

# Magnesia Synthesis via Sol–Gel: Structure and Reactivity<sup>†</sup>

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2-Propanol decomposition at 200 °C on magnesium oxide prepared by the hydrolysis of magnesium ethoxide (sol–gel method) is reported. With hydrochloride acid, acetic acid, or ammonia as hydrolysis alkoxide catalysts, the acidity and the selectivity to propene of the catalysts follow the sequence, MgO (HCl) > MgO (acetic acid) > MgO (ammonia). In contrast, the basicity of the samples, defined as the ratio dehydrogenation rate/dehydration rate, is found to be depending on the particle size of the catalysts. The basicity trend found is MgO (acetic acid) > MgO (ammonia) > MgO (HCl) and it correlates well with the particle size determined by the Rietveld refinement.

## Introduction

Basic catalysts like magnesium oxide were traditionally prepared by the thermal decomposition of the corresponding mineral salts. Magnesium carbonate decomposition at temperatures higher than 500 °C is the most common method reported for the preparation of magnesium oxide. However, in the resulting solids the residual acidity, particle size as well as the reactivity is determined by the thermal treatments applied to the carbonate decomposition. These are disadvantages that should disqualify it as a good performance catalyst. However, varying the synthesis method, it should be possible to overcome the disadvantages and to use MgO as an innovative catalysts.

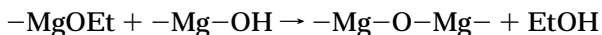
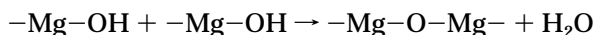
In this work we have chosen a nonconventional synthesis technique, the sol–gel technique<sup>1</sup> which has offered advantages such as high purity, high specific surface area, and homogeneous final solids when applied to the preparation of SiO<sub>2</sub>,<sup>2,3</sup> Al<sub>2</sub>O<sub>3</sub>,<sup>4</sup> TiO<sub>2</sub>,<sup>5</sup> and MgO.<sup>6</sup>

The sol–gel synthesis method is described, essentially by the following reactions<sup>7</sup>

hydrolysis



condensation



In these reactions, ethanol is used as solvent, but it is also a reaction byproduct. As the hydrolysis is almost simultaneous to polymerization, the intermediary ethoxy

(–Mg–OEt) and hydroxy (–Mg–OH) groups coexist. The hydroxylation of MgO precursors is expected to depend on the pH values of the reaction solution. When the gels are thermally treated, the magnesium hydroxide (brucite) is dehydroxylated until magnesium oxide is obtained. In previous work it has been shown that pH and water concentrations can be used to control hydrolysis and polymerization.<sup>8,9</sup>

In the present work MgO has been synthesized at various pH values using different hydrolysis catalysts. The role of the hydrolysis catalysts is expected to be crucial in the textural and hence in the catalytic properties of the final solid. Our catalysts were prepared by the hydrolysis of magnesium diethoxide using three different hydrolysis catalysts: hydrochloride acid, acetic acid, and ammonia. The solids were characterized by FTIR spectroscopy, ammonia thermodesorption, X-ray diffraction, and the catalytic activity for the 2-propanol decomposition.

## Experimental Section

**Catalyst Preparation.** The catalysts were prepared by refluxing 5.72 g of Mg(OEt)<sub>2</sub> with 4.5 mL of water and 50 mL of ethanol. The hydrolysis of the magnesium alkoxide was accomplished by adding to the refluxed solution 0.60 mL of HCl (pH 3), by adding 2.00 g of acetic acid (pH 5), or by adding 3.0 mL of NH<sub>4</sub>OH (36 vol %, pH 9). The homogeneous solutions were maintained in reflux until the gel is formed. Before characterization the MgO samples were dried at 70 °C for 12 h and then calcined in air during 4 h at 300, 600, and 900 °C.

