

MgO SUPPORTED Al₂O₃ OXIDE: A NEW, EFFICIENT, AND REUSABLE CATALYST FOR SYNTHESIS OF CHALCONES

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Abstract. We have studied a series of mixed metal oxides of Mg-Al by a simple co-precipitation technique. Various characterization techniques, including XRD, SEM, EDS, TEM, BET and CO₂-TPD were carried out to investigate their physicochemical properties. An efficient and facile protocol has been documented for the synthesis of chalcones using different aldehydes and acetophenone using Mg-Al oxide under reflux conditions in ethanol affording good to excellent yields. Recyclability of a catalyst is a significant feature of this protocol. Moreover, it was proposed that MgO can disperse and increase the basicity, pore size and catalytic activity.

Keywords: Mg-Al oxide, environmental catalysis, recyclability, multicomponent reaction.

1. Introduction

As a typical solid base, magnesium oxide (MgO) has been widely used as the active catalyst or the catalyst support in a variety of organic reactions, such as transesterification [1], dehydrogenation of alcohol [2], aldol condensation [3-6], isomerization of alkenes [7, 8] and cycloaddition of CO₂ 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 disadvantages, like small specific surface areas, inhomogeneous morphologies, and varied grain size, which considerably limit 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). For instance, nanoscale MgO materials with

high specific surface area have been synthesized by sol-gel methods combining a supercritical drying procedure [12-15]. MgO cubes with controllable particle size could be obtained by a chemical vapor deposition method [16]. However, these approaches appear to be rather expensive and complex for conventional catalytic applications. Furthermore, a 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 interesting to develop a simple and efficient method for synthesizing MgO-based materials with advantageous features.

Mg-Al mixed oxides are usually prepared by a thermal pretreatment of Mg-Al hydrotalcites and possess a strong surface basicity, high surface area and high quantity of defects resulting from the incorporation of Al³⁺ in the MgO lattice [17]. Mainly due to these properties Mg-Al mixed oxides are attractive catalysts for many reactions such as transesterification [18, 19], Michael additions [20] and alkylation of phenol [21]. However, the major part of works deals with Mg-Al mixed oxides as the catalysts for the condensation reactions [22-24].

Chalcones are a group of compounds with various substitution patterns on two aromatic rings of 1,3-diphenyl-2-propen-1-one. Chalcones constitute an important class of natural products belonging to the flavonoid family, which have been reported to possess a wide spectrum of biological activities, including antibacterial, antifungal, anti-inflammatory, antitumor, antifeedant and antimutagenic properties [15-27]. Additionally, some of chalcone derivatives have been found to inhibit several important enzymes in cellular systems, such as xanthine oxidase [28] and protein tyrosine kinase [29, 30]. Chalcones are also key precursors in the synthesis of many biologically important heterocycles such as benzothiazepine [31], pyrazolines [32], 1,4-diketones [33] and flavones [34]. Hence, the synthesis of chalcones has generated a vast interest among organic, as well as medicinal chemists.

Several methods have been reported for the chalcones synthesis. The most commonly used method is the base catalyzed Claisen-Schmidt reaction of a methyl ketone and aldehyde using sodium hydroxide (NaOH) [35], potassium hydroxide (KOH) [36], barium hydroxide

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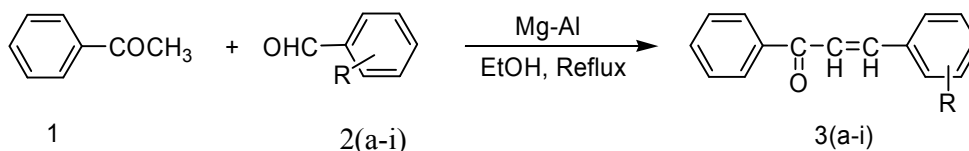
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Ba(OH)₂ [37] and lithium hydroxide (LiOH·H₂O) [38]. The acid-catalyzed synthesis of chalcones using aluminum trichloride (AlCl₃) [39], dry HCl [40], boron trifluoride etherate (BF₃·Et₂O) [41], titanium tetrachloride (TiCl₄) [42] and ruthenium trichloride (RuCl₃) [43] has also been reported. Reaction is carried out under acid-catalyzed conditions, especially when one of the components, either the aldehyde or the acetophenone, is base sensitive [44, 45]. Although, the reaction can be promoted by the above reagents and catalysts, it suffers from several disadvantages like long reaction time and drastic reaction conditions. In addition, these catalysts are



