Biogasoline synthesis by catalytic cracking of used cooking oil catalysed by chicken eggshell-based CaO impregnated onto γ-Al_2O_3

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Abstract

This research aims to synthesize biogasoline from used cooking oil using a catalyst of chicken eggshell based CaO impregnated with γ-Al_2O_3 through catalytic cracking, as well as to characterize the final product. Cracking optimization was carried out by varying the catalyst calcination temperature (650, 750 and 850 °C) and the catalyst concentration (1, 2 and 3 wt.%). The stages of this research were (1) characterization of used cooking oil (density, viscosity, refractive index, and iodine number) (2) synthesis of the CaO/γ-Al_2O_3, (3) catalytic cracking of used cooking oil with the use of the synthesized catalyst, and (4) characterization and identification of biogasoline. The results showed that the optimum condition for catalytic cracking was obtained at the calcination temperature of 650 °C. The synthesized biogasoline has the following characteristics, *i.e.* density of 0.776 g mL^{-1}, viscosity of 1.84 mm^2 s^{-1}, a refractive index of 1.43, and the iodine number of 22.85 g I_2 *per* 100 g. The synthesized biogasoline contained alkane, alkene and carboxylic acid compounds, C_2-C_{19}. This composition was dominated by compounds belonging to biogasoline (C_5-C_{12}) amounting to 94.5 wt.% as confirmed by gas chromatography-mass spectrometry analyses.

Keywords: biofuel; biomass; heterogeneous catalyst; oxide; waste oil; modified material.

1. INTRODUCTION

The increase in the number of motorized vehicles results in increasing fuel consumption, which is mostly supplied from petroleum. However, this increasing need for fuel does not align with the decreasing availability of petroleum resources. It is estimated that the oil demand in 2050 will increase to 943.3 BL of oil in a rapid energy transition scenario [1]. For this reason, the transition from petroleum to renewable energy sources began to be encouraged.

One of the renewable energies is biogasoline produced by conversion of used cooking oil. Cooking oil consumption in Indonesia reached 16.2 billion litters in 2019. It can be estimated that the average used cooking oil produced is in the range of 40 to 60 % of the oil or in the range of 6.46 to 9.72 billion litters [2]. Used cooking oil is obtained by repeated heating the cooking oil. As a result, used cooking oil contains ~42 % oleic acid ($C_{18}H_{34}O_2$) and ~35 % palmitic acid ($C_{16}H_{32}O_2$) [3]. Currently, used cooking oil has been utilized as a raw material in biogasoline synthesis [4–6]. To become a low fraction such as fuel, it is necessary to break down the long hydrocarbon chains through the cracking process [7]. There are two main types of cracking: thermal cracking and catalytic cracking [8]. Since the thermal cracking requires high temperatures [9], catalytic cracking is considered to be safer due to lower temperatures and higher product conversion, making this process more efficient and economically favourable [10].

In this research, a CaO catalyst derived from chicken eggshells impregnated onto γ-Al_2O_3 was used for catalytic cracking. CaO was selected due to its alkaline nature, high catalytic activity, easy preparation process, abundant availability and environmental friendliness. On the other hand, CaO also has a high decarboxylation capacity with low solubility in fuel [11], and reduces the acidity of cracking products [12]. One of CaO sources in nature is chicken eggshells,

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which contain 97 % CaO, 0.263 % Fe_2O_3 and 0.123 % SO_3 [13]. Eggshell based CaO has been used in the production of biogasoline from used cooking oil with a yield of 4.5 % [6]. Cracking of used cooking oil with the CaO/SBA-15 catalyst resulted in a biogasoline yield of 69.70 % [4]. To increase the catalytic activity of CaO, it was impregnated onto γ-Al_2O_3, which is a metastable form of alumina, most often used as a catalyst [14]. It has a large pore volume and surface area and is stable in various catalytic reaction temperature ranges [15]. In addition, it has Brønsted acid sites and Lewis acid sites that contribute to the catalytic cracking process [16]. γ-Al_2O_3 as a catalyst in palm oil cracking resulted in a biogasoline yield of 45.35 % [17]. Also a Co-Mo/Al_2O_3 catalyst was used in cracking of Nyamplung oil with a biogasoline yield of 25.63 % [18], while the use of Ni-Mo/Al_2O_3 in another study resulted in a biogasoline yield of 59.50 % [5]. The present research aims to synthesize and characterize biogasoline from used cooking oil utilizing a catalyst composed of chicken eggshell based CaO impregnated onto γ-Al_2O_3 through catalytic cracking.