**Catalysts Characterization.** The BET area was calculated from the adsorption isotherm using nitrogen as adsorbant (Micromeritics Sorptometer). Acidity was analyzed by ammonia pulse chemisorption using a conventional static adsorption system. The samples, evacuated at 500 °C for 1 h, were then exposed to ammonia gas at 200 °C. A total acidity was then obtained from the amount of adsorbed ammonia. FTIR characterization was done using a Nicolet MX-1 apparatus; pressure was applied to the solid powder until the pellet was transparent.

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**Table 1. Gelation Time and Acidity for the Various MgO Sol-Gel Catalysts**

	gelation time (min)	acidity (NH <sub>3</sub> mequiv/g)
MgO (HCl)	89	0.047
MgO (acetic)	48	0.032
MgO (ammonia)	29	0.018

X-ray powder diffraction data were obtained at room temperature with a Siemens D-5000 diffractometer. Rietveld analysis was done in the conditions reported elsewhere.<sup>10</sup> Catalytic activities in the 2-propanol decomposition were determined in flowing hydrogen in a conventional continuous flow microreactor system coupled to a gas chromatograph. Typically, tests were done using 20–30 mg of catalyst sample reactivated in situ at 500 °C. The gas carrier subsequently feeds the reactant by a saturator system. The 2-propanol conversion was performed at 200 °C using a reactant partial pressure of 22.4 Torr. Under these conditions the only detected products were acetone and propene.

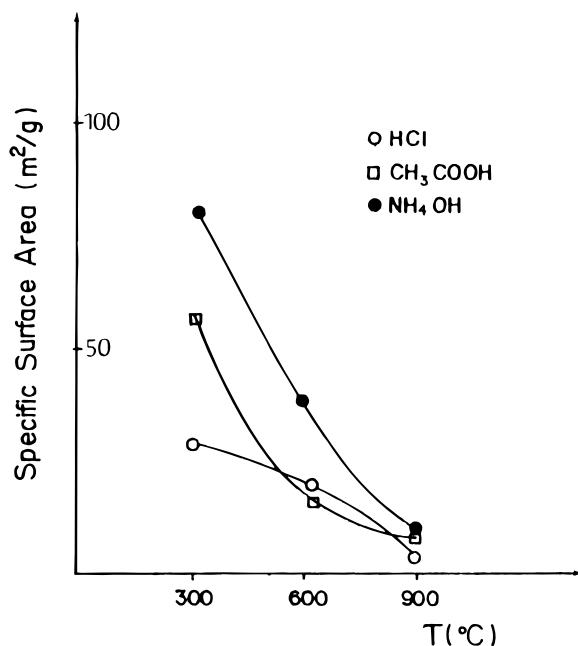
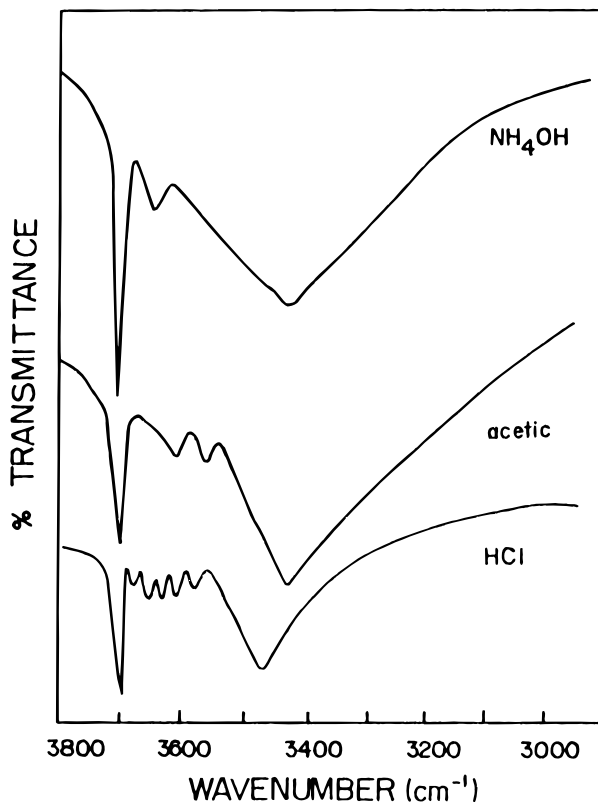
### Results

