

H. Waisi et al. Influence of process parameters on transesterification reaction of corn oil ...

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Scientific paper

ISSN 0351-9465, E-ISSN 2466-2585

UDC:665.333.4.033

doi: 10.5937/ZasMat1603473W



Zastita Materijala 57 (3)  
473 - 479 (2016)

## Influence of process parameters on transesterification reaction of corn oil over base promoted $\gamma$ -alumina

### ABSTRACT

Due to the frequent use of fossil fuels, which has negative effects on the environment, there is a need to find a new, environmentally acceptable replacements for fossil fuels. One of the possible replacement is the inclusion of green technologies, in order to obtain the type of fuel that would be acceptable from an environmental and economic point of view. Biodiesel represents renewable and less toxic substituent for fossil fuels, which consists of esters of higher fatty acids and depending on the environmental conditions, can be manufactured from various types of oils, both plant and animal origin. Since corn is one of the most cultivated plants in Serbia, the research included the transesterification reaction of corn oil on a heterogeneous catalyst. The paper examined the activity of heterogeneous base catalyst ( $\text{CaO} / \gamma\text{-Al}_2\text{O}_3$ ) and the influence of various parameters on the conversion of corn oil. From the optimization of process parameters, it was found that the optimal conditions for transesterification of corn oil to 25%  $\text{CaO} / \gamma\text{-Al}_2\text{O}_3$ : molar ratio of methanol to oil 1:12; stirring speed 900rpm; reflux temperature of the methanol; reaction time of 6 hours; the amount of catalyst in the reaction of 5wt. %.

**Keywords:** corn oil, heterogeneous catalyst, process parameters, transesterification

### 1. INTRODUCTION

Since the earliest stages of industry development, need for energy has been increased [1]. With the increase of population, the requirement for energy has also been increased. The economic growth is affected by the climatic change, fuel price, and wasting of fuel reserves. Therefore, to increase energy security for economic development, there is a need for finding a new, renewable fuel types [2]. Use of fossil fuels is usually related to environmental concerns, such as air pollution, acid precipitation, forest destruction and increased emission of exhaust gases. With technological progress, renewable energy has grown rapidly in last few decades. Between the scientists, there is an urgent need for the developing different green energy strategies for sustainable future without any negative environmental impacts. One of the possible ways of alternative renewable energy is by using human and agricultural waste materials [3].

Biodiesel is alternative fuel for internal combustion engines and can be used for running diesel engines or it can be used as a mixture. Biodiesel emits low greenhouse gases and it can be used for reducing net carbon dioxide emissions, in comparing to conventional diesel fuel [4]. Biodiesel refers to a diesel-equivalent renewable, sustainable, biodegradable fuel derived by chemical modification or specifically by the transesterification of fats and vegetable oils [5]. Production of biodiesel requires a feedstock (fat or oil) and an alcohol. Typical feedstocks for biodiesel are rapeseed oil, canola oil, soybean oil, sunflower oil and palm oil, while other sources are still exploring [6]. The conventional biodiesel production technique is transesterification by the chemical approach which is a well-developed technology that has been commercialized across the world. Typically, depending on the quality of the feedstock, reactions used for biodiesel production, are esterification or transesterification.

Transesterification reaction can be acid or base catalysed process [7]. Base catalysed transesterification process which uses KOH or NaOH as homogeneous catalyst and methanol is most applied industrial method for biodiesel production. This method has any disadvantages as waste

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Paper received: 11. 04. 2016.  
Paper corrected: 21. 05. 2016.  
Paper accepted: 31. 05. 2016.  
Paper is available on the website:  
www.idk.org.rs/journal

water and process of separation. Heterogeneous catalysis can be used for avoidance of these limitations. Main advantages of heterogeneous catalysis is in reusing catalyst and easier separation of biodiesel from glycerol. Also, waste water is reduced [8, 9]. There is a great varieties of heterogeneous catalysts for transesterification reaction such as modified zeolites, hydro talcites, doped oxides, doped  $\gamma$ -alumina, etc. Most of these catalysts are alkali supported/ promoted alkaline oxides. Recent studies showed that loading of CaO on  $\gamma$ -alumina can lead to high activity of catalyst on transesterification reaction [10]. Maize (*Zea mays* L.) is one of the most important cereal crops, and one of the most significant naturally renewable raw materials for the production of energy [11]. Maize oil has been used for the production of biodiesel, together with other types of oils. Main goal of studies was to improve the properties of biodiesel through applying new technologies [12].

