Catalytic Performance of Li/Mg Composites for the Synthesis of Glycerol Carbonate from Glycerol and Dimethyl Carbonate

Zhenmin Liu,* Bin Li, Fengrui Qiao, Yan Zhang, Xiaoxiao Wang, Ziyuan Niu, Jin Wang, Haiqiang Lu, Shen Su, Ruili Pan, Yuanyang Wang, and Yongbing Xue

ABSTRACT: A series of Li/Mg composites were synthesized by the coprecipitation method using magnesium and lithium nitrates, and then used for the synthesis of glycerol carbonate (GC) from glycerol and dimethyl carbonate (DMC). The experimental results indicated that Li/Mg composites were prospective catalysts for GC synthesis. 92.05% glycerol conversion and 90.61% GC yield were obtained after reacting at 80 °C for 2 h in the presence of Li/Mg composites. The structure and properties of Li/Mg composites were characterized by X-ray diffraction (XRD), Fourier transform infrared (FT-IR), Brunauer−Emmett−Teller (BET), and CO2−temperature-programmed desorption (TPD) techniques. It was inferred that the basic strength and basicity of Li/Mg composites were improved with increase in Li content. It was concluded that Li2CO3 was the main reactive species. A too-strong basic strength of Li/Mg composites could facilitate the glycerol conversion but impair GC selectivity.

INTRODUCTION

Biodiesel production from vegetable−animal fats and higher alcohols has been attracting increasing attention, since it is a prospective substitute to fossil fuel. However, 10 wt % of glycerol is formed as an undesired co-product in the production of biodiesel, which not only abates the atom economy of this production process but also results in the concentration of glycerol, and further has a negative impact on the balance of the glycerol market.1,2 Thus, it is especially advisable to explore new applications for glycerol or transform it into useful derivate. Several important chemicals, such as propane diols, acrolein, glyceric acid, and ethers, have been synthesized from glycerol via oxidation, dehydrogenation, esterification, etherification, polymerization, etc.3,4

Glycerol carbonate (GC) is an important glycerol derivate in the chemical industry. Because of its excellent properties, such as low toxicity, low vapor pressure, and high boiling point, GC has been widely used as a novel component of biolubricants, and as a solvent for coatings, cosmetics, and detergents. Additionally, GC can also be used for the intermediate synthesis of value-added glycidol, and for new functionalized polyesters and polyurethanes.5

Considering the safety and convenience of the production route, the reaction of glycerol with dimethyl carbonate (DMC) was supposed to the mildest and prospective method for GC synthesis in comparison with carbon dioxide, carbon monoxide, urea, and diethyl carbonate.1,6,7 The catalysts have played an important role in GC synthesis. There are three main types of catalysts: acidic, basic, and enzyme catalysts, which can be used in the reaction of glycerol with DMC. However, the basic catalysts are more attractive due to their faster speed than acid catalysts and lower price than enzyme catalysts for glycerol transformation.8

The composite solid bases are one important kind of solid base catalyst, which are formed from more than two kinds of metal compounds. In the synthesis of composite solid base catalysts, a considerable number of composites take shape because of the strong interaction of the components. Especially, the calcination of the composite solid base catalysts at high temperature induces dehydration and decomposition, forming composite oxides with a poorly crystallized structure, large specific surface area, and basic properties, which are attributed to the different O2−−Mn+ acid−base pairs and O2−anions.9,10 Thus, they are potential for catalytic support and heterogeneous basic catalysts.

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Zheng et al. synthesized the Ca–Al hydrocalumite for the reaction of glycerol with DMC at 70 °C for 3 h, and obtained 93% conversion of glycerol and 97% selectivity of GC. It indicated that the conversion of glycerol depended mainly on the number of strong basic sites of the calcined Ca–Al catalysts and that the Ca₃Al₁₄O₃₃ phase in the calcined catalysts was stable, but CaO was lost in the recycle experiments and thus brought about deactivation. Alvarez et al. used Mg/Al mixed oxides as a solid base catalyst for GC synthesis from glycerol at 130 °C for 10 h and obtained 65% of GC yield. Marimuthu et al. prepared the Cu/Mg/Al mixed-oxide catalysts for GC synthesis and obtained 91.2% of GC yield. Additionally, it was proved that the appropriate Cu molar ratio and strong surface basicity were responsible for the catalytic performance. Furthermore, it was the incorporation of metal ions, lithium introduction into MgO is considered to create the strongest basic sites.