The obtained solids are white powders whose gelation rate depends on the pH value used during the hydrolysis step. A basic pH promotes condensation and the geling time is 29 h. In contrast, when acid pH (HCl) is used, hydrolysis is promoted and condensation is diminished, in this case the gel is obtained after 89 h (see Table 1.) The effect of the pH on the specific surface area is of crucial importance. The catalyst prepared at basic pH shows the highest surface area, while the preparations at acid pH show the lowest values. Thermal treatments diminish the specific surface area and low values are obtained when the samples are treated at 900 °C, Figure 1.

Acidity of the samples treated at 600 °C are reported in Table 1. The values show that all the samples are low acidity ones. Values of 0.047–0.018 NH<sub>3</sub> mequiv/g are obtained, and they are very low if they are compared with those traditionally reported for alumina (0.2–0.4) or zeolites (0.4–0.8). The low acidity reported for magnesia catalysts is also found in the sol-gel samples.<sup>11</sup>

FTIR spectroscopy studies of the sol-gel MgO show that in samples treated at 70 °C (fresh samples) OH groups generate infrared vibrations located in the high energy zone of the spectra (4000–3000 cm<sup>-1</sup>), Figure 2. All the samples show an intense band at 3699 cm<sup>-1</sup> which is attributed to an OH stretching frequency. This band has been previously characterized by Kirklin et al.<sup>12</sup> and assigned due to the Mg(OH)<sub>2</sub> (brucite) present in the oxide, Figure 2. If the solid is prepared at pH 3 (HCl), five adsorption bands located at 3682, 3652, 3624, 3603, and 3574 cm<sup>-1</sup> are clearly resolved. The five vibration bands reported here are due to –Mg–OH groups. Indeed, the hydrolysis rate is higher than the condensation rate. This last remark explains how nonpolymerized hydroxy species exist even after the gelation point. Lastly, at 3475 cm<sup>-1</sup> a broad and intense band has to be assigned to OH stretching vibrations due to water and ethanol occluded into the sample during the polymerization.

If pH is increased to 5 (acetic acid), the number of bands decreases because the hydroxylation in the final oxide is lower. At higher pH values (pH 9) the Mg(OH) band is observed, as well as another one at 3649 cm<sup>-1</sup> caused by the hydroxylation of the solid. A shifted band due to the occluded water is seen at 3384 cm<sup>-1</sup>.

**Figure 1.** Specific surface area of the various MgO sol-gel catalysts as a function of the temperature treatment.**Figure 2.** FTIR spectra of the various MgO sol-gel catalysts.

Thermal treatment of the samples shows a notable decrease in the intensity of the Mg–OH bands. At 600 °C, only one band is recorded in all the samples. At 900 °C no hydroxylation band is observed.

The evolution of the crystalline phase in MgO sol-gel catalysts as a function of the temperature was followed by X-ray diffraction analysis and the figures are reported in recent paper.<sup>10</sup> Using these data for the Rietveld refinement, we have obtained the lattice parameter reported in Table 2. The results show that the average particle size increases with temperature. If we assume the existence of OH<sup>-</sup> near the vacant Mg sites in the lattice,

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**Table 2. Average Particle Size (Å), Mg:O Molar Ratio, and Lattice Parameter in Å as a Function of Temperature and Hydrolysis Catalysts for MgO Sol–Gel Catalysts**

