Transesterification of Canola Oil to Biodiesel Using CaO/Talc Nanopowder as a Mixed Oxide Catalyst

A series of heterogeneous catalysts including different molar ratios of CaO/talc was synthesized to study the transesterification reaction of canola oil and methanol under different reaction conditions. Characterization and kinetic results revealed that the activity of this catalyst was enhanced due to the increase of CaO/talc molar ratio value leading to an improvement in the biodiesel production. Moreover, the effect of various parameters on the activity of the undertaken catalysts was studied in order to determine the optimum process conditions. Leaching measurements and the durability of the CaO/talc catalyst under several reaction cycles were evaluated and proved it to be a stable catalyst.

Keywords: Biodiesel, CaO/talc nanopowder, Heterogeneous catalyst, Methanolysis, Talc nanoparticles

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1 Introduction

The high costs of energy due to the industrialization and environmental contamination caused by fossil fuels forced researchers to find a green alternative [1–3]. Biodiesel became a well-known choice compared to fossil fuel considering its advantages in terms of eco-friendly, nontoxic, biodegradable, and renewable features. In addition, biodiesel leads to low emission of toxic materials such as aromatic hydrocarbons, CO, NOX, SOX, and greenhouse gases.

Structurally viewed, biodiesel is a fatty acid alkyl ester generated by the reaction of triglycerides with short-chain alcohols, e.g., methanol and ethanol as demonstrated in Scheme 1. It is considered as transesterification reaction activated through a basic or acidic catalyst or enzyme [4,5]. Vegetable oils, animal fats, waste greases, and algae provide the main sources of triglycerides utilized as feedstocks for biodiesel production in the presence of homogeneous or heterogeneous catalysts. Homogeneous alkali catalysts such as NaOH and KOH are applied for industrial production of biodiesel due to their high activity under mild reaction conditions. However, such materials possess some major drawbacks such as saponification, equipment corrosion, etc. On the other hand, scientists focused on heterogeneous catalysts for being recyclable, less sensitive to moisture and free fatty acids, and because no refinement is needed, i.e., catalyst separation from products [6–8].

A number of solid catalysts were reported for the transesterification reaction and biodiesel production [9,10]. However, heterogeneous catalysts have their own disadvantages. In some cases, severe reaction conditions, i.e., high pressure and temperature, were needed to boost up the low catalytic activity [11,12]. Moreover, significant leaching of active components in the reaction media led to lower activity and stability [13,14]. K/Ca-Al-graphite [15] and Na2ZrO3 [16] showed high activity at low temperatures but deactivation due to leaching was significant.

Calcium-based catalysts are well-known amongst solid materials for the transesterification reaction of oils and alcohols. Nonetheless, they suffer from leaching problems necessitating further refinement to remove Ca contaminants from the production [17–20]. To improve the performance and reliability of the calcium-based catalysts, calcium was mixed with other metal oxides featured as an active support [21,22]. D’Cruz [23] reported a low activity (70.7 % conversion) for a Li/CaO catalyst in the transesterification of canola oil after 6 h of reaction. The mixed oxide of CaO-MgO showed fair catalytic activity, although a significant leaching problem was reported [24]. Taufiq-Yap and Teo [25] stated that with CaO-La2O3 prepared through coprecipitation, the fatty acid methyl ester (FAME) conversion of 86.51 % was achieved after 6 h of reaction at 65 °C. In this case, the reaction time was long, and the FAME yield was low.

In addition, studies on application of clay and minerals such as dolomite, zeolite, and bentonite as solid catalysts attracted attention for biodiesel production [26–28]. However, no report on the use of talc nanopowder as a heterogeneous catalyst support for the transesterification reaction is available to date. Talc is a thermally stable material having a naturally hydrous mag-
nesium silicate structure with the chemical formula Mg₃Si₄O₁₀(OH)₂. It consists of a sheet of brucite or Mg(OH)₂ placed between SiO₂ sheets. Each of these compounds separately was used as a support for the catalyzed production of biodiesel [24, 29].

According to the aforementioned reports and in order to cope with the challenges of solid catalysts, it was desirable to prepare a novel catalyst with high activity, reliability, and convenience for biodiesel production from laboratory to industrial scale. Here, a series of CaO/talc catalysts were synthesized by a simple wet impregnation method. In addition, characterization of this new catalyst and its respective parameters affecting the biodiesel production was done to determine the optimum reaction conditions. Moreover, the durability of the synthesized materials was evaluated.

