

Calcium Oxide from Limestone as Solid Base Catalyst in Transesterification of *Reutealis trisperma* Oil

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ABSTRACT

CaO catalysts were synthesized from limestone by thermal decomposition and coprecipitation methods. The CaO and MgO reference catalysts were also synthesized for comparison. The catalysts were characterized by X-ray diffractometer (XRD) and the analysis result was refined by Rietica software. CaO catalyst obtained by coprecipitation method has higher purity of CaO and lower MgO content than those of calcined CaO. The catalysts were also characterized by Fourier Transform Infrared (FTIR) spectroscopy. FTIR spectra showed that the catalysts can be easily hydrated and carbonated in air. The catalytic activity of the catalyst was studied in transesterification reaction of *Reutealis trisperma* (Kemiri Sunan) oil with methanol. Transesterification reaction was carried out at oil to methanol molar ratio 1:1 and 1% of catalyst at 60 °C for 2 h. Catalytic activity of CaO catalyst obtained by coprecipitation was higher than calcined CaO. The methyl ester yield obtained from synthesized CaO, CaO from coprecipitation, calcined CaO, and synthesized MgO catalysts were 56.13; 37.74; 15.97; and 3.61%, respectively.

Keywords: limestone; CaO; coprecipitation; biodiesel; *Reutealis trisperma* oil

ABSTRAK

Katalis CaO dari batu kapur (limestone) telah berhasil disintesis melalui metode dekomposisi termal dan kopresipitasi. Katalis CaO dan MgO pembanding juga disintesis untuk dibandingkan hasilnya. Katalis hasil sintesis dikarakterisasi dengan Difraktometer Sinar-X (XRD) kemudian hasil analisisnya diolah dengan perangkat lunak Rietica. Katalis juga dianalisis menggunakan Spektroskopi Infra Merah yang dilengkapi dengan Fourier Transform (FTIR). Spektra FTIR menunjukkan bahwa katalis hasil sintesis mudah terhidrasi dan terkarbonasi oleh udara. Aktivitas katalisis untuk reaksi transesterifikasi minyak Kemiri Sunan dan metanol dipelajari pada suhu 60 °C selama 2 jam dengan rasio molar minyak:metanol 1:1 dan katalis 1%. Aktivitas katalisis CaO hasil kopresipitasi lebih tinggi daripada CaO hasil kalsinasi. Yield metil ester yang diperoleh dari katalis CaO hasil sintesis, CaO hasil kopresipitasi, CaO hasil kalsinasi dan MgO hasil sintesis adalah 56.13; 37.74; 15.97; dan 3.61%.

Kata Kunci: batu kapur; CaO; kopresipitasi; biodiesel; minyak Kemiri Sunan

INTRODUCTION

The advancement of technology and industry has the consequence in the increasing of energy consumption, especially fossil fuel. In fact, fossil fuel is unrenovable energy that become limited in amount, so eco-friendly and renewable energy such as biodiesel is required to substitute fossil fuel [1].

Biodiesel is a mixture of monoalkyl esters of long chain fatty acids obtained from vegetable oils or animal fats by transesterification reaction. Transesterification

reaction of triglycerides mainly with methanol converted into fatty acid methyl ester or biodiesel and glycerol as a by-product [2]. The reaction is usually carried out in the presence of homogeneous alkaline or acid catalyst. The homogeneous catalyst have same phase with reactant while heterogeneous catalyst is different [3]. Many researchers have been studying heterogeneous base catalysts such as calcined Mg-Al hydrotalcite; zeolite and metal oxides such as calcium oxide (CaO), magnesium oxide (MgO), zirconium oxide (ZrO₂); and supported catalyst. Metal oxide catalysts have

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substantial amount of covalent character facilitates in the transesterification reaction. The report says that over more 90% methyl ester yield was produced by these catalysts. Among them, CaO has many attention used from metal oxide groups as heterogeneous catalyst for biodiesel production since CaO has high basic strength, less environmental impacts, and can be synthesized from cheap sources like limestone and calcium hydroxide [4].