	300 °C	600 °C	900 °C
Particle Size (Å)			
MgO (HCl)		1518(25)	1555(26)
MgO (acetic)	332(7)	396(8)	634(10)
MgO (ammonia)	198(4)	644(15)	874(28)
Mg:O Molar Ratio			
MgO (HCl)		3.79(2):4.0	3.78(2):4.0
MgO (acetic)	3.69(1):4.0	3.75(1):4.0	3.79(1):4.0
MgO (ammonia)		3.82(2):4.0	3.85(3):4.0
Lattice Parameter			
MgO (HCl)		4.21032(2)	4.21022(2)
MgO (acetic)	4.21663(7)	4.21149(5)	4.21058(4)
MgO (ammonia)		4.21179(9)	4.21014(8)

**Table 3. Catalytic Activity and Selectivity in the 2-Propanol Conversion at 200 °C for the Various MgO Sol–Gel Catalysts Treated at 600 °C**

catalyst	% C <sup>a</sup>	% C (m <sup>2</sup> /g)	$V_{oa}/V_{op}$ <sup>b</sup>	selectivity <sup>c</sup> (mol %)	$K_d$ <sup>d</sup>
MgO (HCl)	18.5	1.80	0.1	93.3	12.6
MgO (acetic)	0.6	0.04	30.3	15.6	49.2
MgO (ammonia)	2.1	0.07	27.8	3.5	3.5

<sup>a</sup> Total conversion. <sup>b</sup> Dehydrogenation/dehydration rate. <sup>c</sup> Selectivity to propene. <sup>d</sup> Constant deactivation.

**Table 4. Catalytic Activity and Selectivity in the 2-Propanol Conversion at 200 °C for the Various MgO Sol–Gel Catalysts Treated at 900 °C**

catalyst	% C <sup>a</sup>	% C (m <sup>2</sup> /g)	$V_{oa}/V_{op}$ <sup>b</sup>	selectivity <sup>c</sup> (mol %)	$K_d$ <sup>d</sup>
MgO (HCl)	7.3	0.82	0.1	97.1	18.6
MgO (acetic)	0.4	0.10	19.6	17.9	34.0
MgO (ammonia)	1.3	0.13	52.2	1.4	4.0

<sup>a</sup> Total conversion. <sup>b</sup> Dehydrogenation/dehydration rate. <sup>c</sup> Selectivity to propene. <sup>d</sup> Constant deactivation.

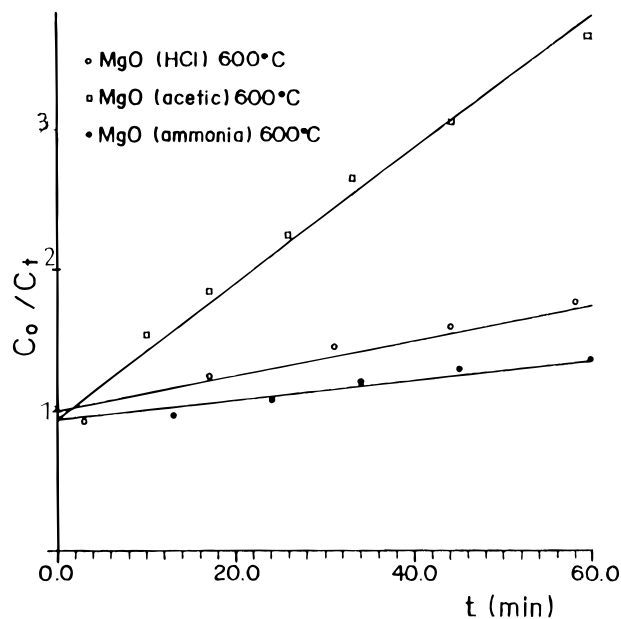
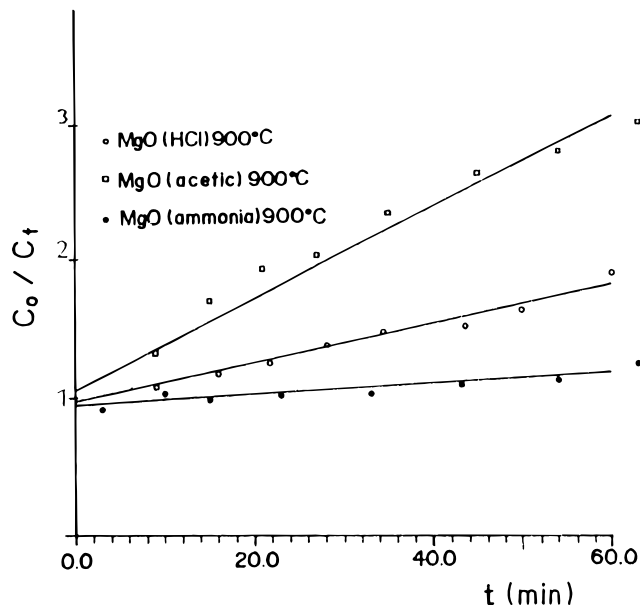
as was proposed by Derouane et al.,<sup>13</sup> then an increase in the particle size will give rise to a loss of OH<sup>−</sup> ions from the lattice, augmenting simultaneously with the Mg:O molar ratio. From Table 2 we also conclude that the MgO lattice contracts as the OH<sup>−</sup> ions leave it.