In this sense, main goal of these study was to reveal influence of different preparation process of catalyst on the catalytic performance and also influence of molar ratio, reaction time, steering speed and reaction temperature on transesterification of corn oil over base promoted  $\gamma$ -alumina.

## 2. EXPERIMENTAL

### 2.1. Catalyst preparation

Nano-crystalline gamma alumina support was prepared by modified sol-gel method [13]. The adequate amount of aluminum-tri-sec butoxide (Aldrich Co., purity 97 %) was dissolved in 2-propanol (J.T.Baker, HPLC grade) at room temperature in ultrasonic bath. Than a solution made from the appropriate volumes of 2-propanol and water was added to the solution of aluminum-tri-sec butoxide dissolved in 2-propanol. In next 2 hours drop by drop mixing of solutions was performed on magnetic stirrer. The pH value was maintained at 12.0 with 1 M NaOH solution, and the aging of sol was conveyed for 3 h at 50 °C. The precipitate was dried at 120 °C for 3 h and washed several times with water and 2-propanol. Than precipitate was calcinated at 900 °C for 3 h. The CaO/Al<sub>2</sub>O<sub>3</sub> catalysts were prepared by incipient wet impregnation of  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with desired amounts of Ca(CH<sub>3</sub>COO)<sub>2</sub> aqueous solutions to nominal amounts of CaO (5,10,15,20 and 25 wt. %). After that, the catalysts were dried at 120 °C for 3 h and then the catalysts were activated by thermal treating at 900 °C with a temperature rate of 10 °C/min for 3 h.

### 2.2. Catalyst characterization

The physical and chemical properties of the catalysts were determined by using the X-ray

powder diffraction (XRD) analysis for determination of the crystalline phase and crystallite sizes of the catalysts. Measurements were conducted on Philips AMD 1011 X-ray diffractometer with a radiation source Cu K<sub>α</sub> ( $\lambda = 0.154$  nm) at 40 kV and 50 mA. Data were collected over a  $2\theta$  range 10 – 80 °C with a step size of 0.017 ° at a scanning speed of 5 ° min<sup>-1</sup>. In order to calculate the oxide samples specific surface area and the porosity Brunauer-Emmett-Teller (BET) surface area was calculated using the BET method based on the adsorption/desorption of liquid nitrogen in the range of relative pressures  $p/p_0=0.05-0.25$  and pore volume were calculated from volume adsorbed at the relative pressure of 0.99 by using desorption branch of isotherms by Barrett-Joyner-Halenda (BJH) method. The results concerning textural properties of the catalysts were obtained using a Micromeritics ASAP 2010 instrument.

### 2.3. Transesterification reaction

Commercial edible grade cold-pressed oil from corn germ was obtained from a local market. According to GC-MS analysis (Agilent 5975C), The fatty acid contents consisted of linoleic acid 51.21%, oleic acid 31.27 %, and palmitic acid 13.19%, stearic acid 3.43%, and linoleic acid 0.9%. A 250 mL one-necked glass flask equipped with a water cooled condenser and a magnetic stirrer was charged with adequate volume of corn oil, different volumes, exactly molar ratios of HPLC methanol (from 1:6 to 1:12) and various amounts of catalyst, *i.e.* 1-5 wt. %. Each mixture was stirred at various mixing speeds, *i.e.* 300 RPM-1200 RPM and various temperatures, *i.e.* 20 °C- 65 °C for the required reaction time of (6 h). After separation of reaction products, product determination was conducted by <sup>1</sup>H NMR spectroscopy (Bruker, 400MHz). Reaction mixture was separated from heterogeneous catalyst by filtration and dissolved in chlorophorm. Afterwards, solution was added to separation funnel and mixture was several times washed with water. The organic phase was separated by decantation, dried with anhydrous maghnesium sulfate and submitted to NMR analysis in CDCl<sub>3</sub> using TMS as internal standard.