Moreover, it has been reported in the literature that lithium incorporation into solid catalysts could increase the catalysts’ basicity and further enhance the catalytic performance. Compared with other alkali (Na, K, and Cs) or alkaline earth–metal ions, lithium’s introduction into MgO is considered to create the strongest basic sites.

As far as we know, there are few reports about the application of Li/Mg composites for GC synthesis from glycerol. In this work, a series of Li/Mg composites was prepared by a coprecipitation method, and their catalytic performance was studied in synthesis of GC from glycerol. Furthermore, the physicochemical properties of Li/Mg composites were studied using X-ray diffraction (XRD), Fourier transform infrared (FT-IR), Brunauer–Emmett–Teller (BET), and CO₂-temperature-programmed desorption (TPD). The relationship of the catalysts’ basic properties with their catalytic activities was also discussed.

### RESULTS AND DISCUSSION

**Product Analysis.** The FT-IR spectra of the synthesized GC are shown in Figure 1(2). It was seen that the peak at 3200–3500 cm⁻¹ belonged to the stretching vibration of the –OH group. The appearances at 2930, 2860, and 1460 cm⁻¹ were attributed to the absorption peak of the –CH₂– group. The band at 1050 cm⁻¹ displayed the existence of C–O valence vibrations. Based on the above analysis, it was shown that the –CH₂OH group existed in the synthesized product. Furthermore, it was demonstrated that the bands at 1800, 1181, and 1086 cm⁻¹ were the characteristics of the five-membered cyclic carbonate group. Moreover, the standard sample of GC was scanned by the infrared spectrometer and its FT-IR spectra are displayed in Figure 1(1). Compared with the FT-IR spectra of the standard sample, the synthesized product might be the desired GC.

**Figure 2** shows the mass spectra of the GC product. It was reported that the ratio of mass to charge at 31 and 87 was attributed to the hydroxymethyl group and cyclic carbonate group, respectively, because GC was bombarded by the strong flow of electrons and then decomposed into pieces. This result was in accordance with the FT-IR analysis. The main peak in the ratio of mass to charge at 44 corresponded to the CO₂ molecule, due to the further crack of the cyclic carbonate group. In one experiment, it was found that CO₂ gas escaped from the synthesized compound after stewing for several days in atmospheric environment, which might prove the appearance of the main peak at the ratio of mass to charge of 44.

Based on the analysis of FT-IR and MS spectra, it was inferred that the synthesized product was the desired GC, and that the synthesized Li/Mg composites could act as a prospective catalyst for the reaction of glycerol with DMC.

**Characterization of Li/Mg Composite Catalysts.** The XRD patterns of the composite catalysts with various molar ratios of Li to Mg are displayed in Figure 3. For none of the composite catalysts were any characteristic diffraction peaks about the raw materials detected by XRD characterization, which indicated that these raw materials reacted completely. Moreover, some newly formed characteristic diffraction peaks were found to have emerged. The characteristic diffraction peaks at 2θ = 22.834, 29.432, 38.996, 42.500, 46.649, 47.996, 48.427, 55.568, and 56.515° were ascribed to the newly formed NaNO₃ [PDF 85-0850] species from the reaction of Na⁺ and NO₃⁻ in the mixed solution. The characteristic diffraction peaks at 2θ = 32.557, 42.608, 62.381, and 66.584° were attributed to the produced MgCO₃ [PDF 86-2344] species.

