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# Chemical Synthesis and Characterisation of Multimetaloxidezno-Mgo–Zro2nanocomposite

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***Abstract*- Nanotechnology is a facilitating technology that deals with nanometre sized particles in several fields of science such as Chemistry, Physics,Biotechnology and also in Material science. A heterogeneous, versatile multi metal oxide ZnO-MgO–ZrO2 nano catalyst was prepared by chemical co-precipitation method. The synthesized multimetaloxide nanoparticles were characterized by several analytical techniques such as UV, FT-IR spectroscopy, XRD (X ray Diffraction Studies) and SEM (scanning electron microscope). The UV−visible absorption spectrum showed a characteristic optical absorption peak of ZnO-MgO–ZrO2 nano catalyst at 260 nm. The X-ray diffraction pattern suggested the formation and crystallinity of ZnO-MgO–ZrO2 NPs. Spherical ZnO-MgO–ZrO2 NPs synthesized with an average particle size of 26 nm were confirmed by scanning electron microscopy. Photo catalytic degradation was also investigated with crystal violet dye under UV-irradiation source.**

**Keywords: - Multi metal oxide,ZnO-MgO–ZrO2 , XRD, SEM.**

## I. INTRODUCTION

The uncontrolled release of colored waste water contaminated with dyes from textile, paper, rubber and plastic industries have led to serious environmental contamination [1]. These adversely affect the quality of water, inhibit sunlight penetration and reduce photosynthetic reactions. In addition, some dyes are either toxic or carcinogenic [2]. Currently available water treatment technologies either concentrate the pollutants present by transferring them to other phases (adsorption or coagulation) or involve high operating costs with possible generation of toxic secondary pollutants into the ecosystem (sedimentation, filtration, chemical and membrane technologies) [3]. Recent studies have shown that heterogeneous semiconductor photocatalysis can be an alternative to conventional methods for the removal of dye pollutants from water [4].

In heterogeneous catalysis, mixed metal oxides (MMOs) play a very important role because of their versatile chemical and physical properties and wide applications in the field of catalysis and technology.Mixed metal oxides represent one of the most important and widely employed classes of solid catalysts, either as active phases or supports. Mixed oxides are applied both for their acid–base and redox properties and constitute the largest family of catalysts in heterogeneous catalysis. [5]Magnesium oxide (MgO) is important oxide materials that are used in many applications such as catalysis, catalyst supports, toxic wastes remediation, refractory materials and adsorbents . Magnesium oxides are used as destructive adsorbents for toxic chemical agents. Multi-metal oxides containing aluminium have also been found to have several applications in catalysis. Zinc oxide (ZnO) is an exclusive material that has encouraged a vast amount of research in different areas. It is a wide-bandgap semiconductor with the bandgap energy of 3.3eV. It has potential as a luminescent material because the binding energy of exciton is 60 meV which is higher than 70that of the other semiconductors

## II. MATERIALS AND METHODS

### 2.1.Materials

All the materials used for the research work were reagent grade and collected from Sigma-Aldrich.UV-Vis spectrophotometer (JASCO V-650)was used to obtain the absorption spectrum of the synthesized MgO·ZrO2·ZnO metal oxide nanocomposite. The synthesized nanocomposites were investigated with a Philips CM 200 model using 200kV electronacceleration voltage and with a resolution of 2.4A0. XRD measurements were made by PanalyticalX'Pert Powder X'CeleratorDiffractometer measurement range: 0 to 80 degree in 2θ.

### 2.2.Methods

#### 2.2.1.Synthesis of MgO·ZrO2·ZnOnanocomposite

MgO·ZrO2·ZnO metal oxide composite was synthesized by chemical co precipitation method from the aqueous solution of the metal salts. Solutions of 0.01M MgSO4, 0.01M Zr(NO3)2and 0.01MZnCl2 and a solution of 1M Na2CO3 in distilled water were prepared. MgSO4, Zr(NO3)2 and ZnCl2solutions were mixed together in a beaker in 1:1:1 ratio and stirred vigorously at room temperature for five minutes. Then the solution of 1M Na2CO3 was added slowly with agitation, until precipitation was complete. The resultant mixture was stirred for further 4 h at 60 ºC with

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Vol 3, Issue 4, April 2018

constant stirring. After terminating the reaction, the white metal carbonate precipitate was separated from the solution by centrifugation, washed several times with deionized water and finally dried at 220 ºC in an oven. The obtained white precipitate was crushed in a mortar to make it amorphous. Then the amorphous powdered sample was calcined in a muffle furnace at 900 ºC for 4 h [6].