In this research, we investigate the using of limestone for CaO catalyst production. In previous study, CaO was obtained from nature i.e limestone. Natural limestone obtained from Madura island has main composition i.e calcite (CaCO_3 , 92.11-98.42%), dolomite ($\text{CaMg}(\text{CO}_3)_2$, 1.15-7.28%), quartz, siderite, and pyrite [5]. Calcite decomposed by calcination at high temperature and produced strong base CaO and small amount of MgO. The catalytic activity of limestone was studied in the transesterification reaction of *Reutealis trisperma* (Kemiri Sunan) oil. *Reutealis trisperma* oil is a new potential source for biodiesel production from non-edible oil. CaO and MgO were synthesized from nitrate salt precursor as comparison.

EXPERIMENTAL SECTION

Materials

Reutealis trisperma oil from PT. Kemiri Sunan Drajat (consists of 2.44% free fatty acid), methanol (Merck, 98%), H_2SO_4 (Merck, 98%), natural limestone, dichloromethane (Merck, 99%), aquadest, HCl, HNO_3 65%, $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (Merck, 99%), $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Merck, 99%), NaOH, Na_2CO_3 , *n*-Hexane, methyl heptadecanoate (internal standard in GC).

Instrumentation

The analysis and characterization were carried out using Techcomp 7900 GC-MS, Philips Expert XRD, Shimadzu *Instrument Spectrum One* 8400S FTIR, and EVO® MA 10 Scanning Electron Micrographs (SEM).

Procedure

CaO catalyst was synthesized from limestone by two methods. The first method was thermal decomposition by tubular furnace at 800 °C for 6 h to transform CaCO_3 into CaO (referred as SK-800). The second method was coprecipitation method by dissolution of limestone in nitric acid then precipitated by NaOH/ Na_2CO_3 (referred as CaO-SK). In order to prepare nitrate salt solution, 1 g of limestone was added

to 2.58 mL HNO_3 65%, the solution was heated then dissolved in 250 mL aquadest. The solution of 200 mL NaOH 0.075 M and Na_2CO_3 0.075 M was added to nitrate salt solution dropwise while stirred at 580 rpm for 12 h at 60 °C. The sediment obtained then centrifuged, neutralized and dried at 105 °C for 12 h then calcined at 800 °C for 6 h.

CaO and MgO were synthesized (referred as CaO-synt and MgO-synt, respectively) from nitrate salt as comparison. $\text{Ca}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (2.36 g) and $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (2.56 g) dissolved in 250 mL aquadest respectively. The sediment obtained was centrifuged, neutralized and dried at 105 °C for 12 h then calcined at 800 °C for 6 h.

Characterizations

XRD measurements were performed on a XRD Philips Expert using Cu K α radiation at 40 kV, 30 mA, in the range of $2\theta = 20\text{-}70^\circ$. The FTIR spectra of the samples were recorded at wavenumber 4000-400 cm^{-1} with FTIR Shimadzu *Instrument Spectrum One* 8400S. Scanning Electron Micrographs (SEM) and TGA-DSC were obtained to observe the morphology and decomposition temperature of the limestone powder.

Transesterification reaction

The esterification reaction was done to minimize the FFA content. Esterification reaction of *Reutealis trisperma* oil and methanol (3:1) was performed in a glass bath reactor with a water-cooled condenser using H_2SO_4 as catalyst at 65 °C for 2 h. *Reutealis trisperma* oil was added into calcined catalyst and methanol at 60 °C and stirred at 900 rpm. Activated catalysts were reacted with methanol to form methoxide calcium [6-7]. The oil on top layer was used in the transesterification reaction.

The transesterification reaction was carried out at methanol/oil molar ratio 1:1, catalyst amount 1% (w/t) of oil weight, and reaction time for 2 h at 60 °C [8]. The mixtures were stirred at 900 rpm [9]. After completion, HCl was added to the mixture to stop the reaction [10]. The catalysts were separated by separating funnel. Three phase layers were observed, i.e aqueous, organic, and catalyst layer, respectively. Organic layer that consist of methyl ester and glycerol was washed by dichloromethane (10 mL) and isolated until 2 layers were obtained. This layer contains methyl ester, less methanol, dichloromethane, catalyst, and mono-, di-triglyceride. The residual methanol and dichloromethane in methyl ester layer was evaporated by rotary evaporator at 60 °C and methyl ester was analyzed by GC-MS Techcomp 7900 with a capillary column.