2. MATERIALS AND METHODS

Along with the standard laboratory tools (*e.g.* pycnometer, Otswald viscometer, cracking tool sets), the following instruments were used: X-ray diffractometer (XRD), PANanalytical X'pert Pro, PANalytical B.V., Netherland; X-ray fluorescence (XRF) spectrometer, PANanalytical Minimal 4, PANalytical B.V., Netherland; scanning electron microscope (SEM), Inspect-S50 FEI type, FEI Company, Netherland; gas chromatography-mass spectrometry instrument (GC-MS), Shimadzu QP2010S, Japan and Fourier transform infrared (FT-IR) spectroscope, IRPrestige 21, Shimadzu, Japan. Furthermore, the materials used in this study were used cooking oil, chicken eggshells, $Al(NO_3)_3\times 9H_2O$ *p.a.* (Merck, Germany), HCl *p.a.* (Sigma Aldrich, USA), NH_4OH *p.a.* (Merck, Germany), ethanol 96 % and distilled water.

2. 1. Synthesis of γ-Al_2O_3

γ-Al_2O_3 was prepared using a precipitation method. First, 1 M $Al(NO_3)_3\times 9H_2O$ precursor was prepared in 0.5 M HCl solution. 0.5 M NH_4OH was then added dropwise until the pH 9, under stirring using a magnetic stirrer for 3 h at 70 °C. After that, the solution was filtered using a Buchner funnel and the precipitate was washed by distilled water and ethanol. The precipitate was dried in an oven at 75 °C for 24 h [19]. Then the precipitate was calcined at 600 °C for 6 h. The obtained γ-Al_2O_3 was analysed by XRD.

2. 2. Synthesis of chicken eggshell-based CaO

Chicken eggshells were washed in distilled water, crushed and sieved to a 100 mesh size. Then the eggshell particles were dried in an oven at 110 °C for 6 h, followed by calcination at 900 °C for 3 h [20]. The eggshell particles were then analysed by XRF and XRD.

2. 3. Production of chicken eggshell-based CaO/γ-Al_2O_3 catalysts

γ-Al_2O_3 was mixed with the eggshell particles (in water) in a mass ratio of 1:1. The obtained suspension was then stirred using a hand stirrer for 15 min, followed by sonication (42 kHz) at 60 °C for 1 h. Next, the solution was filtered using a Buchner funnel and the precipitate was washed with distilled water and dried in the oven at 110 °C for 4 h. The catalyst precursor was then calcined at various temperatures of 650, 750 and 850 °C for 3 h. The obtained catalysts were analysed by XRD, XRF and SEM.

2. 4. Catalytic cracking of used cooking oil

Used cooking oil was first filtered before cracking with the use of a filter paper. Characterization of the oil included determination of density, viscosity, acid number, refractive index, and composition by using FT-IR analysis.

For the catalytic cracking process, used cooking oil (100 g) and CaO/γ-Al_2O_3 (1 g) were put into the cracking reactor. The batch reactor was constructed by authors using a stainless steel cylinder (5 cm diameter, 10 cm height, 62.5 mL volume), supplied with a temperature sensor for measuring the steam temperature, and connected to a condenser and a bottle by a steel pipe. The reactor was placed on an electric stove, heated to the temperature of 600 °C, followed by the increase in the temperature by 100 °C every 20 min to 1500 °C, and the process was continued for 4 h. The distillate resulting from cracking was collected into 3 fractions based on the temperature ranges: fraction F1 (100 to 125 °C), fraction F2 (125 to 150 °C) and fraction F3) (150 to 175 °C). In this study, the catalytic cracking process varied regarding the calcination temperature of the catalyst (650, 750 and 850 °C) and catalyst concentration (1, 2 and 3 wt.% of the oil). The resulting product was characterized regarding the refractive index, density, viscosity, iodine number and composition by using GC-MS and FT-IR analyses.