**Figure 1.** FT-IR spectra of the standard sample (1) and the synthesized sample (2) of GC.
The diﬀraction peaks at 2θ = 21.379, 29.461, 30.647, 31.799, 34.095, and 36.034° belonged to the Li2CO3 [PDF 87-0728] species. In addition, the diﬀraction peaks of Mg(OH)2 species were found at 2θ = 18.636, 38.049, and 50.928°.15 According to the above analysis, it was inferred that the synthesis reaction occurred and produced NaNO3, MgCO3, and Li2CO3 species during the preparation of the composite catalysts. The emergence of the Mg(OH)2 species was probably ascribed to the hydrolysis of the newly formed MgCO3 in the basic condition.16

As seen in Figure 3, the diﬀraction peak intensities of Mg(OH)2 and Li2CO3 were obviously increased with increasing Li content, but the diﬀraction peak intensity of NaNO3 was unchanged basically. This result illustrated that the basic species content of the composite catalysts was improved with increasing ratio of Li to Mg. Bai et al.17 studied the repeatability of the MgO catalyst for GC synthesis and concluded that the activity decrease of MgO is attributable to its conversion to MgCO3 and Mg(OH)2. This result showed that MgCO3 and Mg(OH)2 had lower activity as compared with MgO, and further proved that they were not the main reactive species for GC synthesis. Moreover, in our experiment, it was found that the glyceral conversion was 90, 32, 47, and 0%, respectively, when the pure Li2CO3, MgCO3, Mg(OH)2 and NaNO3 were used as catalysts for GC synthesis at the same reaction conditions. Thus, it was concluded that Li2CO3 was the main reactive species, and MgCO3 and Mg(OH)2 were secondary for GC synthesis.

Figure 4 shows the spectra of the composite catalysts with various molar ratios of Li to Mg. The characteristic peak at 3700 cm−1 was attributed to the stretching vibration of the hydroxyl group on the catalysts, which might have come from the newly formed Mg(OH)2, or the absorbed H2O on the catalyst surface. The bands that emerged at 842, 1018, and 1440 cm−1 were ascribed to the formation of the MgCO3 species.18 The appearances at 510 and 867 cm−1 were attributed to the Li2CO3 species.19 A band belonging to the LiNO3 species was detected at 1380 cm−1.20 Thus, Mg(OH)2, MgCO3, Li2CO3, and LiNO3 species were detected by FT-IR on Li/Mg composite catalysts, which was consistent with the XRD characterization.

The CO2-TPD technique was used to characterize the basic properties of the composite catalysts. The higher the CO2 desorption temperature, the stronger the basic sites. The CO2-TPD proﬁles for the Li/Mg composite catalysts are shown in Figure 5.

Broad desorption peaks from 550 to 650 °C were shown in the CO2-TPD proﬁles of the catalyst, with the molar ratio of Li to Mg being 1 and 2, as shown in Figure 5. These peaks were ascribed to the weak and medium basic sites of the catalyst, mainly corresponding to the surface −OH group and Mg2+−O” pairs.21 Strong basic sites at above 700 °C were observed for catalysts with Li to Mg ratios of 3, 4, and 5, which were ascribed to the production of more Li2CO3 with the increasing Li content. From the CO2-TPD proﬁles of all of the composite catalysts, it was clear that the weak basic sites were transformed into medium and strong basic sites with increasing molar ratio of Li to Mg. This result might be ascribed to the formation of
more Li₂CO₃ and Mg(OH)₂ species, and it was in accordance with the XRD characterization.

It is known that the lithium atom has a small diameter and easily diffuses into the body of the catalyst and then plugs the pore of the catalyst. The surface area and pore volume of the composite catalysts were debased with increasing Li content, as shown in Table 1. This detrimental effect was probably ascribed to the displacement reaction of LiNO₃ with Na₂CO₃, producing new crystal-phase Li₂CO₃ and NaNO₃. The formation of Li₂CO₃ and NaNO₃ was detected by XRD and FT-IR characterization, as shown in Figures 3 and 4.

According to the above characterization results, it was concluded that the texture structure and basic properties of the Li/Mg composites were transformed with increasing Li content, and these differences in structure and basic properties had a deep impact on their catalytic performances, as would be seen in the next section.

**Catalytic Test.** The catalytic performances of the synthesized composite catalysts with various molar ratios of Li to Mg are shown in Figure 6. It was found that glycerol conversion increased obviously; however, GC selectivity decreased slowly. Also, the GC yield reached the maximum when the molar ratio of Li and Mg was 4. This result was probably ascribed to the increasing basicity with increasing Li content. Ochoa-Gómez et al. used different basic catalysts for the GC synthesis and found that the catalytic performance was dependent on the catalyst's basic strength. Malyaadri et al. prepared Mg/Al/Zr catalysts for the synthesis of GC and deemed that the catalytic activities were decided by their basic strength and basicity. Thus, it could be inferred that both the higher basic strength and the larger basicity favored the conversion of glycerol more efficiently. As seen in Figure 3, the diffraction peak intensity of Mg(OH)₂, especially of Li₂CO₃, was obviously increased with increasing Li content. Also, as illustrated in Figure 5, the basicity of medium and strong basic sites was improved with increasing Li content.