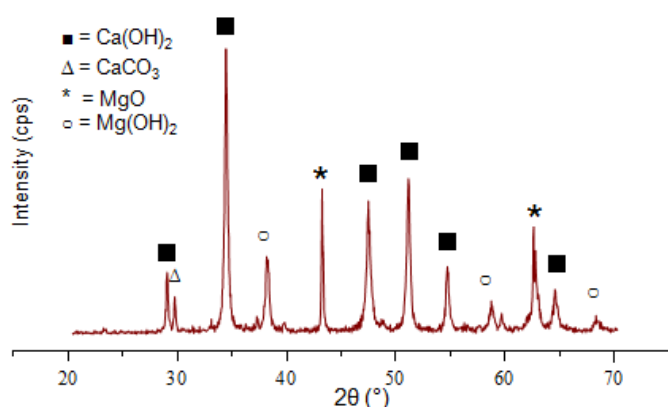


Fig 1. Diffractogram of limestone powder

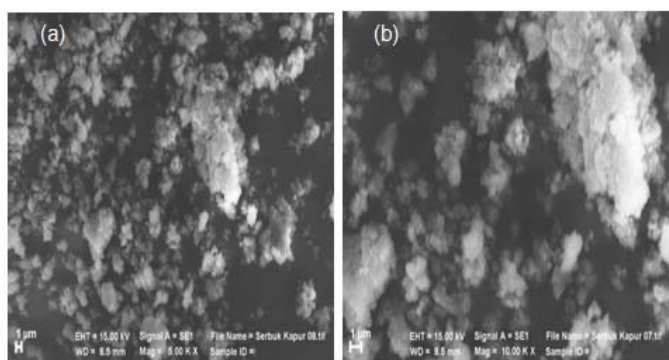


Fig 2. The SEM morphology of limestone powder with magnification of (a) 5,000 x (b) 10,000 x

RESULT AND DISCUSSION

Characteristics of Catalysts

The XRD patterns of the limestone powder at $2\theta = 28.6; 34.1; 37.8; 47.1; 50.8; 54.4; 59.3; 62.5$ and 64.2° which was characteristic of $\text{Ca}(\text{OH})_2$ [11,12], as shown in Fig. 1. The peak characteristics of MgO at $2\theta = 42.9$ and 62.2° ; $\text{Mg}(\text{OH})_2$ at $2\theta = 37.8; 58.4$ and 68.1° ; and CaCO_3 at $2\theta = 29.4^\circ$ [13]. The phase composition and weight percentage of limestone powder calculated using Rietica software [14-15] was $\text{Ca}(\text{OH})_2 = 67.94\%$; $\text{MgO} = 16.24\%$; $\text{Mg}(\text{OH})_2 = 12.01\%$; $\text{CaCO}_3 = 3.81\%$ [16-17].

The morphology of limestone powder is irregular as shown in Fig. 2 (a) and (b) with different magnifications. It can be seen that the particles make aggregates. The small particles indicated as MgO particle while big particles indicated as CaO [16]. The EDX results show that limestone powder consists of Ca, Mg, C, and O elements.

TGA-DSC curves of limestone showed in Fig. 3. The endothermic peaks are recorded at 26.34 and

