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# SYNTHESIS, CHARACTERIZATION AND PHOTOCATALYTIC ACTIVITY OF MgO NANOPARTICLES

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**Abstract**

In the present study, nanoparticles of MgO were synthesised through controlled co-precipitation method and microwave assisted fast synthesis method. In both methods MgO was synthesised in presence of citric acid and also in presence of polyvinyl alcohol (PVA). Structural characterizations of all the four synthesized samples were carried out using XRD. Comparison of XRD's of metal oxides with JCPDS confirmed that the formed metal oxide was MgO. Phase purity and crystallinity of these samples were confirmed from XRD. Crystallite sizes were calculated using Scherrer equation. Magnesium oxide nanoparticles showed visible range absorption in addition to UV range. From band gap calculation it was seen that all of them showed subgaps. The metal oxide reported in the present study showed very good photocatalytic activity. It was observed from the UV/Vis spectral analysis that the nanomaterial showed absorption in visible range in addition to UV light. Hence these materials could be used as photocatalyst in the degradation of organic dyes under solar ray irradiation.

**Keywords:** Nanoparticles, Photocatalysis, Photocatalyst

## 1. INTRODUCTION

Industrial waste water tainted with dyes is often discharged into natural water ways. Most of these dyes represent severe harms to the ecological system and the ejection of these colored wastes into natural water bodies may increase the toxicity and the chemical oxygen demand (COD). So it was necessary to find a new way to remove coloured dyes [1, 2] before discharging them into the environment. In the case of industrial dyes, the various conventional technologies do not work efficiently due to high solubility of dyes as well as their resistance to chemical and biological degradation; also they just transfer the contaminants from one phase to another [3]. Photocatalysis is a promising technology in the field of green technology for the removal of dyes. Presently nanotechnology is widely applied for purification and treatment of waste water. The novel properties of nanomaterials such as large surface area, potential for self assembly, high specificity, high reactivity and catalytic potential make them an excellent candidate for both applications [5]. In the present work magnesium oxide (MgO) nanoparticles is prepared in presence of capping agent, citric acid using co-precipitation method. The aim of the work is to study the variations in the properties

obtained when MgO is prepared using microwave assisted fast synthesis method. Also when MgO was prepared in presence of polyvinyl alcohol instead of citric acid using co-precipitation method and microwave assisted fast synthesis method. The MgO nanoparticles synthesized is characterized using X-ray Diffraction Spectroscopy (XRD). The optical studies of the prepared samples are done using UV/Vis spectroscopy. The present study investigates the photocatalytic activity of the synthesized samples as photocatalyst for the degradation of organic dye Congo Red. The results of all the four samples are compared.

## 2 EXPERIMENTAL

AR grade chemicals obtained from Merck were used for the preparation of MgO nanoparticles. MgO was prepared in presence of citric acid by the co-precipitation (MCA) and microwave method (MCAM) and in presence of polyvinyl alcohol (PVA) MPVA and MPVAM. The samples annealed at $500^0$c were used for analysis. XRD study was carried out using XPERT-PRO model powder diffractometer (PAN analytical, Netherlands) employing Cu- $K_\alpha$ radiation ($\lambda = 1.54060A^o$) operating at 40kV, 30mA. The UV/Vis spectrum was obtained using JASCO V-650 UV visible spectrophotometer.