However, the GC selectivity was varied slowly with increasing Li content. As stated above, the basic strength and the basicity of the composite catalysts were improved with the LiNO₃ content. The reduction of GC selectivity was probably ascribed to the fact that the strong basic species could promote the decomposition of GC into glycidol as a byproduct. Liu et al. has reported that excessively high basic strength and basicity may facilitate glycerol conversion at the expense of the reduction of GC selectivity. Therefore, it could be deduced that the higher basic strength and the larger basicity of catalysts might facilitate the glycerol conversion but impair the GC selectivity.

**Catalyst Reusability for GC Synthesis.** After the reaction using the fresh catalyst, the used catalysts were cleaned twice using 100 mL of methanol, dried at 110 °C for 2 h in an oven for the next run. The performance results of the reused catalysts are given in Table 2. It was seen that the glycerol conversion was decreased gradually and the GC selectivity constant basically in the successive recycling. Generally, the main reasons for catalyst deactivation are the decrease of surface area and the loss of active species. In Table 1, it was shown that the surface area and pore volume were decreased with increasing Li content, due to the formation of the new crystal-phase Li₂CO₃ and NaNO₃. However, the glycerol conversion was increased. Thus, it was concluded that the catalytic performance of the Li/Mg composites was in proportion to the basic strength and the basicity, and had little correlation with the surface area and pore volume. Liu et al. have reported that there is little connection between the poor activity of the reused catalysts and their surface area or pore volume, which further proved the correctness of our experimental results.

In addition, inductively coupled plasma-optical emission spectroscopy (ICP-OES) was used to examine the loss of active species using the synthesized GC product as sample, and the results are given in Table 3. It was shown that the elements Li, Mg, and Na were present in the synthesized GC product. Because of continuous stirring of the mixture of reaction

| Li/Mg | BET area, m²/g | pore volume, cm³/g | pore size, nm |
|-------|----------------|--------------------|--------------|
| 1     | 51.42          | 0.15               | 11.48        |
| 2     | 29.18          | 0.11               | 15.28        |
| 3     | 25.62          | 0.078              | 17.22        |
| 4     | 13.62          | 0.07               | 20.64        |
| 5     | 10.94          | 0.048              | 21.76        |

Table 1. BET Characterization of Li/Mg Composites with Different Molar Ratios of Li to Mg

Figure 6. Catalytic performances of Li/Mg composite catalysts with different molar ratios of Li to Mg. Reaction conditions: molar ratio of DMC/glycerol = 3:1, reaction temperature: 80 °C, reaction time: 2 h, catalyst mass: 4 wt. % of glycerol mass.

Table 2. Reusability of the Synthesized Catalyst

| times of reusability | conversion, % | selectivity, % | yield, % |
|----------------------|---------------|----------------|---------|
| 1                    | 92.05         | 98.44          | 90.61   |
| 2                    | 89.05         | 98.78          | 87.97   |
| 3                    | 85.81         | 98.83          | 84.81   |
| 4                    | 82.54         | 99.26          | 81.93   |

"Reaction condition: ratio of Li/Mg: 4; molar ratio of DMC/glycerol = 3:1; reaction temperature: 80 °C; reaction time: 2 h; catalyst mass: 4 wt. % of the glycerol mass.