**2.2.2. Photocatalytic Measurement:** Nanosized MgO·ZrO2·ZnO nano composite is a good photocatalyst to degrade organic contaminants, such as crystal violet dye. The dye solution was prepared by dissolving 10mg powder of crystal violet dye in 100ml distilled water. 0.1g of MgO·ZrO2·ZnO nanoparticles was added to 100ml of prepared crystal violet dye solution and the mixer was stirred magnetically for 1 h in shadow before exposing to sunlight. Then the colloidal suspension was placed in a closed chamber and irradiated with sunlight. The reactions were observed one by one in every time interval of 10 min for 1hr. Finally, the rate of dye decomposition was monitored by taking 4ml samples from each set and recording the UV-Vis spectra in the wavelength after centrifugation andfiltration [7].

## III. RESULT AND DISCUSSION

### 3.1.Optical Studies

#### 3.1.1.Ultraviolet-Visible Spectral Analysis:

To observe the absorption edge of the assynthesized photocatalyst, UV-Vis absorption spectra was recorded using spectrophotometer. The UV-Vis spectra of prepared catalyst MgO·ZrO2·ZnO by varying the precursors concentration ranging from 0.01M-0.15M are shown in Fig.1. It was noted that, the MgO·ZrO2·ZnO nano composite had characteristic absorption band at 265 nm [8]. From the UV-Vis results, it is noticed that the absorbance increases with increasing the precursor'sconcentration (0.01M-0.15M).There is an increase of absorbance values from 0.7749 to 1.8205 at 265nm in MgO·ZrO2·ZnO nano composite synthesized. Increase in precursor's concentration, leads to increased interaction ofMgO·ZrO2·ZnO nano composite. Thus, this increasing interaction might have caused the increase in the absorbance values.

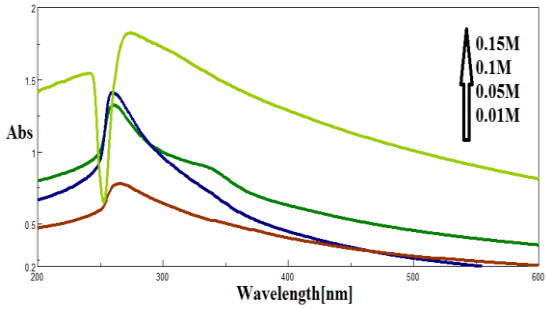


***Fig.1.UV-Visible spectra of MgO·ZrO2·ZnO nano composite by varying the precursor's concentration.***

### 3.2. STRUCTURAL STUDIES

#### 3.2.1. FTIR analysis:

FTIR spectrum of MgO·ZrO2·ZnO nano composite is shown in Fig.2. The bands observed at 3444 cm-1 and 1557 cm-1 are assigned to stretching and bending vibrations of H2O absorbed from the environment [24], respectively for MgO·ZrO2·ZnO. The stretching mode of Zn-O bond appeared at 545 cm−1 [1]. The peaks observed at about 800-400 cm−1 indicating the formation of metal oxide (M-O) bonds. The peak observed at 620 cm−1 indicating the formation of Mg-O bond [9]. The Fourier transform infrared spectrum (FT-IR) of MgO–ZrO2shows a strong absorption at 470 cm−1 due to the Zr–O vibration[10].