## 3 RESULTS AND DISCUSSIONS

### 3.1 XRD Analysis

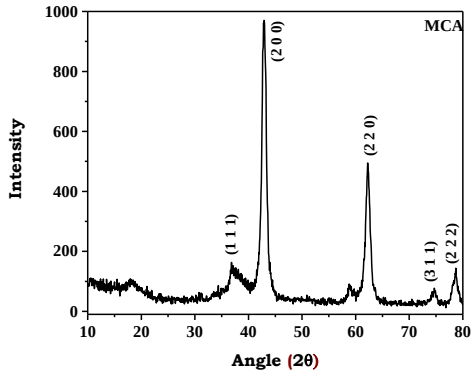


**Fig.1 XRD of MCA**

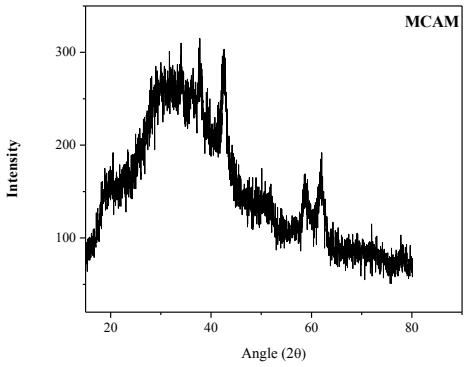


**Fig.2 XRD of MCAM**

Figure 1 shows the XRD of MCA. From the XRD itself it is clear that all the samples formed shows crystalline nature with sharp crystalline peaks. The broader diffraction peaks obtained indicated the smaller crystallite size. The interplanar spacing ($d_{hkl}$ values), 2θ values and relative intensity values of magnesium oxide corresponding to the observed diffraction peaks were compared with the standard values of magnesium oxide as reported by JCPDS-International Centre for Diffraction Data.

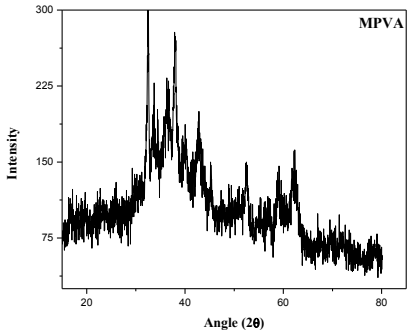


**Fig. 3 XRD of MPVA**

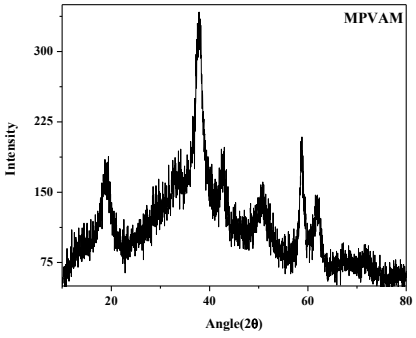


**Fig.4 XRD of MPVAM**

The data obtained for MgO in case of all the four samples was matched with JCPDS-ICDD pattern number #89-7746. The diffraction peaks of the samples matched with the standard MgO cubic system with FCC lattice of MgO (JCPDS: #89-7746). For MCA the diffraction peaks at 2θ values 36.80$^o$, 42.73$^o$, 62.18$^o$, 74.53$^o$ and 78.39$^o$ corresponds to the crystal planes of (111), (200), (220), (311) and (222) respectively with no characteristic peaks corresponding to the impurities, which further confirms the formation of pure stable MgO phase (Figure 3.2). For MPVA, MCAM and MPVAM the diffraction peaks in addition to the 2θ values seen in MCA additional peaks were found. The presence of unremoved PVA and other unreacted ions might have created these impurity peaks. Annealing the samples at higher degrees can remove PVA completely and can give pure crystalline peaks. For MPVA, MCAM and MPVAM the diffraction peaks in addition to the 2θ values seen in MCA additional peaks were found. The presence of unremoved PVA might have created these impurity peaks. Annealing the samples at higher degrees can remove PVA completely and can give pure crystalline peaks. Using curve fitting the full width at half maximum of the major peaks was found and from this the crystallite size of all the samples were found. The result obtained MCA was 11.88 nm For MCAM 10.36 nm, MPVA 14.74 nm and MPVAM 15.55 nm respectively.

### 3.2 UV/Vis Spectrum Analysis

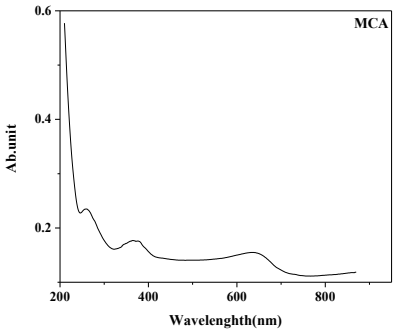


**Fig.5 UV Absorbance spectrum of MCA**

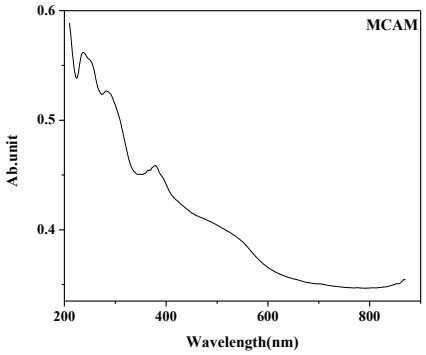


**Fig.6 UV/Vis Absorbance spectrum of MCAM**

The absorbance spectrum of all the four samples were studied and compared. In case of all the four samples of magnesium oxide, MCA, MCAM, MPVA and MPVAM showed a wide band of absorption in both UV and Visible range. In addition to the main peak a weak absorption peak was observed in UV region in case of all the four samples. Figure 5 shows the absorption spectrum of MCA. All the samples showed absorbance in both UV and visible region and also multiple peaks were obtained which shows the presence of subgaps.

