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Synthesis and Characterization of MgO Supported Al_2O_3 Oxide

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ABSTRACT: Synthesis of mixed metal oxide i.e. MgO/Al_2O_3 using Co-precipitation method and their characterization using various Techquies.

Keywords: Mg-Al mixed metal oxide; Co-precipitation.

INTRODUCTION: Mgo used as a catalyst, such as transesterification [1], dehydrogenation of alcohol [2], aldol condensation [3-6], isomerization of alkenes [7,8] and cycloaddition of CO_2 to epoxides [9]. Conventionally, MgO catalysts could be easily obtained by the thermal decomposition of magnesium hydroxide or carbonate [10]. However, the resultant MgO catalysts usually exhibit some disadvantageous, like small specific surface areas, inhomogeneous morphologies, and varied grain size, which are considerably limited their application [11]. Hence, much effort has been devoted to synthesize novel MgO-based materials with desirable characteristics (e.g.,high specific surface area, nanostructure and/or mesoporous porosity).

MgO cubes with controllable particle size could be obtained by chemical vapor deposition method [12]. However, these approaches appear to be rather expensive and complex for conventional catalytic applications. Furthermore, mechanical strength of MgO itself is rather low, and shaping these materials to strong catalyst particles is cumbersome. Taking these aspects into account, it is still an interesting subject to develop simple and efficient method for synthesizing MgO-based materials with advantage features Mg–Al mixed oxides are advantageously prepared by thermal pretreatment of Mg–Al hydrotalcites and possess strong surface basicity, high surface area and high quantity of defects resulting from the incorporation of Al^{3+} in the MgO lattice [13].

MATERIALS AND METHODS: All chemicals are purchased from Aldrich and Rankem chemical suppliers and used as received. The X-ray diffraction patterns were recorded by Bruker 8D advance X-ray diffractometer using monochromator Cu-Kα radiation (40 kV and 30 mV) of wavelength (l)= 1.5405Å. The Scanning Electron microscopy (SEM) and EDS was recorded on JEOL; JSM-6330 LA operated at 20.0 kV and 1.0000 nA. Transmission electron microscopy (TEM) is a microscopy technique recorded on CM-200 PHILIPS transmission electron microscopy (TEM) operated at 200 kV, resolution at 0.23 nm. Temperature programmed desorption (CO_2-TPD) measurements were carried out on a MICROMERITICS CHEMISORB 2750 TPD/TPR. Temperature programmed desorption (TPD) studies were done using 100 mg of the MgO-Al_2O_3 loaded on a quartz reactor. BET surface area was measured by means of N_2 adsorption at 77.74 K preformed on a Micromeritics, ASAP 2010.

Catalyst preparation: In a typical synthesis of mixed metal oxides, an aqueous solution of $MgNO_3$ was added to aqueous solution of $AlNO_3$ under vigorous stirring at room temperature and addition of 20 ml 5% polyethylene glycol (PEG-400) as structure directing agent. This solution was hydrolyzed with 1:1 aqueous ammonia with vigorous stirring until the solution reached to (pH = 9) and the mixture was stirred for 4h at room temperature. The reaction mixture was digest at 60°C in an electric oven for 12 h. The resulting precipitate was filtered, washed with deionized water and dried at 120°C for 12 h. Finally, the dried powders were calcined at 450°C for 2h in air atmosphere named as M1. Similarly, M2, M3, M4, M5, M6 and M7 were prepared.

RESULTS AND DISCUSSION:

Catalyst characterization: The X-ray diffraction of all calcined material is shown in Fig. 1 (M1-M7). The obtained results reveal that the prepared samples have a hexagonal structure with sharp symmetric peaks and broad asymmetric peaks. Calcinations of mixed metal oxides at temperature 450^0C resulted in a broad peak from $2\theta = 20^o$ to 34^o seemed to indicate also the presence of amorphous phase, more important when increasing Mg content. The positions of diffraction lines correspond to the (111), (200) and (220) diffractions of mixed metal oxide.