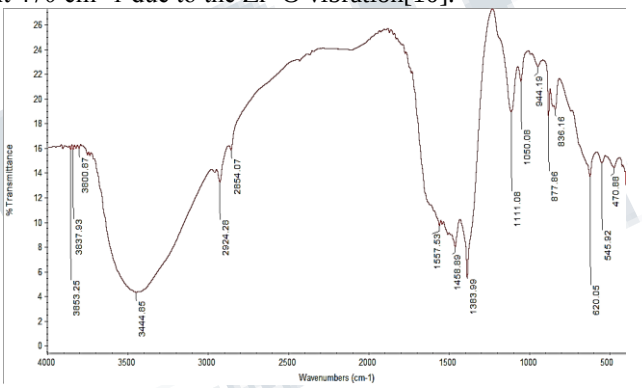


***Fig. 2 FT-IR spectra of MgO·ZrO2·ZnO nanocomposite***

#### 3.2.2. X-Ray Diffraction Analysis

Fig.3shows the XRD pattern of MgO·ZrO2·ZnO.The crystallite sizes of the samples were calculated by using Scherrer's formula [11]d = k λ / β cosθwhere k (= 0.94) is the shape factor, d= average particle size, β is full width at half maxima (FWHM), θ is the Bragg angle, λ(=1.5406 Å) is the wavelength of Cu-Kα. The average particle size of MgO·ZrO2·ZnO nano composite was found to be 26 nm.

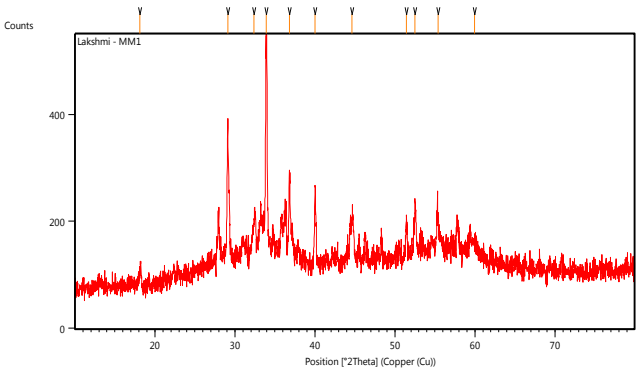

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*Fig. 3 X-Ray Diffraction pattern of MgO·ZrO2·ZnO nanocomposite*

### 3.2.3. Scanning Electron Microscope:

The SEM images of MgO·ZrO2·ZnO nano compositeat different magnifications have been recorded. The SEM images of multi metal oxide nanocomposites were obtained to observe the particle size and morphology. A characteristic textures and morphology of MgO·ZrO2·ZnOhave been revealed by the SEM study as shown in Fig. 4. From the SEM images, it is clear that MgO·ZrO2·ZnOparticles were obtained in a nano size range and showed uniform sized particles with somewhat crystal like morphology with an average size range of 26–31 nm [10].

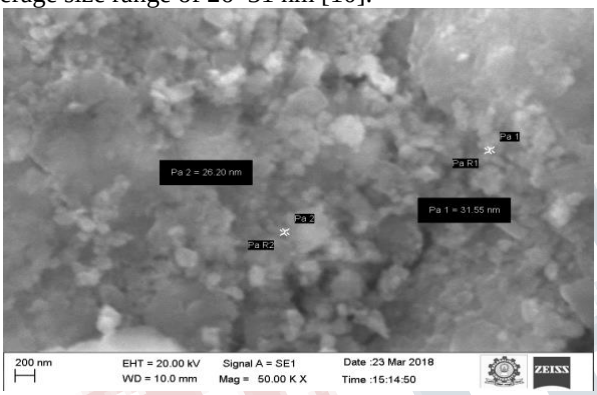


*Fig. 4 SEM image of MgO·ZrO2·ZnO nanocomposite*

### 3.3.Evaluation Photocatalytic activity ofMgO·ZrO2·ZnO nano composite:

The UV visible absorbance values of pure crystal violetdye solution shows at590nm. The characteristic absorbance value at 590nm was used to track the photocatalytic degradation process. From Fig.5ait can be clearly noticed from the recorded values that no significant changes of the concentration of Crystal violet dye after 3hirradiation, which indicated that pure Crystal violetdye solution, cannot be easily degraded by UV light. The degradation efficiency of pure crystal violet dye within 3h irradiation time was about 41% and it was shown in Table.1. The crystal violet dye degradation in the presence of MgO·ZrO2·ZnO nano compositewas verified by the decrease of the peak intensity during 90 min exposure in solar light shown in Fig.6. The dye degradation (%) was calculated by using the following equation (2) and its variant with the time of sunlight exposure was shown in Fig.5b.