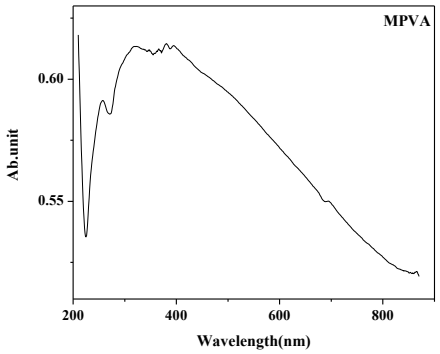


**Fig.7 UV/Vis Absorbance spectrum of MPVA**

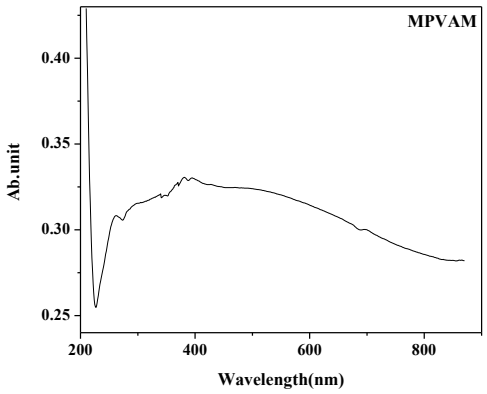


**Fig.8 UV/Vis Absorbance spectrum of MPVAM**

From absorbance spectrum Tauc plot was drawn and the corresponding energy gap was found. The energy band gap of the material is related to the absorption coefficient α by the Tauc relation, $\alpha = A(h\nu - E_g)^n$, where A is a constant, hν is the photon energy ($\nu = c/\lambda$), $E_g$ is the band gap and n is either 2 for an indirect transition or ½ for a direct transition. The $(\alpha h\nu)^2$ vs hν for all the samples were also plotted. Figure 9 shows the Tauc plot drawn for MCA. Similar results were obtained for other three samples.