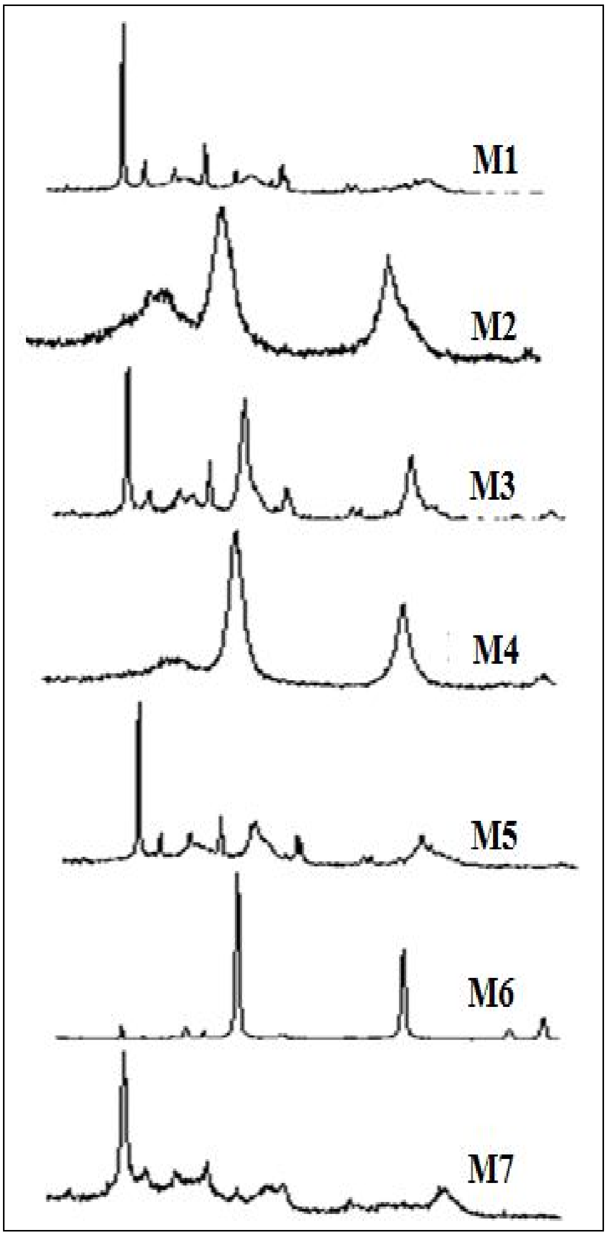
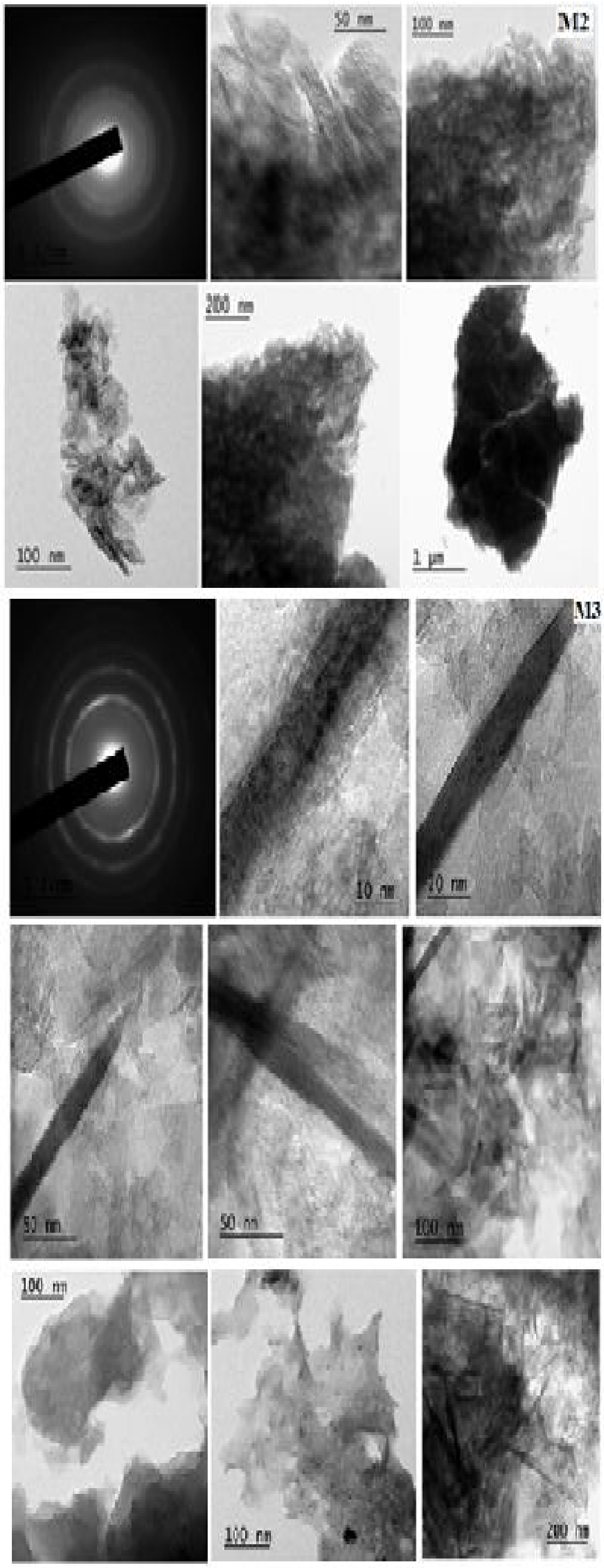


Figure 1: XRD patterns of calcined material (M1) $MgO-Al_2O_3$ (0.2:0.8) (M2) MgO- Al_2O_3 (0.4:0.6) (M3) MgO- Al_2O_3 (0.6:0.4) (M4) MgO- Al_2O_3 (0.8:0.2) (M5) MgO- Al_2O_3 (0.5:0.5) (M6) Pure MgO (M7) Pure Al_2O_3.