Scheme 1

2. Experimental

2.1. Materials and Methods

All chemicals were purchased from Aldrich and Rankem chemical suppliers and used as received. The uncorrected melting points of compounds were taken in an open capillary in a paraffin bath. ¹H NMR spectra were recorded on an 300 MHz FT-NMR spectrometer in CDCl₃ as a solvent and chemical shift values are recorded in δ (ppm) relative to tetramethylsilane (Me₄Si) as an internal standard. The X-ray diffraction patterns were recorded by Bruker 8D advance X-ray diffractometer using a monochromator Cu-Kα radiation (40 kV and 30 mV) of wavelength $\lambda = 1.5405 \text{ \AA}$. The SEM and EDS data were 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 with the resolution of 0.23 nm. Temperature programmed desorption (CO₂-TPD) measurements were carried out on a Micromeritics Chemisorb 2750 TPD/TPR. Temperature programmed desorption (TPD) studies were done using 100 mg of MgO-Al₂O₃ loaded in a quartz reactor. BET surface area was measured by means of N₂ adsorption at 77.74 K performed on a Micromeritics, ASAP 2010.

2.2. Catalyst preparation

In a typical synthesis of mixed metal oxides, an aqueous solution of MgNO₃ was added to the aqueous solution of Al(NO₃)₃ under vigorous stirring at room temperature. Then 20 ml of 5% polyethylene glycol (PEG-400) as a structure directing agent were added. This

not environmentally friendly and not attractive for commercial adoption due activity of the catalyst and the generation of a corrosive waste.

Hence, there is a need for simple and clean reaction chemistry in order to increase the atom-economy of the process. So far the use of mixed metal oxide as a catalyst for the reaction has not been explored and in continuation of our interest in a solid acid-base catalyst for organic transformations [46-60], we herein developed a green, simple and convenient method for the synthesis of chalcones from a substituted aldehyde and acetophenone in ethanol under reflux conditions (Scheme 1).

solution was hydrolyzed with 1:1 aqueous ammonia under vigorous stirring until the solution reached pH 9 and the mixture was stirred for 4 h at room temperature. The reaction mixture was digested at 333 K in an electric oven for 12 h. The resulting precipitate was filtered, washed with deionized water and dried at 393 K for 12 h. Finally, the dried powders were calcined at 723 K for 2 h in an air atmosphere.

2.3. General Procedure for the Chalcones Synthesis

An equimolar mixture of acetophenone, 4-chlorobenzaldehyde and Mg-Al catalyst (2 mmol) was stirred in ethanol (10 ml) under reflux conditions. After the completion of the reaction (monitored by TLC), the crude mixture was worked up in ice-cold water and purified by recrystallization.

3. Results and Discussion

3.1. Optimization Results for Model Reaction

In the experiments concerning the activity of different Mg-Al, the chalcones synthesis with benzaldehyde and acetophenone were used as a model reaction. It was found that Mg-Al (M4) shows the excellent activity (Table 1, entry 4). The increase in the loading increases the product yield.

To examine the solvent effect the chalcones synthesis using benzaldehyde and acetophenone catalyzed by Mg-Al oxide was selected as a model reaction (Table 1). The reaction in DCM gave a poor yield (Table 1, entry 1).

Both CH₃CN and THF gave a very poor yield (entries 2, 3). DMF gave a modest yield (entry 4). Ethanol provided the best yield of 94 % (entry 5). Therefore, ethanol was chosen as the optimized solvent for the synthesis of chalcones.