The conversion of the cold-pressed oil from corn germ to a mixture of methyl esters was determined by the ratio of the signals at 3.68 ppm (methoxy groups of methyl esters) and 2.30 ppm (-carbon CH<sub>2</sub> groups of all fatty acid derivatives) according to previously reported data [14].

## 3. RESULTS AND DISCUSSION

### 3.1. Textural properties

The textural properties of the CaO/Al<sub>2</sub>O<sub>3</sub>-based catalysts samples and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> used as a support

(BET surface area, BJH cumulative desorption pore volume and average pore diameter) are shown in Table 1. Catalytic efficiency in the

transesterification of corn oil over the heterogeneous  $\text{CaO}/\text{Al}_2\text{O}_3$ -based catalyst could be strongly affected by these textural properties.

Table 1 - The textural properties of the  $\text{CaO}/\text{Al}_2\text{O}_3$ -based catalysts samples and  $\gamma\text{-Al}_2\text{O}_3$  used as a support

| Catalyst                                      | BET surface area ( $\text{m}^2 \text{g}^{-1}$ ) | BJH cumulative desorption pore volume ( $\text{cm}^3 \text{g}^{-1}$ ) | Mean pore diameter (nm) |
|---|---|---|-------------------------|
| $\gamma\text{-Al}_2\text{O}_3$                | 141.72  | 0.32  | 6.22                    |
| 5% $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$  | 117.33  | 0.19  | 8.32                    |
| 10% $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$ | 68.31   | 0.18  | 10.13                   |
| 15% $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$ | 38.56   | 0.21  | 15.15                   |
| 20% $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$ | 13.45   | 0.17  | 18.71                   |
| 25% $\text{CaO}/\gamma\text{-Al}_2\text{O}_3$ | 8.86  | 0.16  | 21.62                   |

The results presented in Table 1. are indicating that after impregnation of  $\text{CaO}$  onto the  $\gamma\text{-Al}_2\text{O}_3$  support, there was a significant reduction of the BET surface area. BET surface area was decreased as the loading amount of  $\text{CaO}$  was increased on the  $\gamma\text{-Al}_2\text{O}_3$  support. Also, it was observed a reduction in pore volume values after impregnation of  $\text{CaO}$  onto the  $\gamma\text{-Al}_2\text{O}_3$  support. The decrease in the BET surface area and the BJH cumulative desorption pore volume is probably connected to the blocking of the pores present in the support by  $\text{CaO}$  [15, 16]. The pore system is in the mesopores range for all catalysts with maxima in pore size between 4-8 nm and 15-25 nm with an average pore diameter from around 6 nm to around 21 nm in size according to the BJH desorption isotherms. The average pore diameter of the

$\text{CaO}/\text{Al}_2\text{O}_3$  catalysts is higher than 2.5 nm and this may be important because of supporting effective process of mass transfer of triglycerides (namely, around 2.5 nm is methyl-oleate molecule in size [17], during the catalytic run of reactants conversion.

### 3.2. Structural properties

The XRD patterns of  $\text{CaO}/\text{Al}_2\text{O}_3$ -based catalysts samples and  $\gamma\text{-Al}_2\text{O}_3$  used as a support are presented in Figure 1. The XRD patterns of the catalyst support exhibit typical  $2\theta$  reflections of  $\gamma\text{-Al}_2\text{O}_3$  at  $33.7^\circ$ ,  $45.7^\circ$  and  $66.7^\circ$  (JCPDS 50-0741). As expected, characteristic  $2\theta$  reflections originating from  $\gamma\text{-Al}_2\text{O}_3$  are present in all catalyst samples, differ only in intensity of characteristic peaks.