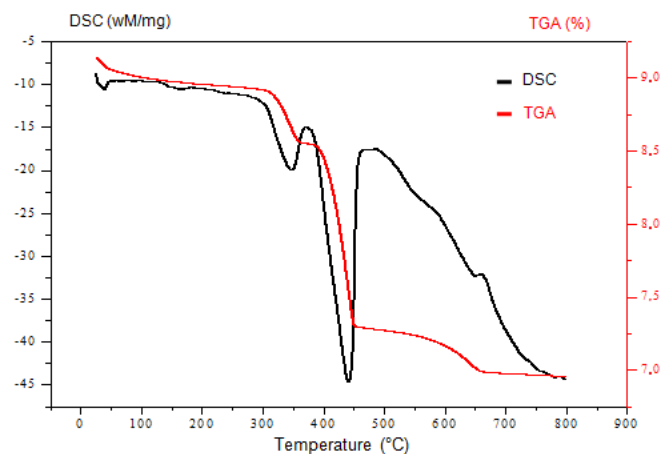


Fig 3. TGA-DSC curves of limestone at 30-800 °C

Step	result
$\text{Ca}^{2+}_{(\text{aq})} + \text{Mg}^{2+}_{(\text{aq})} + 2\text{NO}_3^{-2}_{(\text{aq})} + \text{nH}_2\text{O}_{(\text{aq})}$	pH ~ 0-1
pH increase when $\text{NaOH}/\text{Na}_2\text{CO}_3$ added	pH $\text{NaOH}_{(\text{aq})} = 13$ pH $\text{Na}_2\text{CO}_3_{(\text{aq})} = 11$
$2\text{Ca}^{2+}_{(\text{aq})} + \text{Mg}^{2+}_{(\text{aq})} + 2\text{NO}_3^{-2}_{(\text{aq})} + \text{mNa}^+_{(\text{aq})} + \text{m}(\text{CO}_3^{2-})_{(\text{aq})} + \text{mOH}^-_{(\text{aq})} + \text{nH}_2\text{O}_{(\text{aq})}$	pH ~ 6-7
$\text{CaMg}(\text{OH})_2(\text{CO}_3) \cdot \text{nH}_2\text{O}_{(\text{sediment})} + 2\text{NaNO}_3_{(\text{titrate})}$	pH ~ 6-7
separation and washing	Cleaned NaNO_3
$\text{CaMg}(\text{OH})_2(\text{CO}_3) \cdot \text{nH}_2\text{O}_{(\text{sediment})}$	White suspension
drying	Drying at 105°C
$\text{CaMg}(\text{OH})_2(\text{CO}_3) \cdot \text{nH}_2\text{O}_{(\text{dried})}$	Dried sediment

Fig 4. Coprecipitation method of CaO from limestone

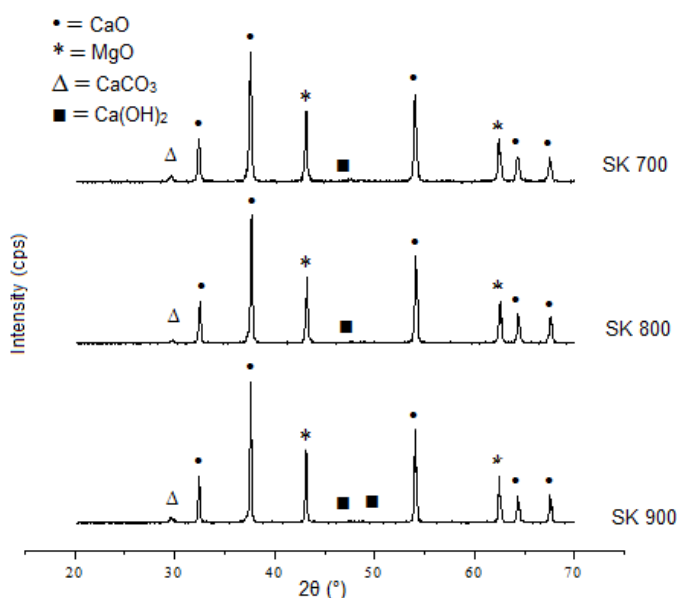
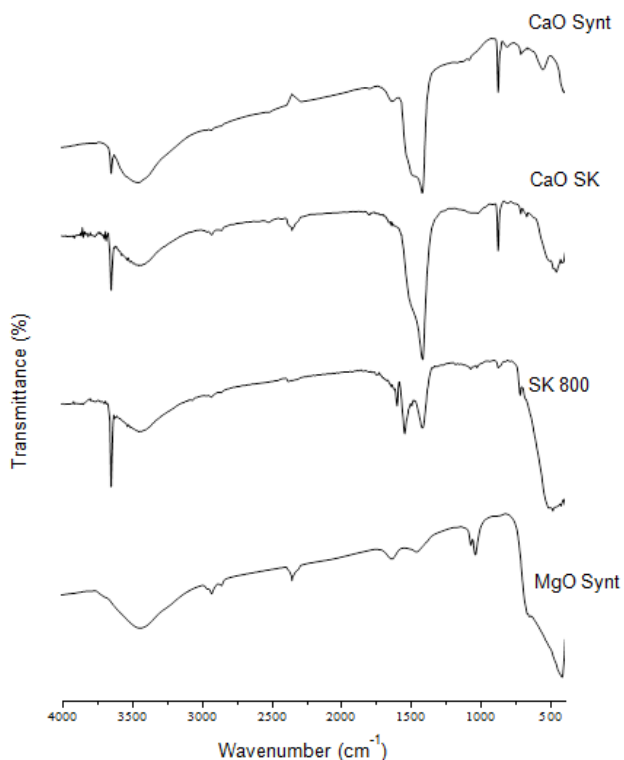
347.94°C which is attributed to decomposition of H_2O physically and chemically bind to CaCO_3 , respectively. Peak at 441.56°C is hydroxide base dehydroxylation as shown by DSC curve as endothermic reaction that occurs during heating process [17]. Meanwhile, decomposition of carbonate group shown at 798.48°C as exothermic peak of CO_2 released from carbonate groups [18].