Activity results in the 2-propanol decomposition are reported in Tables 3 and 4 for samples treated at 600 and 900 °C, respectively. At both temperatures the hydrochloride preparation shows a very high activity. It is around 8 times higher than that shown by the samples prepared with ammonia and between 30 and 18 times higher than the corresponding activity shown by the samples prepared in acetic medium.

Some deactivation was noticed as a function of time on stream. To calculate the deactivation constant of the reaction, the second-order law described in detail in previous works for sol–gel catalysts is applied here.<sup>14,15</sup> We used the equation

$$C_0/C_t = 1 + k_d t$$

where  $C_0$  and  $C_t$  are the conversion at time zero and the conversion at time  $t$ , respectively. The slope obtained by plotting  $C_0/C_t$  versus time ( $t$ ) gives the deactivation constant  $k_d$  (Figures 3 and 4). The results of Tables 3 and 4 show that the acetic preparations are those which

**Figure 3.** Catalyst deactivation constant for the 2-propanol decomposition at 200 °C of the various MgO sol–gel catalysts treated at 600 °C.**Figure 4.** Catalyst deactivation constant for the 2-propanol decomposition at 200 °C of the various MgO sol–gel catalysts treated at 900 °C.

deactivate faster. Deactivation values of 12.6, 49.2, and 5.5 for HCl, acetic, or ammonia preparations are observed in Table 3 for samples treated at 600 °C. In Table 4 the deactivation values for the samples treated at 900 °C are reported. The  $k_d$  values increase in the thermal treated samples for acetic and HCl preparations. For ammonia the  $k_d$  value is almost constant.

The selectivity to the formation of propene obtained during the 2-propanol decomposition is reported in Tables 3 and 4 in mole percent. It can be seen that a high value corresponds to the HCl preparation (93.3% of propene). Nevertheless, when acetic acid or ammonia are used as gelation catalysts, the selectivity to propene is very low. In the last case (ammonia) a high formation of acetone (96.5% of acetone formation) can be observed.

The initial rates for the dehydrogenation  $V_{oa}$  and dehydration  $V_{op}$  reactions were calculated and reported as the ratio  $V_{oa}/V_{op}$  in Tables 3 and 4 for 600 and 900 °C

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thermal treated samples, respectively. It can be seen that the higher ratio corresponds to the acetic and ammonia preparations.

### Discussion

Catalyst characterization shows that the specific surface area of the magnesia sol-gel samples strongly depends on the hydrolysis catalysts used and on the thermal treatment (Figure 1). The lowest areas correspond to the samples prepared using acetic or hydrochloride acid. On the other hand, if ammonia is used the specific area shown by the samples is the highest in all the range of thermal treatment studied.