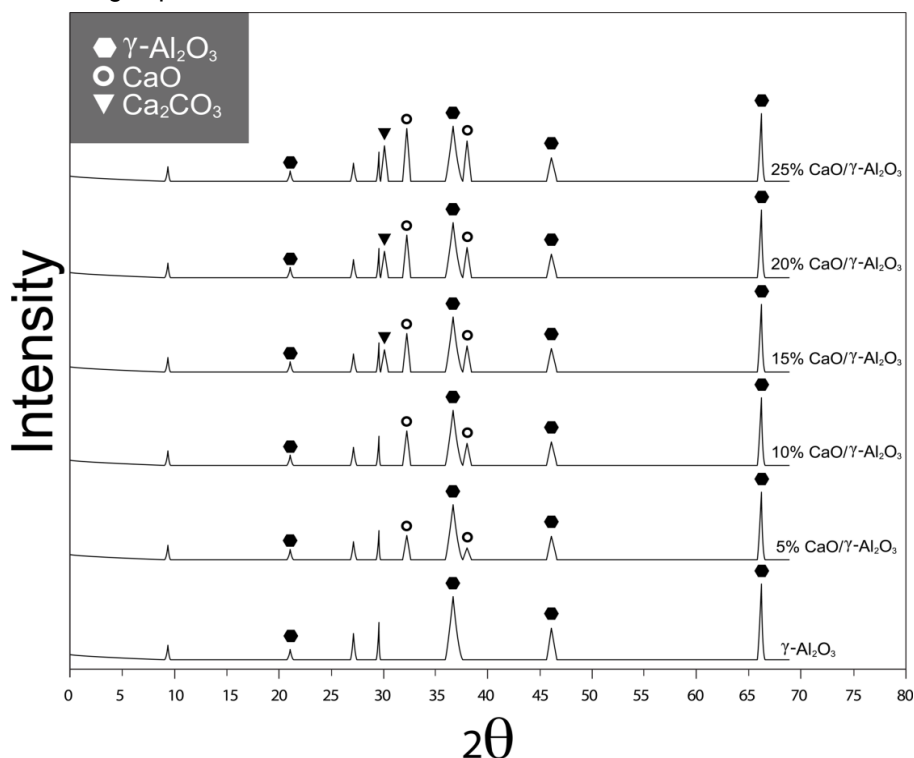


Figure 1 - Influence of the  $\text{CaO}$  loading amount onto  $\gamma\text{-Al}_2\text{O}_3$  on catalytic activity

The presence of calcium acetate used as the precursor salt was not registered on diffractograms, probably due to high calcination temperature (900°C). The existence of CaO in all catalysts has been confirmed and detected by diffractograms reflections at ( $2\theta=32.3^\circ$  and  $37.4^\circ$ , JCPDS 82-1690, [10]). For both reflections, especially for  $2\theta=37.4^\circ$ , broadening of peaks with increasing the amount of CaO is evident. Calcium carbonate present in the catalyst samples was identified with characteristic reflection at  $2\theta=29.5^\circ$  (JCPDS 50-0741). The occurrence of small amounts of  $\text{CaCO}_3$  on the surface of the catalyst might be caused by the

contact of the catalyst with  $\text{CO}_2$  from the air during the measurement procedure.

As expected, increase in CaO loading amount on to  $\gamma\text{-Al}_2\text{O}_3$  support, as shown in Figure 2., increase conversion of catalysts in transesterification reaction of corn oil with methanol, probably due to increased basicity of catalysts. Main results gained for the most optimal conditions for transesterification reaction of corn oil over base promoted  $\gamma$ -alumina, compared with similar research [10] showed that probably higher amount of active CaO species in reaction mixture is responsible for higher efficiency and level of conversion of corn oil.