2 Experimental

2.1 Materials

Commercial food-grade canola oil was obtained from a local store (Kadbanoo Co., Iran) and used with no further purification. Its chemical and physical properties are summarized in Table 1. Methanol and sodium hydroxide were of analytical grade purchased from Merck Company. Calcium nitrate tetrahydrate [Ca(NO₃)₂·4H₂O] and talc nanoparticles [Mg₃Si₄O₁₀(OH)₂] were obtained from Nanoshel.

![Scheme 1. Transesterification reaction of triglyceride with alcohol in the presence of a catalyst.](image)

Table 1. Chemical and physical properties of canola oil.

<table>
<thead>
<tr>
<th>Composition</th>
<th>Amount</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oleic acid [wt %]</td>
<td>59</td>
</tr>
<tr>
<td>Linoleic acid [wt %]</td>
<td>22</td>
</tr>
<tr>
<td>Linolenic acid [wt %]</td>
<td>9</td>
</tr>
<tr>
<td>Palmitic acid [wt %]</td>
<td>5</td>
</tr>
<tr>
<td>Stearic acid [wt %]</td>
<td>3</td>
</tr>
<tr>
<td>Erucic acid [wt %]</td>
<td>2</td>
</tr>
<tr>
<td>Saponification value [mg KOH g⁻¹]</td>
<td>187</td>
</tr>
<tr>
<td>Acid value [mg KOH g⁻¹]</td>
<td>0.071</td>
</tr>
<tr>
<td>Water content [wt %]</td>
<td>0.98</td>
</tr>
</tbody>
</table>

2.2 Catalyst Preparation

The CaO/talc catalysts were prepared applying the wet impregnation method in the following manner. Defined amounts of Ca(NO₃)₂·4H₂O and talc nanoparticles were mixed at different molar ratios (1:0, 3:1, 1:1, 1:3, and 0:1) to prepare a white slurry solution. This was poured into the rotary evaporator flask to perform impregnation. The process took place at 70 °C and 30 rpm under vacuum until complete vaporization. Finally, the solid state was collected, dried at 120 °C for 24 h, and calcined at 600 °C for 5 h.

2.3 Methods for Characterization

Hamnett indicators were utilized to determine the basic strength of the catalyst materials (Hₗ) [30]. Approximately 25 mg of sample was mixed with 0.5 mL methanol solution of the Hamnett indicator. After a duration of 2 h, the solution reached an equilibrium as soon as no more additional color changes were observed. Bromothymol blue (Hₗ = 7.2), phenolphthalein (Hₗ = 9.8), indigo carmine (Hₗ = 12.2), 2, and 4-dinitroaniline (Hₗ = 15.0) each at a concentration of 0.02 mol L⁻¹ served as indicators. Catalyst morphologies were analyzed by field emission scanning electron microscopy (FESEM; MIRA FEG-SEM). The surfaces of dried solid samples were coated with gold employing a sputter coater (Pishtaz Engineering Co. High Vacuum Technology ACECR-Sharif University of Technology Branch-Iran) before FESEM analysis.

Powder X-ray diffraction (XRD) peaks were obtained with a Rigaku RU2000 rotating anode powder diffractometer (Woodlands, TX) equipped with a scanning speed of 58 min⁻¹ and Cu-Kα radiation (40 kV, 200 mA), over a 2θ range of 20–120° with a step size of 0.03°. In order to calculate the surface area of the CaO/talc catalysts, N₂ adsorption/desorption measurements at 77 K were performed utilizing the Brunauer-Emmett-Teller (BET) method.

For Fourier transform infrared (FTIR) spectroscopy, samples were prepared in KBr matrix on an ABB Bomem spectrometer in the wavenumber range of 400–4000 cm⁻¹. The analysis was conducted on an ABB Bomem spectrometer, collected and evaluated by a data acquisition system. Spectral calculations were averaged against 32 scans with a resolution of 2 cm⁻¹. The product samples were analyzed by an Agilent Technologies 7890A gas chromatograph equipped with a flame ionization detector (FID) and a capillary column of CP-Sil 8 CB (60 m × 0.25 mm × 0.5 μm). Helium was used as the carrier gas. The injector and detector temperatures were 150°C and 290°C, respectively. Methyl ester samples were injected by a sampler at an oven temperature of 120°C. After an isothermal period of 3 min, the GC oven was heated at 8°C min⁻¹ to 280°C and held there for 5 min.

