticles

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***Abstract:*-- MgO-ZrO$_2$nanoparticles were synthesized by wet chemical method by mixing the solutions of magnesium sulphate (0.45 M) and zirconium oxychloride (0.25M) in aqueous sodium hydroxide and refluxed at elevated temperature. The prepared mixed oxide nanoparticles were characterized by FT-IR, XRD, SEM, EDAX, DSC and CV studies. From XRD studies the average size of the nanoparticles was determined to be 9.44 nm in diameter. The elemental composition and morphology of mixed metal oxide nanoparticles have been analyzed by EDAX set up attached with scanning electron microscope (SEM). EDAX analysis indicate the presence of elements like Mg, Zr and O. SEM morphological studies of the nano MgO-ZrO$_2$ particles revealed the particle distribution with somewhat spherical like structure. Cyclic voltammetric studies exhibit good adherent behavior on electrode surface and good electroactivity. DSC thermogram of mixed oxide nanoparticles were recorded at the heating rate of 50°C/min. Antimicrobial activity of the synthesized mixed oxide was also investigated.**

***Keywords:*-- EDAX, DSC, SEM,nanoparticles**

## 1. INTRODUCTION

Mixed metal oxidenanoparticles (MMOs) can play significant role in various branches of chemistry and material Science.MMOs are widely used for acid-base, electronic active component devices and heterogeneous catalysis. The distinctive electronic and magnetic properties procure when combining two metals in oxide matrixes. [1, 4]

Nanoparticles have been used in many commercial applications and entered probability into aquatic ecosystems .The toxicity and discharge of nanoparticles on human health have received more attention in recent years [8]. There are various methods for the preparation of MOs such as sol-gel, wet impregnation, [1] Mechano-chemical synthesis, hydrothermal methods and co precipitation method.In this work, wet chemical method wasimplementing to synthesize MgO-ZrO$_2$NPs. The advantages of MgO-ZrO$_2$ NPs were studied for various analytical technique and antibacterial activity.

## 2. EXPERIMENTAL METHOD

The precursors Magnesium Sulphate ($MgSO_4.7H_2O$), Zirconium oxychloride ($ZrOCl_2.8H_2O$) and the precipitant (NaOH) are purchased from Sigma Aldrich company. All solutions are made up with deionized water.

### *2.1. Preparation of Reagents*

$MgSO_4.7H_2O$ (0.45 M) is prepared by dissolving 3.085 g in 50mL deionized water.$ZrOCl_2.8H_2O$ (0.25 M) is prepared by dissolving 7.250 g in 50mL deionized water. NaOH (1.0 M) is prepared by dissolving 8g in 200mL deionized water.

### *2.2. PREPARATION OF MgO Nano particles:*

50mL of 0.45M $MgSO_4.7H_2O$ was added in drop wise to an aqueous solution of NaOH(50mL,1M),making a final volume of 100ML.The mixture was stirred well and refluxed at an elevated temperature for 2hrs .The sample was collected by centrifugation ,washed with water and dried over for two days at room temperature

### *2.3. PREPARATION OF MgO-ZrO$_2$ MIXED OXIDENano particles:*

Nano MgO-ZrO$_2$ mixed oxide was prepared by wet chemical method .In this method 50mL of 0.45M $MgSO_4.7H_2O$ was added to an aqueous solution of 100mL of 1M NaOH and stirred well. To this mixture 50mL of 0.25M $ZrOCl_2.8H_2O$ was added making a final volume of 200mL.The resulting mixture was stirred well and refluxed at an elevated temperature for 2h. The sample was collected by centrifugation, washed with water for the removal of anions and dried over two days at room temperature [14].

### *2.4. Instrumentation*

The FTIR spectra were recorded using a shimadzu instrument. The computer controlled XRD system JEOL IDX8030 was used to record the X-ray differaction of samples.EDAX and SEM measurements were carried out by JEOL JSM-6700 filed emission and scanning electron microscope .The cyclic voltammetry studies were carried out using electrochemical work station (model 650C) CH-

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Instrument Inc.,TX,USA. DSC thermogram was performed using Perkin Elmer DSC 4000.