TEM images shows various particle size and we have got the images as shown in Fig. 2.



(a)

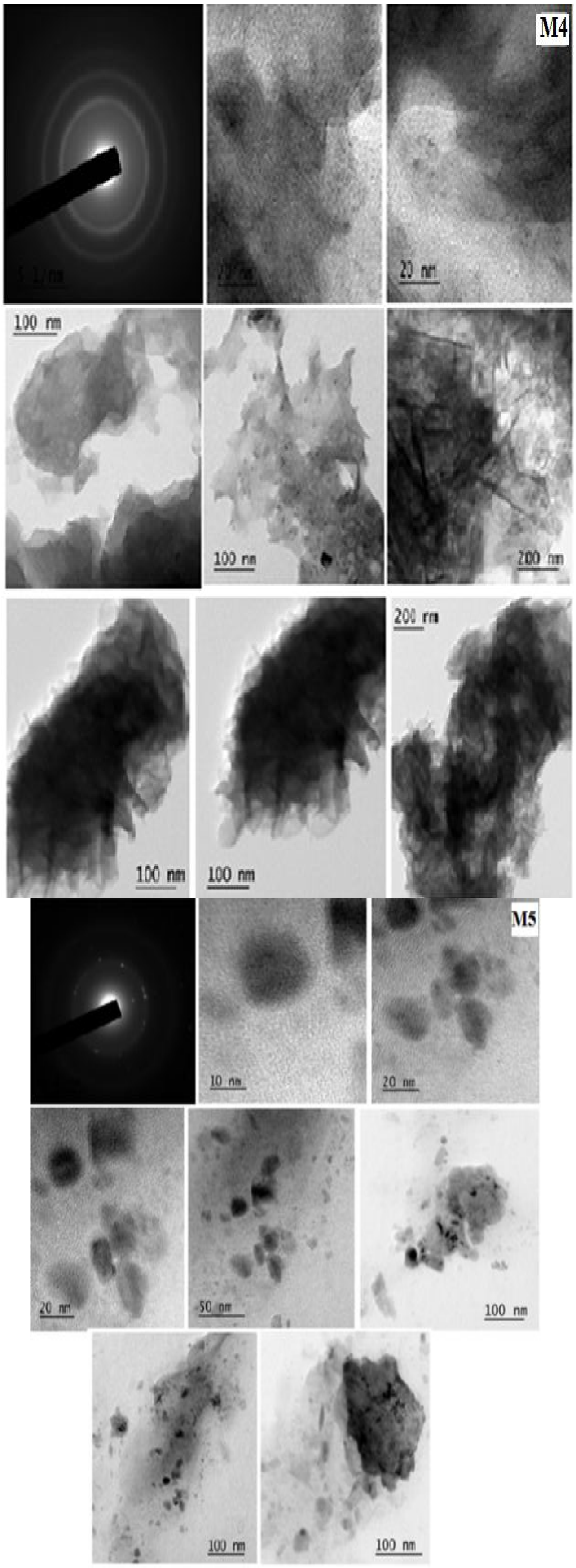


Figure 2: TEM images of calcined samples.

Nitrogen adsorption–desorption isotherms results of calcined samples are shown in Table 2. It is shown that the adsorption-desorption isotherms for M4 catalysts providing further evidence on the enhancing of mesoporous structure after incorporation of Mg precursors. Our result certifies that the inclusion of Magnesium creates a mesoporous structure. Furthermore, the textural parameters of catalysts were distinctly changed by increase in the amount of MgO, where considerable increases in pore volume and pore diameter are observed (Table 2, Sample M4).

Table 2: Surface area, pore structure of calcined samples (M2-M5).

Samples	**BET Area**	**Pore Volume Cm^3/gm**	**Pore Diameter Å**
M4	7.27	0.0860	237.1

CO_2-TPD result after calcinations of sample are shown Table 3. The large basic site 3.3527 m.mol/gm is present in the M4 sample. Increase in the amount of MgO, increases the number of the weak and strong basic sites, however more pronounced changes are visible in the area of the stronger sites.

Table 3: Basicity measurements.

Samples	**CO_2-TPD(m.mol/gm)**
M4	3.3527

CONCLUSION: We have synthesized mixed metal oxide with cost effective and time consuming procedure, with these advantages in future work we use as catalyst for the various organic transformations.

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