Efficiency=[1-C/C0]x100

Where C0 is the initial concentration of crystal violet and C is the concentration of the dye solution at the selected irradiationtime. From the absorbance spectra, it was observed that the maximum degradation efficiency of crystal violet dye within 90 min irradiation time was about 82% for multimetal oxide MgO·ZrO2·ZnO nanoparticles. The absorbance values and degradation efficiency of multimetal oxide MgO·ZrO2·ZnO nanoparticles synthesized is shown in Table.2.The results showed that MgO·ZrO2·ZnO nano compositeincreases the efficiency of degradation. Thus, the multimetal oxide nanoparticles possess much higher photocatalytic activity than pure dye [7].

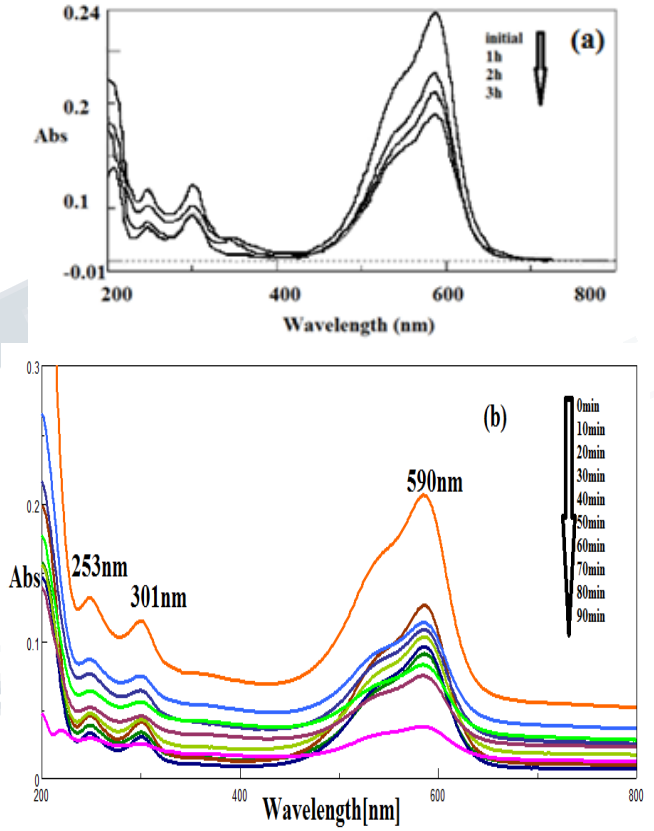


*Fig.5.(a) UV-Visible spectrum of pure Crystal dye under UV light irradiation at different time intervals*

(b)Photocatalytic degradation of Crystal violet dye in the presence of MgO·ZrO2·ZnO nanocompositeunder UV light irradiation at different time intervals

*Table.1. Degradation efficiency of pure Crystal violet dye*

| Time(h) | Degradation (%) |
|---|---|
| 0 | - |
| 1 | 24 |
| 2 | 32 |
| 3 | 41 |

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*Table.2.Absorbance values and degradation efficiency of MgO·ZrO2·ZnO nanocomposite*

| Time (min) | Absorbance | Efficiency (%) |
|---|---|---|
| 0 | 0.2042 | - |
| 10 | 0.1255 | 38 |
| 20 | 0.1129 | 44 |
| 30 | 0.1079 | 47 |
| 40 | 0.1031 | 50 |
| 50 | 0.0954 | 53 |
| 60 | 0.0904 | 56 |
| 70 | 0.0818 | 60 |
| 80 | 0.0745 | 64 |
| 90 | 0.0377 | 82 |

## IV. CONCLUSION

Multi metal oxide nanocomposite MgO·ZrO2·ZnO was successfully synthesized by chemical co-precipitation method. The prepared product was characterized by UV-Vis, FT-IR, XRD and SEM. UV-Vis spectra show the absorption band at 260 nm. The Fourier transform infrared spectrum Confirmed the formation of M-O of nano composite. The particle size was calculated by Scherrer's formula using XRD data and the average particle size was found to be 26 nm. SEM analysis shows the size of the particles was found to be in the range of 26-31 nm.The photo-catalytic activity of synthesized composite MgO·ZrO2·ZnO was tested on crystal violet dye. The highest photocatalytic efficiency of MgO·ZrO2·ZnO was found to be 82%. Therefore they can find application in water purification and textile industries.

## V. ACKNOWLEDGEMENT

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