Coprecipitation method in this research was carried out using nitrate salt solution as precursor as shown in Fig. 4. Diffractograms of CaO -synt shows peaks with low intensity at $2\theta = 34.1; 47.1; 50.7^\circ$ correspondent to $\text{Ca}(\text{OH})_2$ and peak at $2\theta = 29.4^\circ$ correspondent to CaCO_3 [12-13]. Sample MgO -synt shows peaks at $2\theta = 36.8^\circ; 42.8^\circ; 62.2^\circ$ correspondent to pure MgO phase [14].

XRD pattern of CaO -SK sample shows main peaks with high intensity at $2\theta = 32.1; 37.3; 53.8; 64.1; 67.3^\circ$ indicated the peaks of CaO , while low intensity peaks at $2\theta = 29.4$ and 42.8° indicated the peaks of CaCO_3 and MgO , respectively [13]. These peaks also observed in sample SK-800 with higher intensity of CaO and MgO than in CaO -SK. MgO peaks in sample

Table 1. Phase composition and weight percentage ratio calculated by Rietica Software.

Sample name	Phase composition	Weight percentage			
		CaO	MgO	CaCO ₃	Ca(OH) ₂
CaO-SK	CaO, MgO, CaCO ₃ , Ca(OH) ₂	92.50	2.32	5.18	-
CaO Synt	CaO, CaCO ₃ , Ca(OH) ₂	73.16	-	16.49	10.36
SK-800	CaO, MgO, CaCO ₃ , Ca(OH) ₂	51.07	46.43	1.89	0.61
MgO synt	MgO	-	100.00	-	-

**Fig 5.** Diffractogram of SK 700, SK 800 and SK 900**Fig 6.** FT-IR spectra of CaO-synt, CaO-SK, SK-800 and MgO-synt

SK-800 observed at $2\theta = 42.8^\circ; 62.2^\circ$ [14] while Ca(OH)_2 peak observed at 47.1° as shown in Fig. 5 [12].

Table 1 shows that main phase of sample SK-800, CaO-SK and CaO-synt is CaO phase with the highest intensity in sample CaO-SK. Whereas, MgO phase found in sample MgO-synt, CaO-SK and SK-800. Sample SK-800 has higher intensity of MgO peak than in CaO-SK. This indicates that MgO phase at CaO-SK is lower than SK-800. Phase composition and weight percentage ratio were calculated by Rietveld method through Rietica software [15-16]. From ICSD data, the crystal form of CaO and MgO are cubic while CaCO_3 and Ca(OH)_2 are rhombohedral.