3. RESULTS AND DISCUSSION

3. 1. Characterizations of γ-Al_2O_3 and chicken eggshell based CaO

Formation of γ-Al_2O_3 was confirmed qualitatively by XRD showing peaks at the diffractogram at 2θ = 39.37, 45.75, 66.91 and 85.07° (Figure 1). These results correspond to γ-Al_2O_3 peaks in the XRD pattern database with COD ID. 00-101-0461 at 2θ = 39.48, 45.91, 66.95 and 84.99°. The same result is reported in the previous study [21]. Therefore, it was concluded that γ-Al_2O_3 in this study was successfully synthesized.

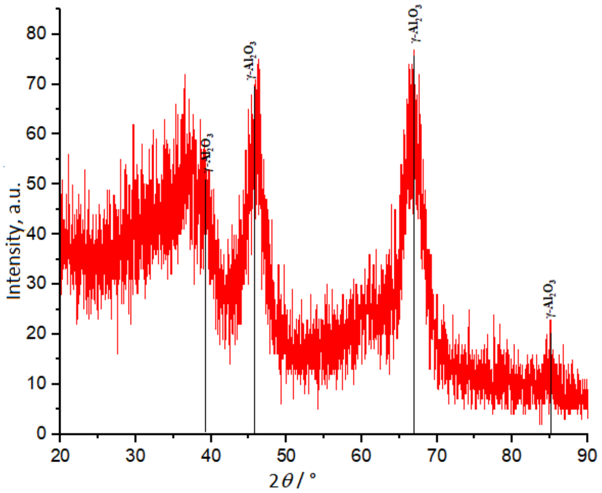

Figure 1. XRD results of the synthesized γ-Al_2O_3

In the final calcination process, the obtained white powder (*i.e.* CaO from chicken eggshells) was analysed by XRD and XRF. Typical CaO peaks were recorded, namely 2θ= 37.47, 53.97, 64.25, 67.47, 79.77 and 86.63° (Figure 2) according to the XRD database with COD ID. 00-101-1095, at 2θ= 37.40, 53.93, 64.24, 67.47, 79.77 and 86.63°. Furthermore, based on XRF analysis the composition of chicken eggshells calcined at 900 °C for 3 h, was determined as: 0.090 wt.% SO_3, 99.590 wt.% CaO, 0.069 wt.% Fe_2O_3, and 0.100 wt.% Co_3O_4. In the other study, the result was obtained at more than 95 wt.% CaO [22]. Therefore, chicken eggshell based CaO in this study showed high purity after the synthesis.

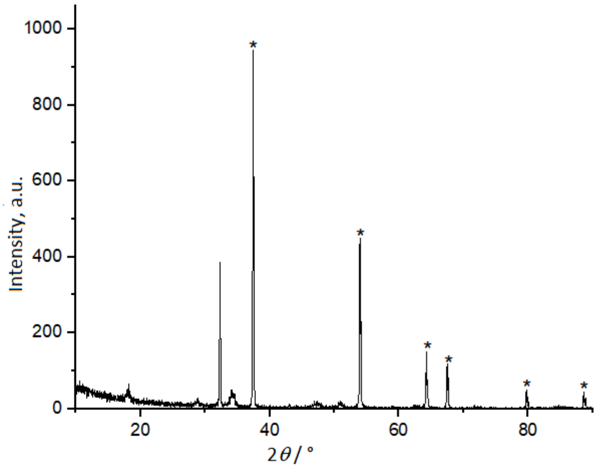

Figure 2. XRD results of chicken eggshell based CaO after calcination at 900 °C

3. 2. Synthesis of chicken eggshell based CaO/γ-Al_2O_3 catalyst

CaO/γ-Al_2O_3 catalyst was synthesized by impregnation of chicken eggshell based CaO onto γ-Al_2O_3 in a mass ratio of 1:1 under ultra-sonication (42 kHz). The catalyst was then calcined at different temperatures of 650, 750 and 850 °C for 3 h. The obtained catalysts were analysed next by XRD, XRF and SEM.