## 3. RESULT AND DISCUSSION

### *3.1. FTIR Studies*

Fig.1shows FT-IR spectrum of MgO-$ZrO_2$.The strong absorption band at 469 $cm^{-1}$was assigned to the Zr-O vibration [10]. The intense bands observed at 3445 and 1638 $cm^{-1}$ could be due to the presence of hydrated compounds. The peak obtained at around 619 $cm^{-1}$was due to the stretching vibration of Zr-O-Zr bond [6] .The frequency of OH region shows broad band at 3445 $cm^{-1}$, [5] and this band corresponds to –OH stretching vibration of surface hydroxyl groups. The peaks observed at 1121$cm^{-1}$ and 1383 $cm^{-1}$ could be due to Mg-O interaction.

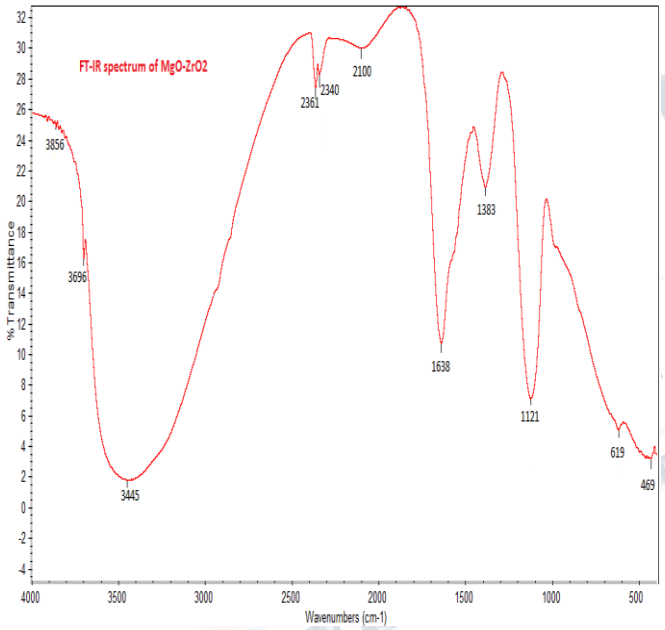


***Fig.1. FTIR Spectrum of MgO-$ZrO_2$***

### *3.2.X-Ray Diffraction Pattern*

The X-ray diffraction pattern (XRD) recorded at 2θ values between 10° and 100° are represented in Fig. 2. Nano MgO-$ZrO_2$ has 3 prominent peaks at 2θ value $19.11^0$, $51.04^0$ and $77.31^0$. It is partly amorphous and partly crystalline in nature. The average size of the MgO-$ZrO_2$ NPs calculated using Debye Scherer's formula is 10 nm. The XRD peaks at $51.04^0$ correspond to the monoclinic phase zirconia NPs [3, 4].The peak at 2θ value$77.31^0$ indicates [5] the formation of nano MgO-$ZrO_2$ [10]. There is a less intense diffraction band centered at about 19° in the 2θ axis, which corresponds to the amorphous MgO-$ZrO_2$ matrix due to the interconversion phase changes [2, 9]