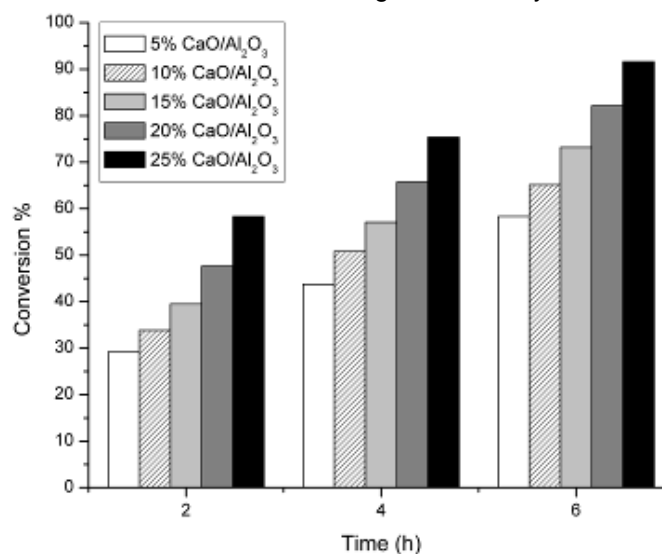


Figure 2 - Influence of the CaO loading amount on to  $\gamma\text{-Al}_2\text{O}_3$  on the transesterification reaction conversion

In order to shift the reaction equilibrium towards the final products, higher molar ratios than the stoichiometrically determined (3:1 = methanol: corn oil) were tested. As we can see in Figure 3., excess of methanol has increased reactants conversions up to molar ratio of 1:12, which corresponds to previously researched results of other authors [18,19].

This is relatively complicated reaction mixture consisted of three-phases, so the fact where the excess of methanol has increased reactants conversions can be explained with reduction of internal and external diffusion restrictions and limitations over catalytically active sites in the catalyst bulk and surface.

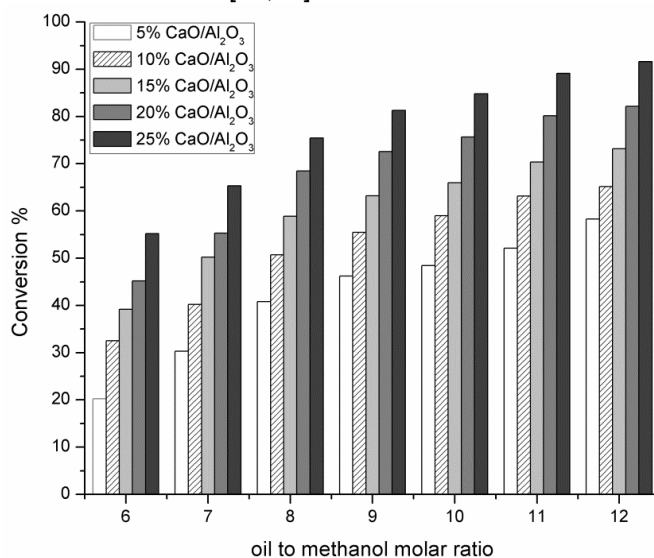


Figure 3 - Influence of molar ratio (methanol to corn oil) on the transesterification reaction conversion

Different catalysts loadings between 1 and 5 wt. % were tested in the transesterification reaction of corn oil with methanol. As presented in Figure 4., the use of larger catalyst loadings in the reaction mixture has caused higher conversions to products. This can be explained by fact that greater amounts of the catalysts provided greater number of catalytically active sites and more effective contacts. In addition, further increase of the

catalyst loadings could result with a shortening of reaction time in order to achieve the same conversions [20].

It is shown in Figure 5., that conversions have increased when longer reaction times have been conducted. After 5 hours from the start of the reaction it can be observed a slight increase in conversions, and further observations of reaction time showed constant values of conversion.