Thus, the hydrolysis catalysts lead to the formation of low hydroxylated samples with base and to high hydroxylated samples with acid. A fast hydrolysis rate occurs at acid pH, producing a high amount of Mg-OH species. At pH 3, the surface is hydroxylated as shown by FTIR spectroscopy, Figure 2. This effect can be explained by a mechanism in which the alkoxide group in the acid environment is easily protonated. Thus a positive charge is generated to give rise to a transition state. The seven hydroxylation FTIR bands observed in HCl (Figure 2) reduce to only two bands for acetic acid and only one for ammonia. Considering the importance that the hydroxyl groups can play toward complete dehydration of the reactant, we shall try to relate this high hydroxylation of the HCl prepared catalyst to its high activity and selectivity toward propene in the following section.

When the pH value reaches 5, the hydroxylation degree is lower (less FTIR bands in the high-energy region). As already mentioned, when acetic acid is used, the Mg(OH) characteristic band is present with only two low-intensity bands. The latter are shifted toward the low energy and can be assigned to nonpolymerized Mg-OH groups. A fourth broad band is due to the water and ethanol occluded into the gel. In contrast in basic media, the hydrolysis is low as shown in Figure 2. One single band is observed in the high-energy region. Comparing the results of Figure 2 and Table 2, it can be seen that the particle size of the samples can be correlated with the highest hydroxylation degree.

The hydrolysis catalysts therefore have also an important effect in the particle size of the solid. In Table 2, it can be seen that a quite different particle size can be obtained as a function of the catalyst used. When HCl acid is used, large particle sizes are obtained showing thermal resistivity. However, when the samples are prepared using acetic acid or ammonia, important sintering is observed by increasing the temperature. The loss in specific surface area must then be larger in the acetic or ammonia preparations than in the HCl one. From Figure 1, the loss of specific area when the samples are compared at 300 and 900 °C can be calculated. They correspond to a loss of specific area of 55% for ammonia preparation, a loss of 87% for acetic, and a loss of only 30% for the HCl preparation.

The effect of the hydrolysis catalyst in the Mg:O ratio is also reported in Table 2. The table shows that all the MgO samples are magnesium-deficient materials; for each series the Mg:O ratio increases as a function of the temperature. For the HCl preparation the value is almost constant; note that these samples show only a slight modification of their particle size (Table 2), if they were treated either at 600 or at 900 °C. This correlation shows that the Mg:O ratio is dependent on the particle size in each series of catalysts. Large particles are the least magnesium-deficient materials. The lattice parameters for each hydrolysis catalyst notably diminish when the particle size increases.

The catalytic properties of such sol-gel catalysts are reported in Tables 3 and 4. It can be seen that the samples with the highest activity are the samples prepared at acid pH using hydrochloride acid as hydrolysis catalyst. On the other hand, it can be seen in Table 2 that such catalysts show the largest particle size.

The activity shown by the acetic preparation is lower than that shown by the ones with the ammonia preparation. It must be noted that the small particle size corresponds dominantly to the acetic samples. Thus, we can say that the activity values can be correlated with the particle size of the samples and such behavior is also observed in the HCl preparation. On the other hand, a correlation between the activity and the acidity of the samples cannot be found; since the activity sequence shown by the samples is  $\text{HCl} > \text{NH}_4\text{OH} > \text{acetic acid}$ , the acidity sequence is as follows:  $\text{HCl} > \text{acetic acid} > \text{NH}_4\text{OH}$ . Effects of specific surface area on the trend of activity and acidity are of minor importance, since the activity values per square meter (Tables 3 and 4) do not noticeably modify the behavior discussed above. These results are of great importance, since they show that the 2-propanol decomposition on magnesium oxide sol-gel catalysts is a particle-size-dependent reaction.