Next, the effect of the catalyst loading was subsequently addressed, with catalyst amounts varied from 0.05 to 0.2 g. Using lower amounts of the catalyst resulted in lower yields, and in the absence of catalyst the yield of the product was found to be very low. The best results were obtained using 0.1 g of the catalyst (Table 2, entry 2). The increase in the catalyst amount (0.15 and 0.2 g) does not increase the product yield (entries 3, 4).

The general applicability of this method was further evaluated for structurally diverse aldehydes under optimized reaction conditions, and the results are depicted in Table 3. A variety of different aldehydes, containing electron-withdrawing or donating groups, were subjected to react with acetophenone to afford the corresponding chalcones in excellent yields (Table 3).

The reusability of the catalyst was checked by separating the Mg-Al oxide from the reaction mixture by a simple filtration, washing with CH₂Cl₂, and drying in a vacuum oven at 333 K for 3 h prior to reuse in subsequent reactions. The recovered catalyst can be reused at least three additional times in subsequent reactions without

significant decrease in a product yield (Table 4). ¹H NMR results are depicted for compounds 3a and 3b (Table 5).

Table 1

Optimization of solvent in the model reaction

Entry	Solvent	Time, h	Isolated yield, %
1	DCM	2	27
2	CH ₃ CN	2	20
3	THF	2	22
4	DMF	2	30
5	EtOH	1	94

Note: benzaldehyde (10 mmol), acetophenone (10 mmol) and ethanol (10 ml) under reflux conditions

Table 2

Optimization of catalyst in the model reaction

Entry	Catalyst amount, g	Time, h	Isolated yield, %
1	0.05	1	38
2	0.1	1	94
3	0.15	1	94
4	0.2	1	94

Note: benzaldehyde (10 mmol), acetophenone (10 mmol) and ethanol (10 ml) under reflux conditions

Table 3

Synthesis of substituted chalcones using Mg-Al oxide

Compound	Aldehyde	Time, min	Isolated yield, %	MP, K
3a	Benzaldehyde	55	95	328–331 [61]
3b	4-chlorobenzaldehyde	49	89	373–376 [61]
3c	3-methoxybenzaldehyde	58	92	331–333 [61]
3d	2-chlorobenzaldehyde	47	91	360–362 [61]
3e	4-nitrobenzaldehyde	63	88	371–373 [61]
3f	2-nitrobenzaldehyde	65	87	689–691 [61]
3g	4-methoxybenzaldehyde	45	91	349–351 [61]
3h	2-methoxybenzaldehyde	49	93	329–331 [61]
3i	4-bromobenzaldehyde	51	91	367–369 [61]

Note: benzaldehyde (10 mmol), acetophenone (10 mmol) and ethanol (10 ml) under reflux conditions

Table 4

Reusability of catalyst in the model reaction

Entry	Run	Isolated yield, %
1	1 st	94
2	2 nd	93
3	3 rd	92

Note: benzaldehyde (10 mmol), acetophenone (10 mmol) and ethanol (10 ml) under reflux conditions

Table 5

Spectral data for compounds

Comp	H-2	H-3	J ₂₋₃ , Hz	H-3', H-4', H-5', Ar-H	H-2', H-6'
3a	7.56, d	7.85, d	15.9	7.69–7.43, m	8.05, dd, J _o = 8.1 Hz, J _m = 1.2 Hz
3b	7.53, d	7.78, d	15.9	7.64–7.40, m	8.04, d, J _o = 7.2 Hz

3.2. Catalyst Characterization

XRD analysis. The X-ray diffraction of all calcined material is shown in Fig. 1. 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 723 K resulted in a broad peak from $2\theta = 20^\circ$ to $2\theta = 40^\circ$ and 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.

Scanning electron microscopy. Fig. 2 shows SEM morphology picture of prepared materials, namely a mesoporous crystal structure for MgO:Al₂O₃ (1:1), pure MgO and Al₂O₃. Further, the series of samples with the addition of Mg shows a successive decrease in the crystallite size and development of porous surface.