CaCO_3 phase on sample CaO-SK, CaO-synt and SK-800 shows carbonation process of CaO and Ca(OH)_2 phase while on sample CaO-synt and SK-800 shows hydration process on CaO surface. When CaO contact with air, the carbonation and hydration process will happened quickly CaO surface [18]. CaO-SK and SK-800 have different weight percentage of CaO and MgO. The weight percentage of CaO and MgO in sample CaO-SK is 92.50 and 2.32% respectively, while in sample SK-800 is 51.07 and 46.43%. MgO phase on SK-800 due to the formation of Mg(OH)_2 that have low solubility in coprecipitation process. Thus Mg^{2+} in precursor is easily form Mg(OH)_2 sediment by addition of OH^- . However, the catalyst synthesized from limestone by coprecipitation method has higher purity of CaO and lower MgO content than catalyst synthesized by calcination method.

FTIR spectra of CaO-synt presents at 3641, 3448, 2966, 2923, 2854, 2522, 2383, 1627, 1481, 1419, 1080, 875 and 405 cm^{-1} as showed in Fig. 6. Peak at 3641 cm^{-1} was assigned to OH groups from Ca(OH)_2 . Wide peak at 3448 cm^{-1} and small peak at 1627 cm^{-1} was assigned to OH stretching and bending vibrations [18]. The peaks at 2966, 2854, 2522 and 2383 cm^{-1} attributed to stretching vibration of carbonyl group (CO) from CO_2 adsorption in air [19]. Peak at 405 cm^{-1} was assigned to stretching vibrations of Ca-O [20]. Peaks at 1481 and 1419 cm^{-1} assigned to the symmetric and asymmetric stretching vibration of O-C-O bonds of unidentate carbonate at CaO surface [21]. The peaks at 1080 and 875 cm^{-1} were also arise from these carbonate groups. The intensity of carbonate peaks was increase as contact time of sample to the air increase [18].

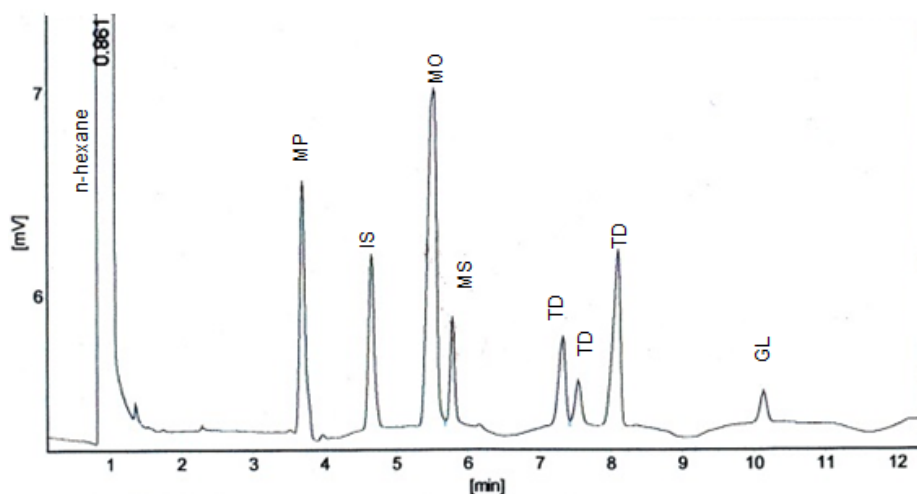


Fig 7. (Chromatogram of CaO-SK transesterification product (note: P= methyl palmitate; IS= internal standart; MO= methyl oleate; MS= methyl stearate; TD= unknown and GL= glycerol)