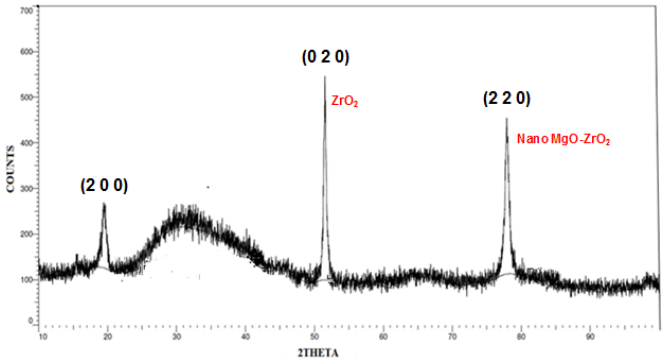


***Fig.2.XRD pattern of MgO-ZrO2nanoparticles***

### *3.3. Scanning Electron Microscopy*

The morphology of the as-prepared MgO-$ZrO_2$ was observed by SEM. Fig.3.a. Shows the SEM images of MgO-$ZrO_2$ nanoparticles at different magnifications. The SEM observation clearly illustrates that the MgO-$ZrO_2$ nanoparticles formed are porous in structure [6, 7] and almost spherical in shape.

### *3.4. EDAX Analysis*

EDAX (Energy Dispersive X-ray) analysis was performed to confirm the elemental form of magnesium and zirconium.Fig.3.b.represents the EDAX profile of MgO-$ZrO_2$ nanoparticles. The EDAX spectrum shows a strong signal at 2 keV and 10.2 Kev, which corresponds to metallic Zirconium and Magnesium. [9, 10]

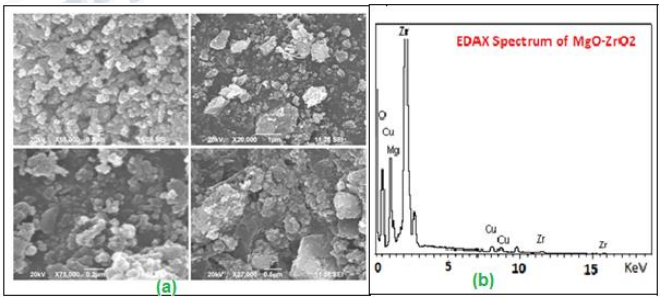


***Fig 3(a). SEM image and (b) EDAX Spectrum of MgO-$ZrO_2$ nanoparticles***

### 3.5. Cyclic Voltammetric behaviour

Cyclic voltammetric behavior of MgO-$ZrO_2$ mixed oxides nanoparticles (Fig.4) was recorded using CHI-650 Electrochemical work station. The potential window was between -0.8V and 1.6V on GCE at 50 mV/S. Cyclic voltammetry behavior of nano of MgO-$ZrO_2$ mixed oxides showed two oxidation peaks one at -0.0275V and other oxidation peak at 0.6195V, which is due to the presence of $ZrO_2$ and MgO respectively. The two reduction peaks observed at 0.1974V and at -0.4970V were entirely different

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from the behavior of oxidation peaks.The peak potential 0.6195V with higher peak current at pH= 4.3 which is different from that of simple oxides, which clearly indicated the formation of the mixed oxides[13].