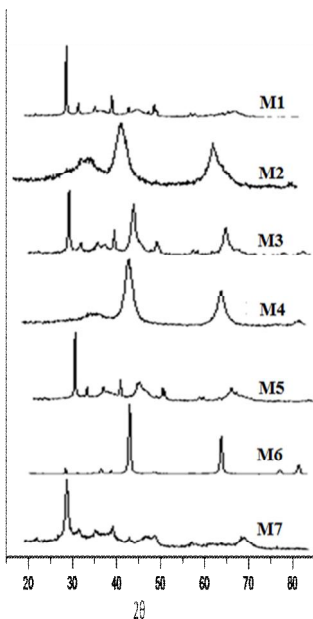


Fig. 1. XRD patterns of calcined materials: MgO-Al₂O₃, 0.2:0.8 (M1); MgO-Al₂O₃, 0.4:0.6 (M2); MgO-Al₂O₃, 0.6:0.4 (M3); MgO-Al₂O₃, 0.8:0.2 (M4); MgO-Al₂O₃, 0.5:0.5 (M5); pure MgO (M6) and pure Al₂O₃ (M7)

Fig. 2 shows the SEM images of M1, M2, M3, M4, M5, M6, and M7 (M1-M7) samples. It can be seen that catalysts agglomerated seriously and showed a bulk shape (Fig. 2, M1, M2 and M3). After the increase in the amount of Mg on Al the sample exhibited the morphology of loose and homogeneous sphere and had a smaller particle size of 10 nm (Fig. 2, M4). However from the SEM micrograph it can be seen that the effect of addition of Mg clearly shows alteration in crystallite size, improvement in morphology and increasing the porosity. EDS analysis shows the composition of metal present in calcined samples (Table 6).

Transmission electron microscopy. The dimensions of the samples M2-M5 were investigated by a transmission electron microscope (Fig. 4). TEM images of calcined samples show dense aggregates of small crystallites (M1), very rarely organized in a filamentous structure (M2, M3). Moreover, rare particles of sizes (10±50 nm) are seen (M4).

BET analysis. Nitrogen adsorption-desorption isotherms for the calcined samples are depicted in Fig. 5. It is shown that the adsorption-desorption isotherms for M4 catalysts provide the enhancing of mesoporous structure after incorporation of MgO precursors. Our result certifies that the inclusion of Mg creates a mesoporous structure. Furthermore, the textural parameters of catalysts were distinctly changed by the increase in the amount of MgO, where considerable increases in pore volume and pore diameter are observed (Table 7, M4).

Temperature programmed desorption (TPD). CO₂-TPD curves of M2-M5 samples after calcinations are shown in Fig. 6. The shape of the curves indicates the existence of the basic sites of the different strengths. The peaks are irregular. All samples have a large desorption peak appeared with a maximum in the range of 573–723 K, which ascribes to the existence of basic sites on the surface of the samples. The large basic site 3.3527 m-mol/g is present in the M4 sample (Table 8, M4). Increase in the amount of MgO increases the number of weak and strong basic sites, however more pronounced changes are visible in the area of the stronger sites.

Table 6

Composition of Mg-Al mixed metal oxides

Samples	wt %			Atomic		
	Mg	Al	O	Mg	Al	O
M2	20.40	18.42	61.58	15.70	12.77	71.53
M3	32.37	7.12	60.51	24.76	4.91	70.33
M4	38.37	1.03	60.60	29.21	0.71	70.09
M5	31.80	8.72	59.49	24.45	6.04	69.51