XRD diffractograms of the synthesized CaO/γ-Al_2O_3 catalysts (Fig. 3a) show typical CaO peaks, namely 2θ = 31.66, 36.61 and 52.79° in accordance with the XRD database with COD ID: 00-900-6719, namely 2θ= 31.61, 36.66, and 52.82°. Furthermore, γ-Al_2O_3 peaks were recorded 2θ= 39.49, 46.03 and 66.99° in accordance with the XRD database with COD ID: 00-101-0461, namely 2θ = 39.48, 45.91 and 66.95°. Results of the XRF analysis of all three CaO/γ-Al_2O_3 catalysts obtained at different calcination temperatures are presented in Table 1.

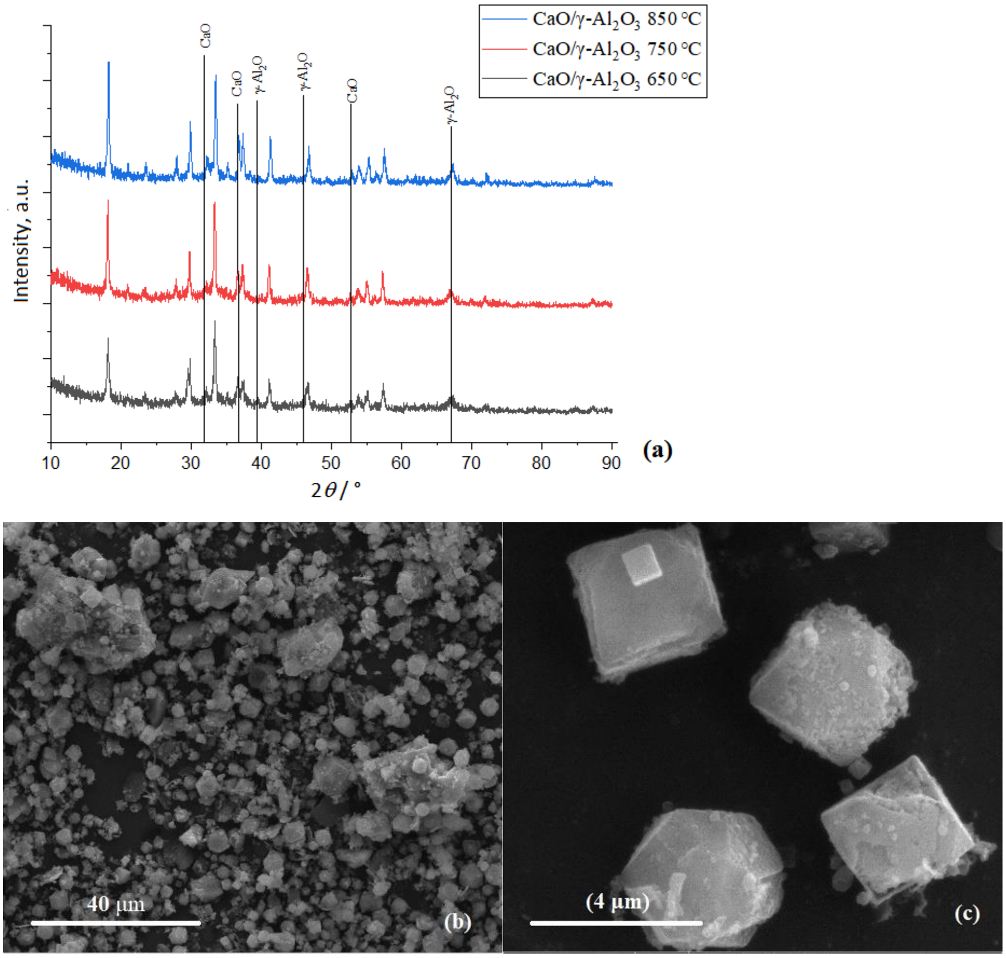

Figure 3. Characterization of chicken eggshell based CaO/γ-Al_2O_3 catalyst (various calcination temperatures of 650, 750, 850 °C): XRD results (a) and scanning electron micrographs of the catalyst calcined at 850 °C (b) and (c)

Table 1. XRF results for chicken eggshell based CaO/γ-Al_2O_3 catalysts calcined at different temperatures