In order to study the leaching of metals (Ca, Mg, and Si) during the reaction, the catalysts were filtered out from the reaction mixture after 2.5 h, and then the liquid media were analyzed by inductively coupled plasma atomic emission spectroscopy (ICP-AES; TJA IRIS 1000).
2.4 Catalytic Activity

The CaO/talc catalysts were examined for their catalytic performance in the transesterification reaction under various reaction conditions. The catalysts were activated at 600 °C under atmospheric pressure and N₂ flow for half an hour before the reaction tests. The reactions were carried out in a 100-mL batch reactor consisting of a three-neck flask attached to a condenser equipped with a sampler, thermometer, magnetic stirrer with a fixed stirring power of 45 rpm, and a water bath in order to adjust the reaction temperature. Each run was repeated three times to minimize possible experimental errors. All the experiments were performed under atmospheric pressure.

In a typical reaction, canola oil and methanol with 5 wt% of catalyst in oil and 15:1 methanol-to-oil molar ratio were charged into the aforementioned flask. The reaction media was heated up to 65 °C under continuous stirring fixed at 450 rpm. Samples were withdrawn at specific time intervals of 0.5 h using a calibrated syringe. After each experiment, the catalyst was separated from the reaction mixture through filtration. The rotary evaporator was employed to extract excess methanol from the reaction media. The samples were diluted with hexane and then analyzed by a GC for evaluating the FAME yield.

2.5 Catalytic Stability

In order to determine the stability of the prepared catalysts, the reaction was conducted at 65 °C with 5 wt% catalyst loading mixed with canola oil and methanol at a molar ratio of 15:1. The reaction was repeated with cycle durations of 2.5 h. After each cycle, the catalyst was separated and applied to a new batch of reactants. For recovery purposes, the used catalyst was washed with hexane to extract the adsorbed organic molecules from the catalyst’s surface and then dried at 120 °C and activated at 600 °C for 30 min. Finally, the FAME yield of each reaction cycle was determined by GC analysis.

2.6 Product Analysis

The FAME products were characterized by GC as described above because of its accuracy and non-destructiveness. The reaction product consisted of methyl esters, monoglycerides, diglycerides, and unreacted triglycerides species. The ratio of biodiesel was measured based on an internal standard using heptadecanoic acid as a reference material. Then the methyl ester, i.e., FAME yield was calculated through the following equation [20]:

\[
\text{FAME yield (wt%) = } \frac{\text{Calculated weight of methyl esters}}{\text{Weight of methyl ester phase}} \times 100%
\]

where \( m_{\text{reference}} \), \( m_{\text{esters}} \), \( A_{\text{reference}} \), and \( A_{\text{ester}} \) are the mass of the internal standard (g), the mass of methyl esters (g), the area of the internal standard, and the area of methyl ester, respectively.

3 Results and Discussion

3.1 Catalyst Characterization

The XRD diffraction patterns of CaO, talc, and CaO/talc catalysts with different molar ratios (3Ca-1talc, 1Ca-1talc, and 1Ca-3talc) are displayed in Fig. 1. The mixed phases of MgSiO₃ (2θ = 36.34°, JCPDS File No. 02-0546) and Mg₂SiO₄ (2θ = 54.65°, JCPDS File No. 13-0230) were observed in the XRD analysis of the 0Ca-1talc catalyst emphasizing that it gen-

Figure 1. Comparison of the powder XRD patterns of CaO/talc catalysts (1Ca-0talc, 3Ca-1talc, 1Ca-1talc, 1Ca-3talc, and 0Ca-1talc) exposed to air for 48 h and of the used 3Ca-1talc catalyst.
erally consisted of a mixture of the aforementioned oxides. XRD analysis of 0Ca-1talc also showed peaks at 2θ = 29.25° and 44.37° (JCPDS File No. 30-0794) referable to MgO as well as a peak at 2θ = 61.84° (from the JCPDS File No. 19-0771) due to the presence of MgO. In the case of 1Ca-0talc, the as-prepared catalyst, i.e., exposed to air for 48 h, gave rise to the appearance of Ca(OH)₂ at 2θ = 35.06° and 47.34° (from the JCPDS File No. 76-0570) and CaO at 2θ = 53.93° (JCPDS File No. 82-1276) [31–33].