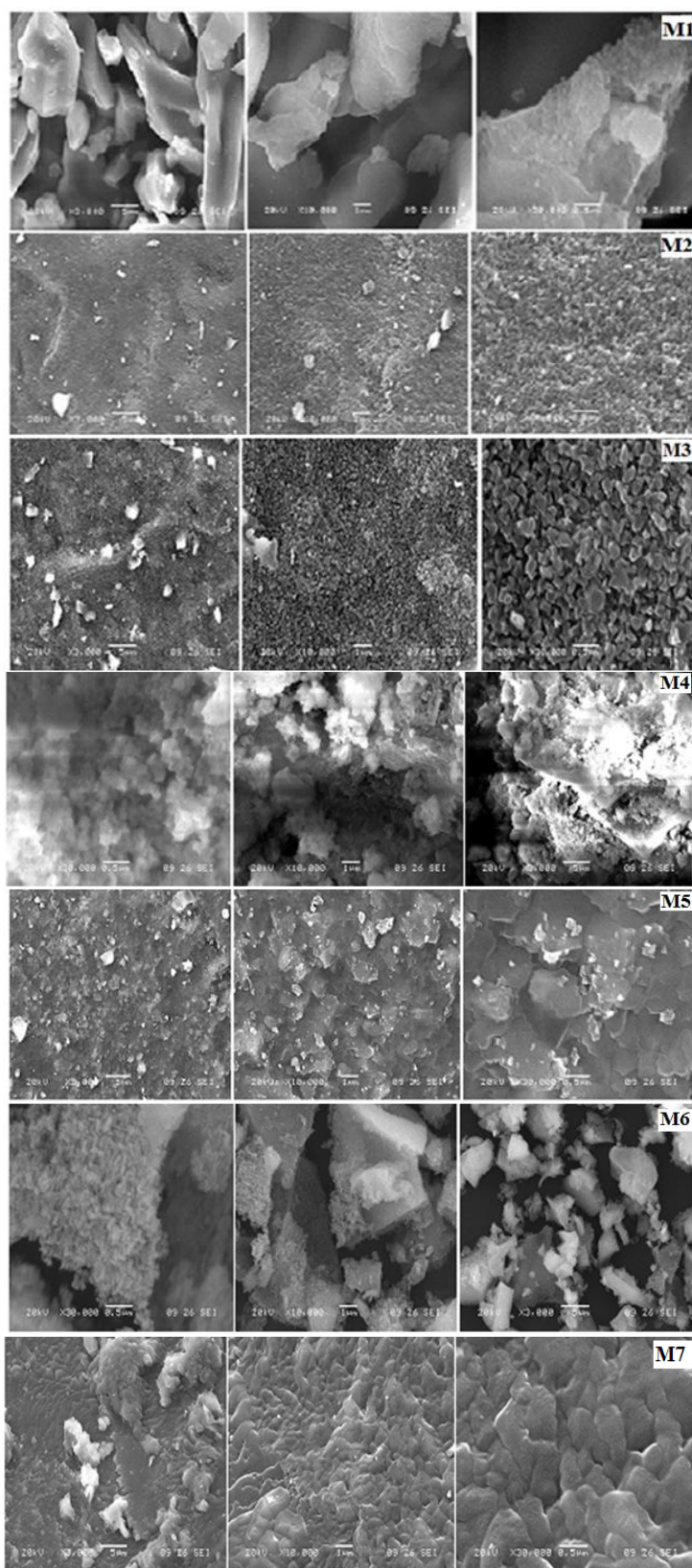


Fig. 2. SEM images of calcined samples M1-M7

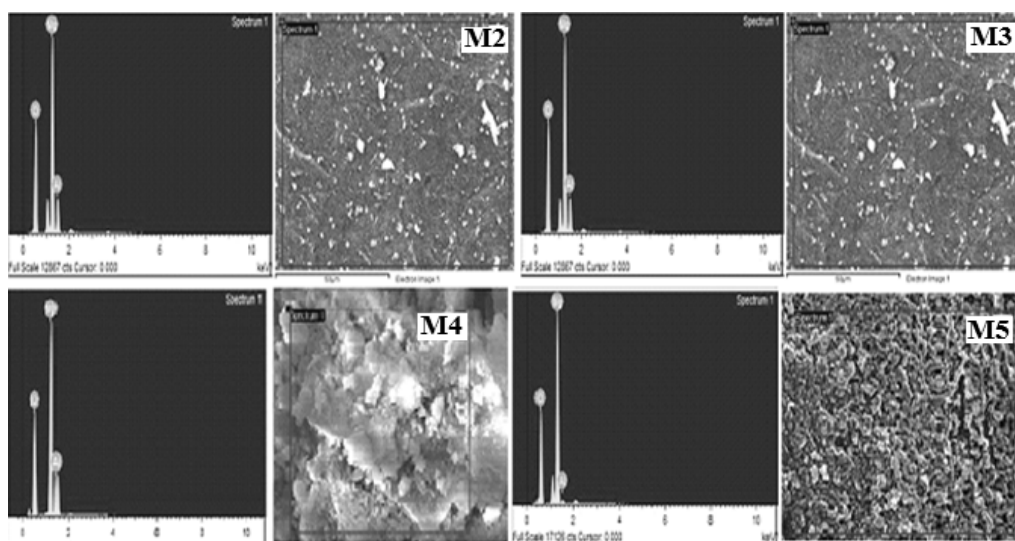


Fig. 3. EDS analysis of calcined samples M2-M5

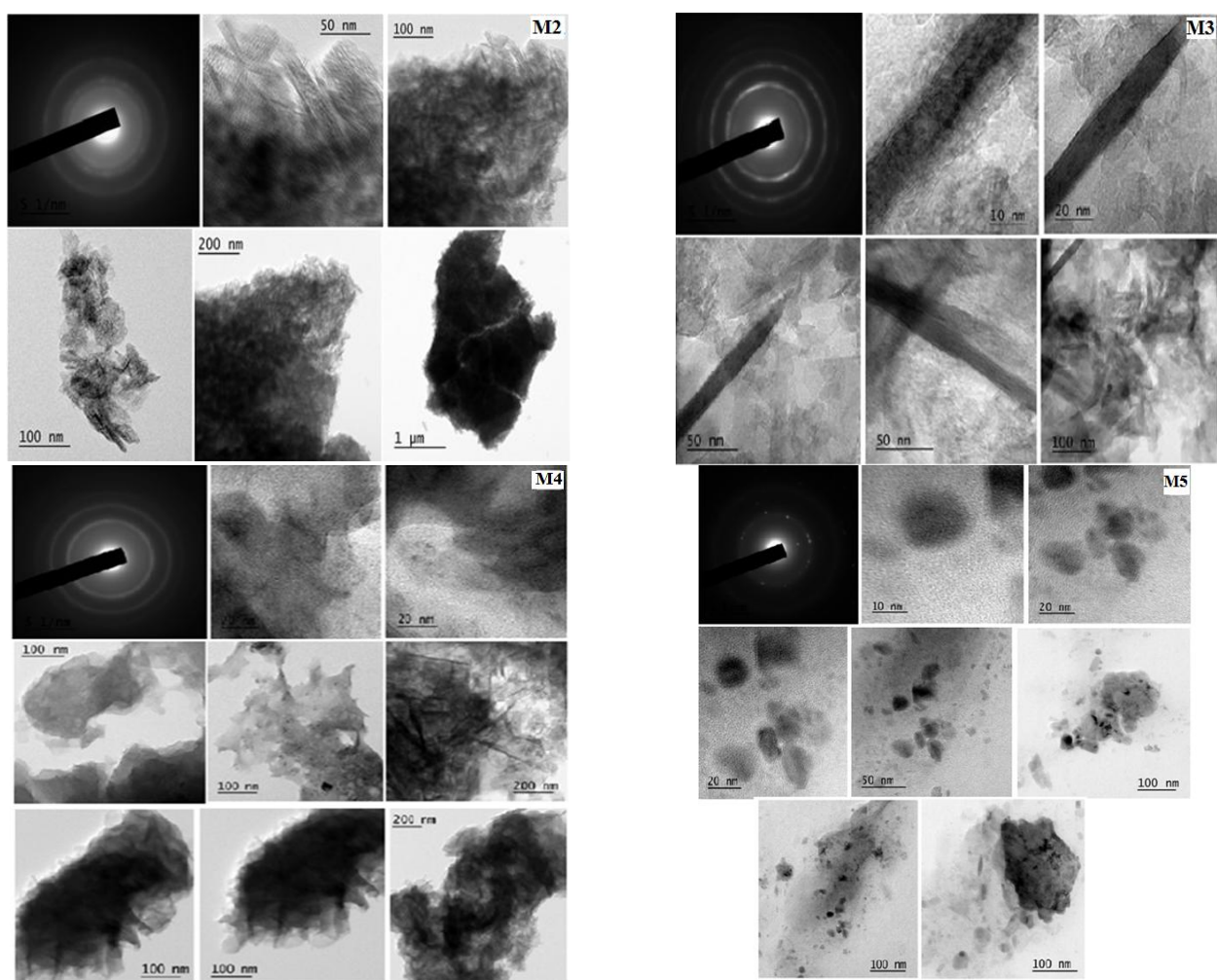


Fig. 4. TEM images of calcined samples M2-M5

Table 7

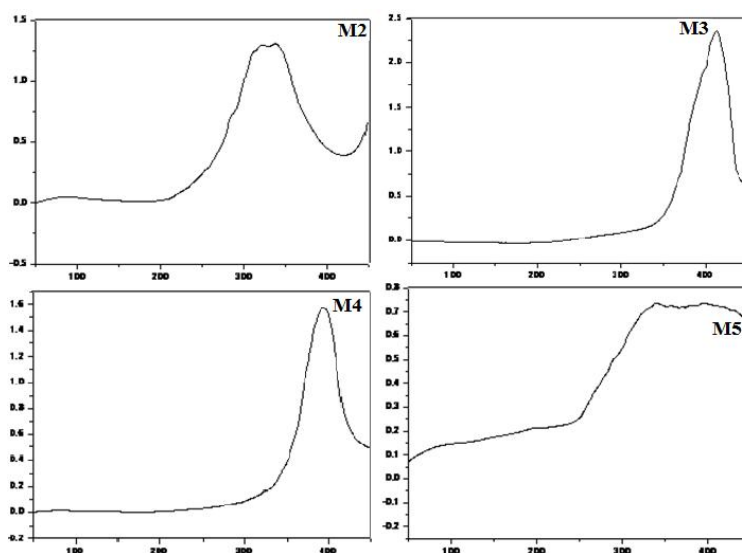
BET analysis results

Sample	BET area	Pore volume, cm ³ /g	Pore diameter, Å
M2	20.3410	0.044951	44.1973
M3	18.6384	0.029568	31.7285
M4	7.2702	0.086086	237.175
M5	2.3093	0.007617	65.9707

Table 8

Basicity measurements

Samples	CO ₂ -TPD, m·mol/g
M2	1.5334
M3	1.7536
M4	3.3527
M5	0.7204

Fig. 6. CO₂-TPD of calcined samples M2-M5

4. Conclusions