Compound	Calcination temperature, °C		
	650	750	850
	Content, wt.%		
Al_2O_3	24.40	25.70	25.70
CaO	71.72	70.46	70.46
SiO_2	2.80	2.80	2.80
Fe_2O_3	0.10	0.10	0.10
CuO	0.03	0.03	0.03
ZnO	0.02	0.02	0.02
MoO_3	0.57	0.58	0.58
Yb_2O_3	0.25	0.24	0.24

The XRF analysis (Table 1) has indicated the CaO/γ-Al_2O_3 mass ratios in the catalysts of 2.9:1 for the calcination temperature of 650 °C and 2.75:1 for the two other calcination temperatures of 750 °C and 850 °C.

Morphology of the CaO/γ-Al_2O_3 catalyst calcined at 850 °C can be seen in Figure 3b,c. Regularity of the particle size in a cubic shape originating from γ-Al_2O_3 [23] can be seen (Fig. 3b). Meanwhile, the catalyst surface contains lumps (Figure 3c) indicating attachment of CaO to the γ-Al_2O_3 surface. Based on the presented results of XRD, XRF, and SEM analyses, it can be deduced that CaO/γ-Al_2O_3 catalysts were successfully synthesized generally retaining characteristics of the raw materials.

3. 3. Catalytic cracking of used cooking oil

Cracking of used cooking oil produces a polar phase, a nonpolar phase, gas, and the remaining reaction residue. We have focused here on the nonpolar phase, namely the oil products. Three types of catalyst regarding the calcination temperature (*i.e*. 650, 750, and 850 °C) were used at three different concentrations (*i.e*. 1, 2 and 3 wt% of the oil). The reaction mixture was heated to 600 °C and subsequently increased every 20 min by 100 °C up to 1500 °C. The cracking process was carried out for 4 h. The yield of cracking products can be seen in Figure 4.

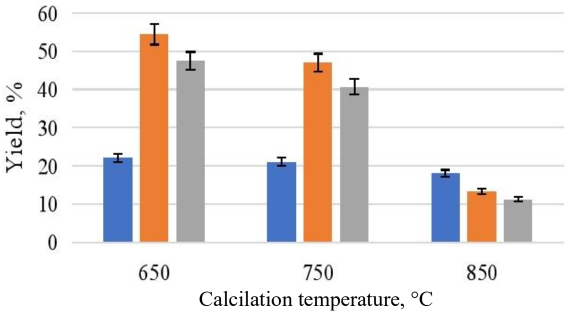

Figure 4. Effects of the calcination temperature (650, 750, and 850 °C) and CaO/γ-Al_2O_3 concentration (1 🟦, 2 🟧, and 3 ■ wt.% of the oil) on the yield of cracking of used cooking oil

Based on Figure 4, the yields of cracking products decrease with increasing the catalyst calcination temperature. Increasing the calcination temperature of the catalyst will cause the pores in γ-Al_2O_3 to collapse and merge with each other, so the available surface of the catalyst decreases [24]. Therefore, the optimal catalyst calcination temperature was found to be 650 °C.

Increasing the catalyst concentration from 1 to 2 wt.% resulted in increasing the yield of cracking products at catalyst calcination temperatures of 650 and 750 °C. However, when the catalyst concentration was increased to 3 wt.%, the cracking product yield decreased because of excessive catalytic reactions, which resulted in the formation of relatively large amounts of residue [32]. The product yield for the catalyst calcination temperature of 850 °C decreased as the catalyst concentration increased. Therefore, at the catalyst calcination temperature of 850 °C, the catalytic cracking gave the worst results as compared to the other two calcination temperatures.

3. 4. Characterizations of cracking products

The obtained mixtures of cracking products were yellow and had a sharp odour in contrast to the physical appearance of used cooking oil, which is brownish in colour and has a rancid odour. Characteristics of cracking products in 3 fractions (F1: 100 to 125 °C, F2: 125 to 150 °C and F3: 150 to 175 °C) obtained at 650 °C calcination temperature and 2 wt.% catalyst concentration together with those of standard gasoline and the starting used cooking oil are shown in Table 2.