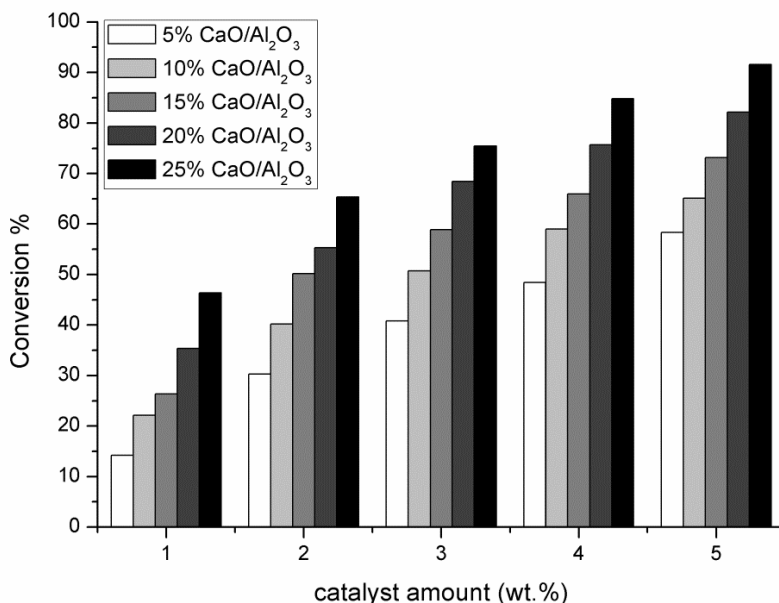


Figure 4 - The influence of catalysts loading amount on reactants conversions in reaction of transesterification of corn oil with methanol

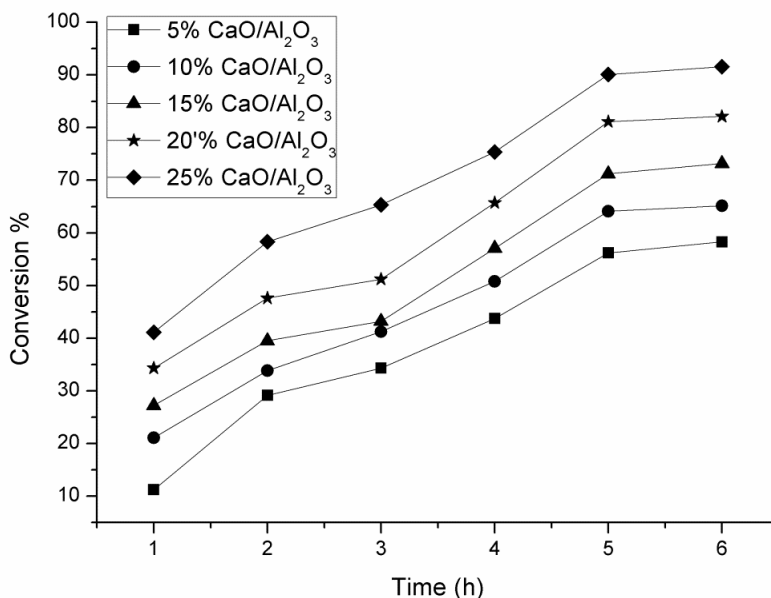


Figure 5 - Influence of reaction time on the transesterification reaction conversion.

The influence of reaction stirring speed is shown in Table 2. Increase in reaction stirring speed results in increase of conversions till

maximum conversions point has been reached and furthermore increase in reaction stirring speed has no impact on reaction conversions.

Table 2 - Influence of stirring speed on conversions in transesterification reaction of corn oil with methanol

| Conversion (%)       |  |   |   |   |   |
|----------------------|--|---|---|---|---|
| Stirring speed (RPM) | 5% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 10% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 15% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 20% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 25% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> |
| 300                  | 20.05  | 31.12   | 42.15   | 47.48   | 57.11   |
| 600                  | 50.17  | 59.14   | 68.15   | 75.17   | 82.21   |
| 900                  | 58.99  | 64.32   | 74.28   | 82.15   | 91.53   |
| 1200                 | 59.05  | 64.37   | 74.31   | 82.18   | 91.58   |

Table 3 - Influence of reaction temperature on conversions in transesterification reaction of corn oil with methanol

| Conversion (%)   |  |   |   |   |   |
|------------------|--|---|---|---|---|
| Temperature (°C) | 5% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 10% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 15% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 20% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> | 25% CaO/ $\gamma$ -Al <sub>2</sub> O <sub>3</sub> |
| 20               | 10.31  | 14.51   | 21.51   | 25.37   | 31.05   |
| 30               | 20.11  | 30.08   | 42.58   | 47.32   | 56.91   |
| 40               | 36.71  | 47.38   | 54.32   | 64.01   | 71.15   |
| 50               | 50.05  | 57.32   | 60.38   | 74.31   | 82.05   |
| 65               | 59.05  | 64.37   | 74.31   | 82.18   | 91.58   |

The influence of reaction temperature on conversions in transesterification reaction of corn oil with methanol are shown in Table 3. Increase in reaction temperature results in increase of reaction conversions, probably because the increase in temperature affects the energy of reactants which leads to an increased number of successful molecular collisions.