The present work described the synthesis details of Mg-Al mixed metal oxides. The prepared mixed metal oxides were characterized by X-ray diffraction (XRD), energy dispersive analysis (EDS), scanning electron microscopy (SEM), transition electron microscope (TEM), BET surface area and CO₂-TPD area. It has been found that the precursor preparation conditions, such as the heat treatment temperature and reaction time have a significant effect on the morphology of the combined oxide powders. The sample M4 (MgO-Al₂O₃, 0.8:0.2) has high stability; good crystallization, the best powder morphology and average particle size of about 10 nm. More importantly, the catalysts did not undergo deactivation during the reaction and finally became stable.

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Al₂O₃ НАНЕСЕНИЙ НА MgO: НОВИЙ, ЕФЕКТИВНИЙ І ВІДНОВЛЮВАЛЬНИЙ КАТАЛІЗАТОР СИНТЕЗУ ХАЛЬКОНІВ

Анотація. Методом ко-осадження вивчено ряд змішаних оксидів металів Mg-Al, фізико-хімічні властивості яких визначені за допомогою методів PCA, PEM, EPC, TEM, BET та CO₂-ТДС. Розроблено ефективний та простий метод синтезу хальконів з використанням різних альдегідів та ацетофенону за допомогою оксиду Mg-Al за умов рефлюксу в етанолі, що забезпечує відмінну продуктивність. Доведено відновлюваність каталізатора, а також здатність MgO до диспергування та підвищення основності, розміру пор і каталітичної активності.

Ключові слова: оксид Mg-Al, екологічний каталіз, відновлюваність, багатокомпонентна реакція.