Table 2. Characteristics of the catalytic cracking products of used cooking oil with the use of 2 wt% catalyst calcined at 650 °C. F1, F2 and F3 represent distillate fractions based on the temperature ranges

Parameter	F1	F2	F3	Conventional gasoline	Used cooking oil
Density at 25 °C, g mL^{-1}	0.779	0.776	0.774	0.770	0.910
Viscosity at 25 °C, mm^2 s^{-1}	1.92	1.84	1.78	0.48	58.01
Reflective index at 25 °C	1.44	1.43	1.42	-	1.46
Iodine number, g I_2 *per* 100 g	28.2	22.6	17.76	-	20.51

It can be deduced that the cracking products have characteristics close to those of conventional gasoline. A previous study also obtained the characteristics similar to the present study with the higher value, *i.e*. cracking product of off grade crude palm oil under condition of 0.5 % Co-Mo 1:1/α-Fe_2O_3 catalyst in a batch reactor [8].

3. 5. FT-IR and GC-MS analyses of the cracking products

The product resulting from catalytic cracking of used cooking oil by utilizing the CaO/γ −Al_2O_3 catalyst calcined at 650 °C at the content of 2 wt.% was analysed using FT-IR and GC-MS techniques. Three product fractions F1, F2 and F3 were analysed. The results of FT-IR and GC-MS analyses are shown in Figures 5 and 6, respectively. Based on the results presented in Figure 5, several functional groups induced increases in peak intensity, namely the alkene and carboxylic acid groups [8].

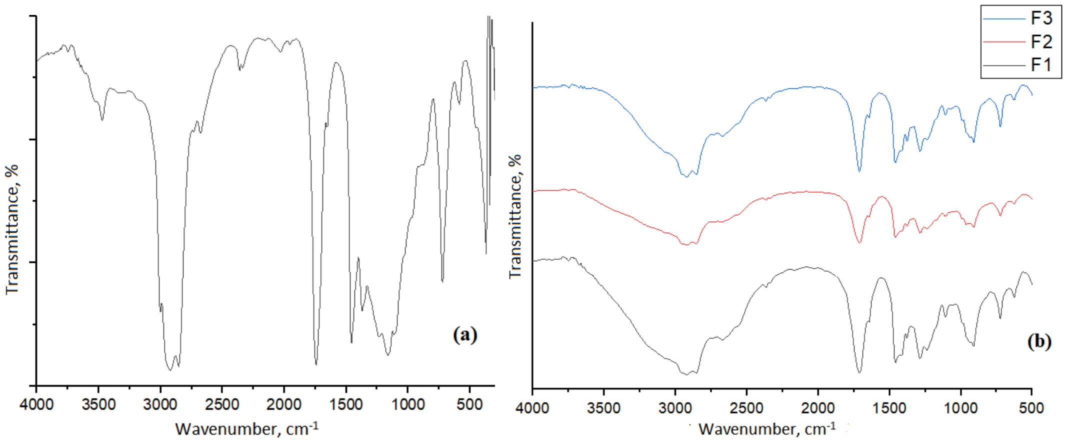

Figure 5. FT-IR spectra of (a) used cooking oil (raw material), and (b) catalytic cracking product fraction