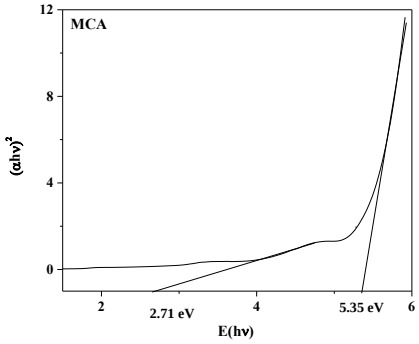


**Fig.9 Tauc plot for MCA**

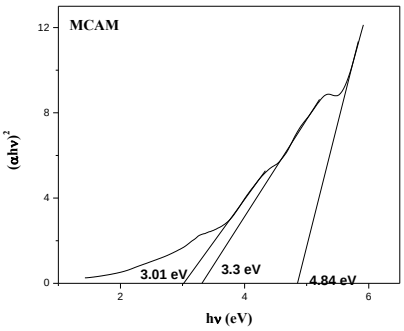


**Fig.10 Tauc plot for MCAM**

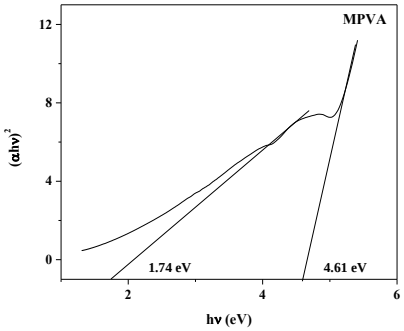


**Fig.11Tauc plot for MPVA**

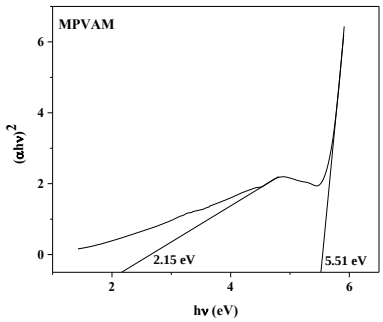


**Fig.12 Tauc plot for MPVAM**

### 3.3 Photocatalytic Studies

The photocatalytic activity of samples for the degradation of the dye; Congo red (CR) were studied using the respective photocatalyst (MCA, MCAM, MPVA, MPVAM) in the presence of UV illumination in a photoreactor. The experiments were performed by suspending the desired amount of photocatalyst into 300 ml of dye solution of desired concentration which was 75 ppm. The experiment was carried out isothermally at 300 K. The concentration of residual dye in the solution after irradiation for 4 hrs was determined by monitoring the absorbance intensity of solution samples at their maximum absorbance wavelength by using UV-Vis spectrophotometer (JASCO V 650 UV/Vis spectrophotometer). To study the effect of contact time on the photodegradation of organic dyes, the following procedure was adopted. To the respective dye solution of concentration, 75 ppm in 300 ml solution kept at 300 K, 0.1 g of the photocatalyst was added and was kept under UV light. After desired time intervals [60, 90, 120, etc], 10 mL of the solution was taken out, centrifuged and UV/Vis absorption spectra was recorded. Figure 13 shows the results obtained for the photodegradation studies of Congo Red done for seven different time intervals, using MCA as photocatalyst. Comparatively MCAM showed enhanced activity. MCA and MPVA showed similar photodegradation activity. Among the four samples MPVAM showed lower level of activity. The activity for four hours was studied. Further increase in time can completely degrade Congo Red completely.

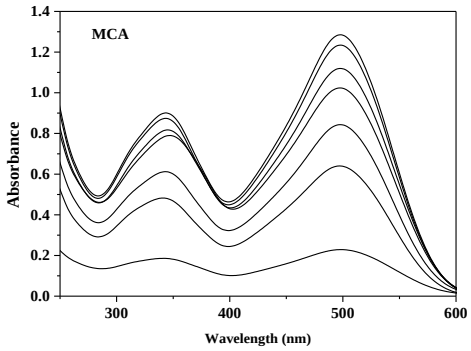


**Fig.13 Photocatalytic degradation of Congo Red using MCA**

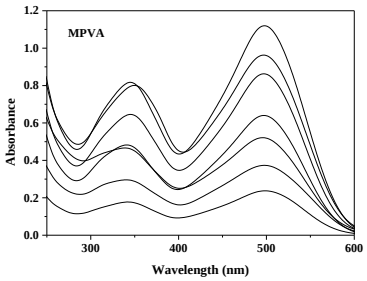


**Fig.14 Photocatalytic degradation of Congo Red using MPVA**

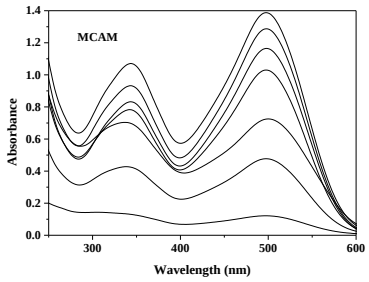


**Fig.15 Photocatalytic degradation of Congo Red using MCAM**

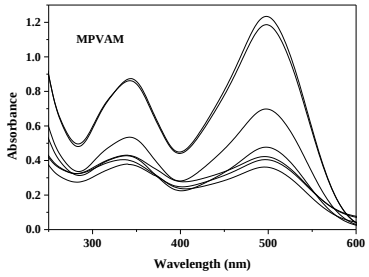


**Fig.16 Photocatalytic degradation of Congo Red using MPVAM**

## 4 CONCLUSIONS

Comparison of XRD's of metal oxides with JCPDS confirmed that the formed metal oxide was MgO. Crystallite sizes were calculated using Scherrer equation. All the four samples prepared in this work showed strong UV as well as visible absorption. From band gap calculation it was seen that all of

them showed subgaps. The metal oxide reported in the present study showed very good photocatalytic activity. It was observed from the UV/Vis spectral analysis that the nanomaterial showed absorption in visible range in addition to UV light. Hence these materials could be used as photocatalyst in the degradation of organic dyes under solar ray irradiation.

## 5. REFERENCES

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