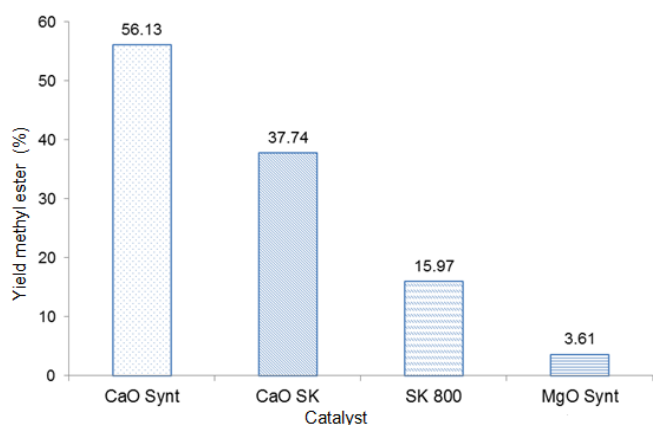


Fig 8. The yield of methyl ester produced by CaO-synt, CaO-SK, SK-800 and MgO-synt catalysts

FTIR spectra of MgO-Synt produce peaks at 3433, 2923, 2854, 2349, 1639, 1458, 1072, 856, and 416 cm^{-1} . C=O groups of CO_2 adsorption from air appears at 2923 and 2349 cm^{-1} [19]. Peak corresponding to MgO presences at wavenumber below 600 cm^{-1} i.e 416 cm^{-1} [14]. The presence of carbonates and hydroxyl peaks in CaO-SK and SK-800 are similar (Fig. 6). Hydroxyl peaks appear at 3600, 3400 and 1600 cm^{-1} [18]. The carbonates peaks appear at around 2900-2300 cm^{-1} , 1540-1070 cm^{-1} and 875 cm^{-1} [21]. Peak at 459 cm^{-1} in CaO-SK assigned to stretching vibrations of Ca-O [22] while in SK-800 found at 482 cm^{-1} and assigned to Ca-Mg peak as reported by Albuquerque et al. which appear at around 450-500 cm^{-1} [13]. The presence of carbonates was indicated by high degree of carbonation and the presence of hydroxyl species. This is might be because of hydration due to adsorption of H_2O from air. The XRD analysis of CaO-SK shows low content of MgO

phase but in FTIR spectra there is no peak of Ca-Mg bonding observed.

Catalytic Activity

Chromatogram of biodiesel produced by CaO SK catalyst can be seen in Fig. 7. Three peaks were observed at retention time 3.687; 5.526 and 5.780 min. These peaks were assigned as methyl palmitate, methyl oleate and methyl stearate. Unknown methyl ester peaks appear at retention time 7.322; 7.536 and 8.101 min. Methyl heptadecanoate and glycerol peaks observed at retention time 4.651 and 10.127 min. Methyl heptadecanoate is the internal standard for methyl ester analysis by gas chromatography. The highest peak at 5.526 min was assigned as methyl oleate compound. The chromatogram of CaO-SK and CaO-synt samples has similar retention time and peak area. Chromatogram of SK-800 and MgO-synt samples has different time retention and low peak area than those of CaO-SK and CaO-synt. The peak area was used to calculate the concentration of methyl ester compound and then to calculate the yield of biodiesel obtained [23].

Fig. 8 shows methyl ester content obtained from transesterification reaction of *Reutealis trisperma* oil with various catalysts. It shows that 56.13% of methyl ester is achieved when CaO-synt is used as the catalyst. CaO SK and SK 800 catalyst from limestone produce methyl ester of 37.74% and 15.97%, respectively. The lowest methyl ester i.e 3.61% was obtained using MgO-synt as catalyst. These results are consistent with the XRD results that high MgO content decreases the catalytic activity in transesterification reaction of *Reutealis trisperma* oil [3]. MgO exhibit less alkalinity than CaO thus MgO provides low basic sites

on the surface which is used in transesterification reactions of triglycerides [13].

CONCLUSION

In summary, CaO catalyst has been successfully synthesized from limestone. Both through calcination (SK-800) and coprecipitation (CaO-SK) method, the catalyst contain of CaO as main composition. CaO catalysts demonstrated higher catalytic activity in the transesterification reaction of *Reutealis trisperma* oil. We found that yield of methyl ester for CaO-SK (37.74%) was higher than SK-800 (15.97%). However, CaO and MgO synthesis as comparison displayed the highest yield of 56.13% for CaO synt and the lowest yield of 3.61% for MgO synt.

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