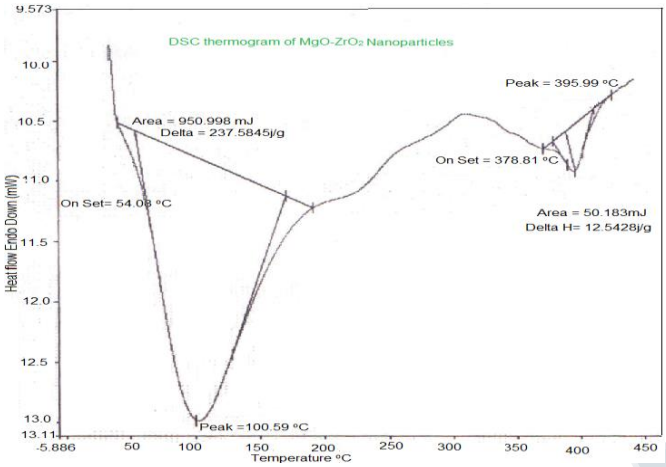


***Fig 4. Cyclic Voltammetry behavior of MgO-$ZrO_2$ nanoparticles***

### *3.6. DSC Analysis*

DCS Thermogram of nano MgO-$ZrO_2$ mixed oxide was recorded at the heating rate of 50°C/min. The glass transition temperature ($T_g$), crystallization temperature ($T_c$) and melting tempareture ($T_m$) of the mixed metal oxide nano particles were determined from the DSC curves shown in Fig.5. The $T_g$ value of MgO was found to be 12.54°C [13]. The $T_c$ value of MgO found to be 54.08and it is melted at 100.59 °C. The crystallization temperature $ZrO_2$is 395.99°Cand it is melted at a temperature of 378.61°C. The melting temperature of bulk simple oxides are higher when compared to MgO-$ZrO_2$, which have lower melting temperature. Since the MgO-$ZrO_2$ are in the nano scale range. Melting temperature of bulk MgO and $ZrO_2$ were 2852°C and 514°C respectively [1, 12]

### *3.7.Antibacterial activity*

The In-vitro antimicrobial activity of MgO-$ZrO_2$ nanoparticles was shown in table.1. The nano MgO-$ZrO_2$ (Fig 6) at high concentration of differerent antimicrobial activity against different test organisms were used for the present study. The maximum inhibition zone 12 mm was observed in E.coli., when compared with other bacterial species [12]. Hence nano MgO-$ZrO_2$ mixed oxide can be used to treat infectious diseases caused by E.coli. The result shows that all the bacterial species used for the present study were moderately sensitive towards MgO-$ZrO_2$ when compared with standard tetracycline.

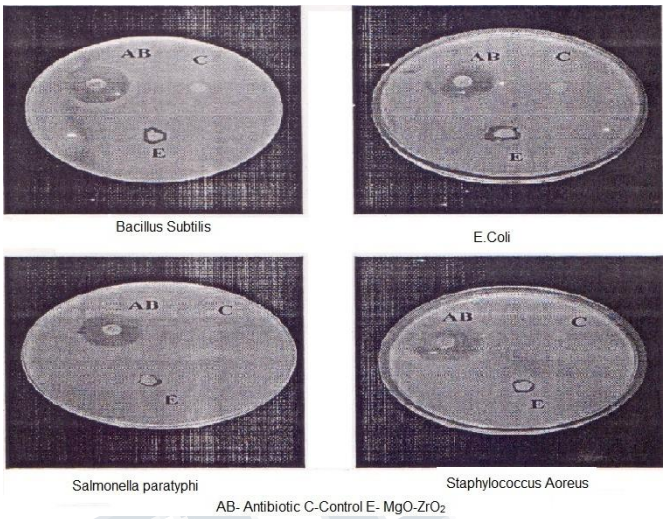


***Fig 6. Antibacterial activity nano of MgO–$ZrO_2$ mixed Oxide***

***Table: 1 Antibacterial activity nano of MgO–$ZrO_2$ mixed Oxide***

| Sl. No | Name of the bacteria | Zone of inhibition (mm) | |
|---|---|---|---|
| | | Std Antibiotic Amikacin | NanoMgO-$ZrO_2$(16.6mg/disc) |
| 1 | E.coli | 25 | 12 |
| 2 | Staphylococcus aureus | 25 | 10 |
| 3 | Bacillus Subtilis | 22 | 9 |
| 4 | Salmonella paratyphi | 21 | 8 |

## 4. CONCLUSION

In summary, Nano MgO–$ZrO_2$ was synthesized by wet chemical method. The chemically synthesized nanoparticles were characterized using FTIR and XRD studies. The size of the synthesized mixed metaloxides was in the nm range and they were found to be thermally stable. SEM analysis showed a spherical like morphology. Cyclic voltammetric studies revealed that the mixed oxide has good adherent and electrochemical activity on glassy carbon electrode and thus it was found to be corrosive protection agent for paints formulation. Antibacterial studies showed that nano MgO-

$ZrO_2$ mixed oxide can be used to treat infectious diseases caused by E.coli.

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