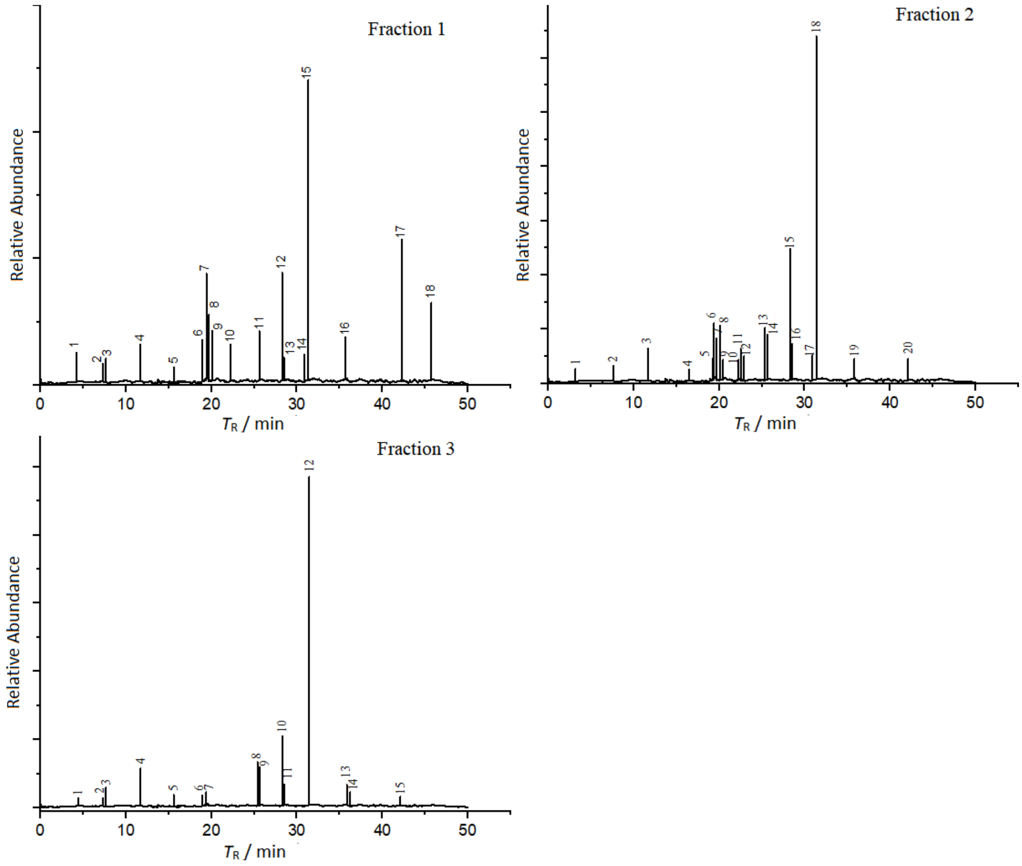

Figure 6. GC-MS chromatograms for the catalytic cracking products in the fractions

Mass spectra at each retention time (T_R) corresponding to the GC-MS chromatograms (Figure 6) are compared for similarities with library data (Table 3).

Table 3. Compound identification and contents (C) of different compounds in 3 fractions of the cracking products

Fraction 1			Fraction 2			Fraction 3		
T_R / min	Compound	C / wt.%	T_R / min	Compound	C / wt.%	T_R / min	Compound	C / wt.%
11.652	n-nonane	3.22	11.651	n-nonane	3.23	4.374	n-heptane	1.41
18.906	1-undecene	3.55	19.387	cis-3-undecene	5.61	7.274	1-hexene	1.40
19.404	cis-3-undecene	8.82	20.076	1,7-octadiene	5.37	7.623	n-octane	2.97
19.675	1-decene	5.55	20.448	heptanoic acid	2.22	11.690	n-nonane	5.82
20.087	1-decune	4.25	22.507	2-methyl decane	3.20	25.369	1-nonene	6.70
25.619	2,7-dimethyl octane	4.25	25.360	1-undecene	5.19	25.641	n-decane	5.93
28.492	Trans-3-undecene	8.88	25.628	2,7-dimethyl octane	4.50	28.286	1-undecene	10.46
31.312	n-decane	24.12	28.297	1-dodecene	12.38	28.525	2-methyl nonane	2.94
35.675	1-dodecene	3.80	28.504	n-undecane	3.65	31.420	n-undecane	48.50
42.273	decanoic acid	11.51	31.358	n-decane	31.98	35.833	1-dodecene	3.35
45.713	9-octadecenoic acid	6.50						

Based on the results of the GC-MS analysis shown in Table 3, F1 consisted of a hydrocarbon with a C_6 - C_{18} bond, *i.e* alkane, alkene and carboxylic acid compounds. In this fraction, biogasoline (C_5 - C_{12}) is dominant, with a content of 93.5 wt.%. Among the compounds found in this fraction, n-decane has the highest concentration of 24.12 wt.%. On the other hand, F2 is hydrocarbons with C_2 - C_{18} bonds consisting of alkane, alkene and carboxylic acid compounds. In this fraction, biogasoline (C_5 - C_{12}) is also dominant, with a similar content of 94.02 wt.%. Here, also, n-decane is found in the highest concentration of 31.98 wt.%. F3 is composed of hydrocarbons with C_6 - C_{19} bonds consisting of alkane, alkene and carboxylic acid compounds with biogasoline (C_5 - C_{12}) being dominant, with the content of 96.08 wt.%. Among the compounds found in this fraction, n-undecane has the highest concentration of 48.50 wt.%. Thus, the average biogasoline content of all three fractions was 94.53 wt.%. Similar hydrocarbon contents in the cracking product were also obtained in a previous study [8]. It should be noted that the catalytic reaction product is a mixture of various compounds [25], so that tracking of cracking products formation is necessary in further research.