#### 4. CONCLUSIONS

CaO impregnated  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> prepared and activated at high temperature has shown itself as a very active catalyst in transesterification of corn oil with methanol. This base promoted  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> catalytic system shown many advantages in biodiesel production process such as: relatively low operating temperature (reflux of methanol) and atmospheric pressure, acceptable molar ratios of reactants (1:12), relatively short contact time (6 h) and reasonably small catalyst loadings (5 wt. %). These all together resulted in high conversions to fatty acids methyl esters. Based on physico-chemical properties of the CaO/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub> catalysts such as acceptable pore volume and average pore diameter, the presence of desirable crystal phases of CaO and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub>, the absence of external and internal diffusion restrictions all together found to be the reasons for the obtained high catalytic activity.

The highest conversions of 91.58 % to fatty acids methyl esters was achieved by using the catalyst impregnated with 25 wt.% of CaO on  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> support under relatively mild conditions (methanol to oil molar ratio 1:12, stirring speed of 900 RPM, reflux temperature of methanol, reaction time 6 h and catalyst loading of 5 wt. %). It is obvious that process parameters as well as

catalyst physico-chemical properties have great impact on transesterification reaction of corn oil over base promoted  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> with methanol.

#### Acknowledgements

This research work was partially supported by the Serbian Ministry of Education, Science and Technological Development under the project number TR31080.

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## IZVOD

### UTICAJ PROCESNIH PARAMETARA NA TRANSESTERIFIKACIJU KUKURUZNOG ULJA NA BAZNO PROMOVISANOJ $\gamma$ - ALUMINI KAO HETEROGENOM KATALIZATORU

Zbog sve učestalijeg korišćenja fosilnih goriva, koja imaju nepoželjne efekte na životnu sredinu, postoji potreba za pronalaženjem nove, ekološki prihvatljive zamene za fosilna goriva. Jednu od mogućih zamena predstavlja uključivanje zelenih tehnologija radi dobijanja vrste goriva koje bi bilo prihvatljivo sa ekološkog i ekonomskog aspekta. Biodizel predstavlja obnovljiv i manje toksičan substituent za fosilna goriva, koji se sastoji od estara viših masnih kiselina i u zavisnosti od podnevlja, može se proizvoditi od različitih vrsta ulja, kako biljnog, tako i životinjskog porekla. Obzirom da je kukuruz jedna od najviše gajenih vrsta biljaka u Srbiji, istraživanja su obuhvatila reakciju transesterifikacije kukuruznog ulja na heterogenom katalizatoru. U radu je ispitivana aktivnost heterogenog baznog katalizatora (CaO/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>) kao i uticaj različitih parametara na konverziju kukuruznog ulja. Optimizacijom procesnih parametara je ustanovljeno da su optimalni uslovi za transesterifikaciju kukuruznog ulja na 25% CaO/ $\gamma$ -Al<sub>2</sub>O<sub>3</sub>: Molarni odnos metanola prema ulju 1:12; brzina mešanja 900rpm; temperatura reakcije refluks metanola; vreme reakcije 6 sati; količina katalizatora u reakciji 5%.

**Ključne reči:** kukuruzno ulje, heterogeni katalizator, procesni parametri, transesterifikacija.

Naučni rad

Rad primljen: 11. 04. 2016.

Rad korigovan: 21. 05. 2016.

Rad prihvaćen: 31. 05. 2016.

Rad je dostupan na sajtu: [www.idk.org.rs/casopis](http://www.idk.org.rs/casopis)