The formation of Ca(OH)₂ was due to the catalyst exposure to air [31]. The XRD patterns of the CaO/talc catalyst indicated the mixed phases of pure CaO and talc, indicating that this catalyst was a mixture of CaO, Ca(OH)₂, MgO, MgO₂, Mg₂SiO₄, and MgSiO₃ materials. Moreover, XRD patterns of the used 3Ca-1talc catalyst are also given in Fig. 1. Compared to the fresh 3Ca-1talc material, the used sample lacked peaks at 29.25° and 53.93° attributed to MgO and CaO, respectively. It can be concluded that the CaO and MgO active phases were hydrated after the reaction cycle occurred and exposure to air took place. This behavior revealed that the used catalyst was probably activated to restore its active sites before participating in the next reaction cycle.

The FTIR spectra of canola oil and a FAME sample are presented in Fig. 2. Referring to the surface –OH group, the band at 3467 cm⁻¹ appeared in both samples [34]. There existed some similar peaks at 721, 1172, 1241, 1365, 1458, 1740, 2855, 2931, and 3008 cm⁻¹ due to the symmetric and asymmetric vibration of aliphatic groups in oil and biodiesel samples [35, 36]. There were also some peaks exclusive to oil and biodiesel as listed in Tab. 2. Hence, the O–CH₃ stretching vibration and CH₃ asymmetric bending showed the replacement of the –OH group by a methyl group at the end of the carbon chains and the conversion of oil to biodiesel.

Fig. 3 presents the FESEM images of the 3Ca-1talc catalyst emphasizing the formation of a porous solid catalyst in clusters of oval and irregularly shaped particles, with a scale bar of 10 μm. The cluster shape formation was due to the agglomeration of the catalyst particles during calcium doping through the wet impregnation step. Further magnification of the FESEM image with a scale bar of 500 nm revealed that the catalyst surface was made up of small spherically shaped particles with an average size of 70–110 nm.

A summary of physical properties and catalytic performance of pure calcium oxide, talc, and CaO/talc catalysts is given in Tab. 3. The BET surface area of pure talc nanopowder was more than that of the pure calcium oxide; therefore, the surface area of the obtained mixed oxide catalysts was expected to be higher than that of the pure CaO. In addition, the higher pore diameter of mixed oxides compared to pure CaO might have reduced the internal-mass transfer limitation in the reaction.

Tab. 3 also compares the basic strength of CaO/talc catalysts possessing different molar ratios of Ca to talc. Pure talc showed the lowest basic strength while 3Ca-1talc as well as pure CaO revealed the highest basic strength (12.2–15) amongst the pre-

**Table 2.** Characteristic regions of canola oil and biodiesel sample in the FTIR spectra.

<table>
<thead>
<tr>
<th>Region</th>
<th>Assignment</th>
<th>Oil</th>
<th>Biodiesel</th>
<th>Ref.</th>
</tr>
</thead>
<tbody>
<tr>
<td>1105 cm⁻¹</td>
<td>O–CH₂–C symmetric axial stretching</td>
<td>Present</td>
<td>Absent</td>
<td>[41]</td>
</tr>
<tr>
<td>1196 cm⁻¹</td>
<td>O–CH₃ stretching</td>
<td>Absent</td>
<td>Present</td>
<td>[41]</td>
</tr>
<tr>
<td>1442 cm⁻¹</td>
<td>CH₃ asymmetric bending</td>
<td>Absent</td>
<td>Present</td>
<td>[36]</td>
</tr>
</tbody>
</table>

pared materials. This emphasized that the 3Ca-1talc catalyst generally maintained its high basic strength after being mixed with talc. Metal leaching data demonstrate that the presence of talc support resulted in lower contents of Ca leaching. This points to the existence of a strong interaction between the calcium and talc powder elements in the structure of the prepared catalyst. The leaching contents of Mg and Si were also negligible from the talc structure indicating its superior stability compared with previous research reports using MgO and SiO$_2$ supports [24, 37]. In conclusion, the enhancement of the BET surface area as well as lowered leaching of active components were due to the synthesis of CaO and talc nanopowders.