4. CONCLUSION

Chicken eggshell-based $CaO/\gamma\text{-}Al_2O_3$ has been successfully synthesized by an impregnation method that was assisted by sonication. The catalyst composition has been confirmed by XRD, XRF and SEM analyses. Impregnation of CaO obtained from chicken eggshells onto $\gamma\text{-}Al_2O_3$ resulted in the $CaO/\gamma\text{-}Al_2O_3$ mass ratios of 2.9:1 for calcination at 650 °C and 2.75:1 for calcination at the other two temperatures of 750 and 850 °C. The best conditions for catalytic cracking of used cooking oil were determined as: the $CaO/\gamma\text{-}Al_2O_3$ catalyst with the mass ratio of 2.9:1 obtained at the calcination temperature of 650 °C and used at the concentration of 2 wt.% of the oil, by which a biogasoline yield of ~55 % was obtained. Furthermore, by characterization of the produced biogasoline, the following results were obtained: density of 0.76 g mL^{-1}, viscosity of 1.84 $mm^2\ s^{-1}$, refractive index of 1.43, and the iodine number of 22.85 g I_2/100 g. The biogasoline contained C_2 - C_{19}, compounds consisting of alkane, alkene and carboxylic acid compounds. This composition is dominated by compounds characteristic for biogasoline (C_5 - C_{12}) with the content of about 94 % as determined by GC-MS analyses. In further studies, there is a need for optimization of the catalyst component ratio as well as duration and temperature of the cracking reaction to produce an economically higher product yield. In addition, analyses of the cracking products formation will be a valuable topic for further research.

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Sinteza biobenzina katalitičkim krekovanjem korišćenog ulja za kuvanje katalizovanim CaO na bazi ljuske kokošjeg jajeta impregniranog na γ-Al_2O_3

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Izvod

Cilj ovog istraživanja je sinteza biobenzina iz korišćenog ulja za kuvanje korišćenjem katalizatora na bazi ljuske kokošjeg jajeta impregniranog sa γ-Al_2O_3 putem katalitičkog krekovanja, kao i karakterizacija finalnih proizvoda. Optimizacija krekovanja je sprovedena variranjem temperature kalcinacije katalizatora (650, 750 i 850 °C) i koncentracije katalizatora (1, 2 i 3 mas.%). Faze ovog istraživanja bile su (1) karakterizacija korišćenog ulja za kuvanje (gustina, viskoznost, indeks prelamanja i jodni broj), (2) sinteza CaO/γ-Al_2O_3, (3) katalitičko krekovanje korišćenog ulja za kuvanje korišćenjem sintetisanog katalizatora i (4) karakterizacija i identifikacija biobenzina. Rezultati su pokazali da su optimalni uslovi za katalitičko krekovanje dobijeni na temperaturi kalcinacije od 650 °C. Sintetizovani biobenzin ima sledeće karakteristike, *tj.* gustinu od 0,776 g ml^{-1}, viskoznost od 1,84 mm^2 s^{-1}, indeks prelamanja od 1,43 i jodni broj od 22,85 g I_2 na 100 g. Sintetizovani biobenzin sadrži jedinjenja alkana, alkena i karboksilnih kiselina, C_2-C_{19}. U ovom sastavu dominirala su jedinjenja koja pripadaju biobenzinu (C_5-C_{12}) u iznosu od 94,5 mas.%, što je potvrđeno analizama gasnom hromatografijom sa masnom spektrometrijom.

Ključne reči: biogorivo; biomasa, heterogeni katalizator; oksid; otpadno ulje; modifikovani materijal