Table 3. Elemental Analysis of the Synthesized GC

|       | Li    | Mg    | Na    |
|-------|-------|-------|-------|
| content, % | 0.0012 | 0.0027 | 0.0050 |
Table 4. Activity Comparison of Li/Mg Composites with Other Reported Catalysts

| catalysts                  | calcination temp, °C | reaction time, h | reaction temp, °C | n(DMC)/n(glycerol) | glycerol conversion, % | GC yield, % | organic solvent | ref |
|----------------------------|----------------------|------------------|-------------------|---------------------|------------------------|-------------|-----------------|-----|
| LDH/SBA-15                 | 100                  | 2                | 100               | 3/1                 | 78                     | 70.2        | DMF             | 24  |
| Mg/Al hydrotalcites        | 100                  | 5                | 100               | 5/1                 | 62                     | 55          | DMF             | 25  |
| Fe–La mixed oxide          | 550                  | 40               | 240               | 4/1                 | 71                     | 71          | free            | 26  |
| Li/Mg composites           | 100                  | 2                | 80                | 3/1                 | 92                     | 90.6        | free            | present work |

reagents and the catalyst in the reaction system, the active species were easily removed from the catalyst surface by collision between the catalyst and stirrer and then dragged into the mixed products. However, the mass fraction of Li and Mg in the synthesized GC product was 0.0012 and 0.0027%, respectively, which meant that the molar ratio of Li/Mg in the synthesized GC was 1:1.5. Thus, it was inferred that the ratio of Mg/Li in the synthesized GC was higher than that in the fresh catalyst. Therefore, it was the loss of the secondary active species that resulted in the decrease of catalytic performance.

**Activity Comparison of Li/Mg Composites with Other Catalysts.** The GC yield for Li/Mg composites was compared with that of other reported catalysts, and the results are shown in Table 4. It was seen that the different basic catalysts in Table 4 displayed various extents of glycerol conversion. Moreover, it was found that the hydrotalcite-like catalysts had lower activity despite the existence of the dimethylformamide (DMF) solvent. This was mainly ascribed to the only weak-strength basic sites in the uncalcined hydrotalcite. Fe–La mixed oxide showed 71% of glycerol conversion without solvent. However, it required a higher calcination temperature and reaction temperature compared with other catalysts. Li/Mg composites presented 92% glycerol conversion and 90.6% GC yield without the organic solvent under mild catalyst and reaction conditions. The above analysis illustrated that the Li/Mg composites were a prospective catalyst for the GC synthesis.

**Experimental Section**

**Materials.** LiNO₃, Mg(NO₃)₂, glycerol, DMC, and biphenyl were purchased from Tianjin Tianti Chemical Reagent Co., Ltd. Glycidol (95 wt %) and GC (90 wt %) for the gas chromatography analysis were purchased from Shanghai Mackin Biochemical Co., Ltd. All other reagents were directly used without further purification.

**Catalysts’ Preparation.** The Li/Mg composites with different Li/Mg molar ratios were prepared by coprecipitation using two kinds of solution: A and B. A was a metallic cation solution composed of 0.8 mol/L LiNO₃ and 0.2 mol/L Mg(NO₃)₂. B was a highly basic carbonate solution comprising a 0.2 mol/L Na₂CO₃ solution. In this experiment, solutions A and B were slowly fed into a three-neck flask equipped with a mechanical stirring bar, a thermometer, and a condenser for separating the byproduct methanol. The reaction was carried out at 80 °C for 2.0 h under stirring.

**Product Analysis.** The reaction products were analyzed using a gas chromatograph (GC-9560, Huai, China) with a capillary column (CBP-20, 25 m × 0.25 mm × 0.25 μm) and FID. The temperature program of the capillary column was initiated at 75 °C for 5 min, then increased up to 250 °C at the rate of 20 °C/min, and kept for 10 min. The temperatures at the injector port and detector were set at 250 and 280 °C. Biphenyl was screened as the internal standard for the quantitative analysis.

**Conclusions**

Li/Mg composite catalysts were prepared for the GC synthesis from glycerol and DMC. The Li/Mg composites presented high glycerol conversion and GC selectivity. A high ratio of n(Li)/n(Mg) could facilitate the improvement of the catalyst’s basic properties, due to the formation of more La₂CO₃ and Mg(OH)₂ species. A too-strong basic strength of Li/Mg composite catalysts could facilitate the glycerol conversion but impair GC selectivity.

**Author Information**

**Corresponding Author**
Zhemin Liu – School of Chemical and Biological Engineering and Shanxi Key Laboratory of High Value Utilization of Coal Gangue, Taiyuan University of Science and Technology, Taiyuan 030024, China; orcid.org/0000-0001-6356-7758; Email: zhmliu@tyust.edu.cn
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