### 3.2 Catalytic Activity

In this section, the effects of different catalyst synthesis parameters including the molar ratio of CaO to talc as well as reaction conditions, e.g., wt % of catalyst loading to canola oil, and methanol-to-oil molar ratio, on catalyst performance were investigated.

#### 3.2.1 Effect of the CaO/talc Molar Ratio

The transesterification reaction was conducted at 65°C catalyzed by a series of CaO/talc catalysts (1Ca-0talc, 3Ca-1talc, 1Ca-1talc, 1Ca-3talc, and 0Ca-1talc). The trends indicated that the molar ratio of the Ca species in the CaO/talc catalyst ought to be increased from 1 to 3 (Fig. 4) in order to enhance the FAME yield. However, a further increase in the Ca content of the catalyst beyond 3:1 did not show any significant influence on the FAME yield. Therefore, the 3Ca-1talc catalyst was taken to be the optimum material for the purpose at hand. Nonetheless, other parameters such as the methanol-to-oil molar ratio and amount of the catalyst loading in terms of the optimum reaction conditions for the transesterification process needed to be determined as described in the following section.

#### 3.2.2 Effect of the Catalyst Amount

The impact of catalyst amount (wt % in oil) on the yield of FAME materials was examined. Various catalyst amounts (1, 3, 5, and 7 wt %) based on the oil weight, at a molar ratio of methanol to oil of 15 and at 65°C, were studied in order to determine an optimum value for this parameter. The conversion of canola oil using CaO/talc catalyst was enhanced as the catalyst amount increased from 1 to 5 wt % (Fig. 5). A further increase of the catalyst amount to 7 wt % lowered the conversion insignificantly. This reduction was perhaps due to the mixing resistance of the reactants, products, and heterogeneous catalysts. This might have derived from some reactants and/or products’ diffusion limitation imposed on the adsorption system. Excess biodiesel previously absorbed onto the catalyst was assumed to lower the biodiesel amount in the reaction media [32]. Therefore, the optimum catalyst loading for the transesterification reaction was determined to be 5 wt % in terms of the oil-to-catalyst ratio. As earlier discussed in this paper, the activity of this catalyst was better compared with those studied for the transesterification reaction utilizing similar calcium-based catalysts [23–25].

<table>
<thead>
<tr>
<th>Catalyst</th>
<th>BET surface area [m$^2$g$^{-1}$]</th>
<th>Pore volume [cm$^3$g$^{-1}$]</th>
<th>Pore diameter [nm]</th>
<th>Basic strength [H$_\text{g}$]</th>
<th>Yield of biodiesel [%]</th>
<th>Metal leaching [ppm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>8.1</td>
<td>0.06</td>
<td>8</td>
<td>12.2–15</td>
<td>91.2</td>
<td>215</td>
</tr>
<tr>
<td>3Ca-1talc</td>
<td>23</td>
<td>0.12</td>
<td>14</td>
<td>12.2–15</td>
<td>96.7</td>
<td>54</td>
</tr>
<tr>
<td>1Ca-1talc</td>
<td>32</td>
<td>0.12</td>
<td>14</td>
<td>9.8–12.2</td>
<td>84.6</td>
<td>42</td>
</tr>
<tr>
<td>1Ca-3talc</td>
<td>41</td>
<td>0.14</td>
<td>16</td>
<td>9.8–12.2</td>
<td>82.8</td>
<td>26</td>
</tr>
<tr>
<td>Talc</td>
<td>59</td>
<td>0.16</td>
<td>18</td>
<td>≤7.2</td>
<td>77.1</td>
<td>–</td>
</tr>
</tbody>
</table>

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*Figure 3. FESEM image of the 3Ca-1talc catalyst.*

*Table 3. Specific surface area, basic strength, and yield of the prepared catalysts. $^a$*

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$^a$) Reaction conditions: methanol-to-oil molar ratio15/1, 65°C, reaction time 2.5 h, 5 wt % catalyst.
3.2.3 Effect of the Methanol-to-Oil Molar Ratio