2-Propanol decomposition is a reaction which essentially gives propene and acetone as main products. The selectivity to propene is a reaction which strongly depends on the acidity of the catalysts,<sup>16-18</sup> while the selectivity to acetone is reported as a reaction which depends on both acid and basic sites.<sup>19-22</sup> The results reported in Tables 3 and 4 show that the highest selectivity to propene (93.3 and 97.1 mol % of propene) corresponds to the samples prepared using HCl. On the other hand when acetic acid was used as the hydrolysis catalyst, the selectivity goes down (15.6 and 17.9). The lowest selectivity corresponds to the ammonia preparations (3.5 and 1.4%). The highest selectivity corresponds to the catalysts showing high acidity and simultaneously large particle size. These results show that the propene formation strongly depends on the total acidity of the catalysts as reported in a great number of papers.<sup>16-18</sup> Comparing the selectivities of HCl and acetic acid preparation, however, we see a large difference in their selectivities. As their acidities are not too different, we propose that the much larger particle sizes for the former is a dominant factor for this difference.

Using the dehydrogenation rate/dehydration rate ratio ( $V_{\text{oa}}/V_{\text{op}}$ ) proposed by Ai,<sup>23</sup> we can determine the "basicity" of the catalysts. Surprising results are observed in Tables 3 and 4. Acetic preparations show a "basicity" comparable to that obtained in ammonia samples, even when the acetic preparation shows a total acidity higher than that of the basic preparation. It seems then that the use of the concept of "basicity" as defined by Ai<sup>23</sup> in our case implies some adjustments. In our study the "basicity" concept requires additional effects to be taken into account, such as particle size effect. Note that the highest "basicity" corresponds to the catalysts showing the lowest particle size (acetic acid and ammonia preparations).

Deactivation was determined in the magnesium oxide sol-gel samples. It can be seen in Tables 3 and 4 that the

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deactivation constant  $k_d$  of the various catalysts depends on the specific surface area shown by them. The highest deactivation value is obtained in acetic preparations and this sample shows the lowest specific surface area, Figure 1. 2-Propanol decomposition produces water as a product of the dehydration reaction and it has been reported that water inhibits such reaction.<sup>24</sup> The water formed during the dehydration reaction is adsorbed on the catalyst surface; thus its effect must be stronger in catalysts showing the low specific area.

### Conclusions

It is shown that in magnesium oxide catalysts prepared by the hydrolysis of magnesium ethoxide, the catalyst used for the hydrolysis has a strong effect in the textural, structural and catalytic properties. The main results can be summarized as follows:

1. FTIR results show that acid hydroxylation catalysts produce highly hydroxylated MgO, specially in the HCl case. In this case the hydrolysis of magnesium alkoxide is favored, instead of the condensation reaction. By use of the X-ray diffraction data for the Rietveld refinement, it can be shown that the particle size of the solids is substantially larger in HCl preparations vis-a-vis the ammonia or acetic ones. For each hydrolysis catalysts, a magnesium deficient ratio Mg:O comprised between 3.69:4.00 and 3.85:4.00 is observed.

2. Activity in the 2-propanol decomposition by MgO shows that this reaction has a much higher activity for HCl-prepared samples than for the others. This seems to be related not so much to acidity as to the much large particle size and the higher degree of hydroxylation of the HCl-prepared samples.

3. The "basicity" of the catalysts determined by their hydrogenation rate/dehydration rate ratio is found to be dependent on particle size rather than on the acidity of the different samples. The high surface area of the ammonia-prepared samples makes their deactivation constant to be quite low as would be expected. It is however, surprising that the deactivation constant for acetic-prepared samples is the highest. Again this cannot be attributed to differences in acidity but rather on the notable difference of the particle size for the acid catalysts.

4. Selectivity to propene is found for the HCl acid samples. This selectivity, however, is only mild for the acetic preparations. For the HCl preparations, the selectivity to propene becomes extremely high, 97%, an unpredicted value for MgO. This added to high activity and relatively small deactivation constant make the HCl sol-gel method very promising for preparing MgO catalysts.

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