Theoretically, from a stoichiometric point of view, the methanol-to-oil molar ratio in the transesterification reaction of methanol and oil is known to be 3:1 for obtaining 100 % conversion. On the one hand, being a reversible reaction, usually transesterification happened with an extra amount of alcohol shifting the equilibrium to the forward direction, thus leading to a maximum methyl ester yield below the aforementioned level. On the other hand, heterogeneous catalysts normally performed the transesterification reaction at a lower reaction rate and longer residence time, due to the phase difference with the reaction medium and possible mass transport resistances existing within both phases. This means that the effectiveness of the solid catalyst was improved when a higher methanol-to-oil ratio was utilized. This in turn might have happened to be a rather comforting factor.

A series of reactions were performed in the presence of 5 wt% 3Ca-1talc catalyst at 65 °C with 3:1 to 21:1 methanol-to-oil molar ratios as displayed in Fig. 6. By raising this ratio from 3:1 to 15:1, the catalytic activity increased, leading to a higher conversion. However, a further increase in this ratio beyond 15:1 decreased the FAME yield as the reaction progressed. In other words, since the catalytic interactions on the molecular level mainly occurred on the surface of the solid material, higher amounts of methanol, i.e., 21:1 molar ratio, in the reaction medium led to coverage, hence, reduction of the available surface sites of the catalyst for interactions with the canola oil. This in turn resulted in the observed lower activity for the catalyst at such higher methanol-to-oil molar ratios.

3.3 Catalyst Reusability

To perform the reusability studies, methanolysis of canola oil using 3Ca-1talc and CaO catalysts was carried out under the above-mentioned optimized reaction condition, namely, 15:1 methanol-to-oil molar ratio, 5 wt % catalyst loading, 2.5 h of reaction at 65 °C. The runs were performed under similar experimental conditions and regeneration methods. The results indicate that the reused 3Ca-1talc catalyst managed to yield 96.7, 95.5, 94.1, and 92.8 % of biodiesel through the first, second, third, and fourth runs (Fig. 7). This reduction in activity may be attributed to the leaching of the active species, e.g., Ca and Mg, from the catalyst, being consistent with the experimental measurements (Tab. 3). Furthermore, the catalyst stability enhancement due to the synergistic effect of CaO and talc mixing was indeed a foregone conclusion compared with that of the pure CaO. Moreover, stability and activity of this mixed material CaO-talc were also better than those reported for transesterification of canola oil utilizing other minerals as supports including dolomite [38, 39] and bentonite [40] species.

From these results, it can be concluded that the reactions were mainly performed by the heterogeneous catalyst. To verify this hypothesis, the 3Ca-1talcalc catalyst was refluxed with pure methanol in the reactor for 2.5h at 65 °C. Afterward, the obtained methanol was employed for the transesterification reaction with a methanol-to-oil molar ratio of 15:1 at 65 °C for 2.5 h. After GC analysis, an insignificant conversion of ~3.7 % of FAME was observed indicating that the leachate was negligible. Thus, the 3CaO-1talcalc catalyst showed a good durability.
for up to five catalytic cycles in comparison with the conventional CaO catalyst.

4 Conclusions

Catalysts with different amounts of Ca supported on talc nanopowder were synthesized, characterized, and used for the transesterification of canola oil with methanol. Molar ratio wise, the 3Ca-1talc catalyst revealed to be efficient for this purpose. Reaction conditions affecting the process including the methanol-to-oil molar ratio and catalyst amount were investigated and their optimum values were determined. Under such conditions, a FAME yield of 96.7% was achieved at 65°C, methanol-to-oil molar ratio of 15:1, and 5 wt % catalyst loading charged into the reactor. Moreover, the catalyst experienced durability tests for five reaction cycles and demonstrated a satisfying performance. In conclusion, the studied CaO/talc provided a promising new catalyst for the transesterification reaction of edible oil in the presence of methanol.

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Research Article: Talc nanopowder is a promising heterogeneous catalyst support for transesterification. CaO/talc catalysts were synthesized by a simple wet impregnation method. CaO/talc and CH₃OH/oil molar ratios and catalyst amount were optimized. The 3CaO-1talc catalyst achieved higher catalytic activity compared to conventional CaO catalysts and showed a good durability for up to five catalytic cycles.

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