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ENGINEERING, ENVIRONMENT
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11th - 13th March 2019

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**“ENGINEERING, ENVIRONMENT AND MATERIALS IN
PROCESSING INDUSTRY”**

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DEACTIVATION ASPECTS OF METHANOLYSIS CATALYST BASED ON CaO LOADED ON MESOPOROUS CARRIER

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Abstract

With the depletion of fossil fuels as a source for fuels and energy, the share of energy supplied by renewable resources such as biomass can be expected to increase in the anticipated future. Biodiesel, composed of fatty acid methyl esters (FAME), is one of the renewable alternative to fossil fuels which is most common obtained in catalyzed transesterification reaction of triacylglycerols (TAG). Nowadays, the most promising process is considered continuous basic heterogeneously-catalyzed methanolysis of sustainable sources of TAG. Heterogeneously-catalyzed process has numerous advantages, but some drawbacks also exists. One of the main concern is catalyst stability and durability. The aim of this study is catalyst durability investigation and determination of the deactivation mechanisms on the case of CaO loaded onto mesoporous neutral carrier. The experiments were carried out in continuous packed bed tubular reactor (PBR) under mild reaction conditions. The CaO-based catalyst showed high activity in continuous reaction conditions during the first 30 h of operation, the FAME content was higher than 91.5%. After 30 h the catalyst activity began to decrease, so that after 36 h the FAME content was only 36%. The analysis of the morphology and the present functional groups on the surface of the catalyst before and after the reaction clearly showed a massive deposition of organic molecules from the reaction mixture on the surface of the catalyst. Since the loss of the active species by leaching was negligible, the sheltering of the catalytic active sites by such massive deposition of the organic molecule resulted in a decrease in the activity of the used CaO-based catalyst.

Key words: Biodiesel, CaO-based catalyst, catalyst deactivation, heterogeneous methanolysis, mesoporous catalyst carrier.

Introduction

Global uncertainty about widely used fossil fuels and the ecological consequences of its use have led to the development of a large number of alternative fuels. One of them is biodiesel, a biorenewable fuel with a lower emission of pollutants, which can be used in existing vehicles engines (Marinković et al., 2016). Biodiesel are mixture of different fatty acid alkyl esters (FAAE) and generally is obtained in catalyzed transesterification reaction of triacylglycerols with short chain alkyl alcohols, wherein glycerol is obtained as a by-product. Homogeneous methanolysis of vegetable oils and fats is a commercially widely used process for biodiesel production, but in recent years the scientific focus has been shifting to a development of viable heterogeneous process. In addition to the numerous advantages of heterogeneous methanolysis in relation to homogenous (catalyst reusability, regenerability, separability and less demanding product purification step), the commercialization of the heterogeneous process should overcome some existing problems: slower reaction rate, catalyst leaching in the reaction mixture and somewhat rapid deactivation of the catalyst during repeated batch cycles or prolonged continuous use (Marinković et al., 2016). The main challenge of heterogeneously catalyzed transesterification is achieving not only a high yield but also its stability and durability. In the

literature, the two problems related to base catalyst, especially in the case of CaO-based catalysts are dominantly emphasized: (a) adsorption of moisture and CO₂ from the air on the base sites and (b) Ca leaching into the reaction medium (Kouzu and Hidaka, 2012). The first problem can be solved by reactivation of the catalyst using thermal or chemical means, making the overall process more complicated and expensive (Marinković et al., 2016). Despite the some CaO-based catalysts that are deactivated in only a few minutes upon being exposed to the air (Kouzu and Hidaka, 2012), the present CaO/ γ -Al₂O₃ catalysts adsorbed no significant amounts of H₂O or CO₂ for 20 min (Marinković et al., 2017). Regarding leaching, this process not only reduces the catalyst amount but also contaminates the reaction products (Marinković et al., 2016).

Many studies provide information on the methods of reactivation of the spent catalysts (Stojković et al., 2014) (Urasaki et al., 2012), with reference to thermal or mechanical treatment (Ho et al., 2014) (Atadashi et al., 2013), drying (Soares Dias et al., 2013) and rinsing with adequate solution (Banković-Ilić et al., 2014). Where, a satisfactory activity of the treated catalyst is obtained after reactivation. However, details on the reasons for the deactivation of these catalysts are still scarce. The focus of this paper is on discovering the deactivation mechanism of a spherical mesoporous CaO-based catalyst in methanolysis reaction.

Experimental

The catalytically active species (CaO) was loaded on the γ -Al₂O₃ carrier by the method of impregnation. Obtained catalyst precursors were activated for 4 h under suitable temperature regime using inert atmosphere. The catalyst had a spherical shape with diameter of 1-3 mm. Details including material preparation and activation optimization study were previously reported (Marinković et al., 2017). Also, comprehensive physicochemical, textural and morphological analyses of all obtained catalytic materials were given elsewhere (Marinković et al., 2017). Disambiguation on the name of the catalyst indicates the precursor salt (Ac for calcium acetate salt and NO₃ for nitrate salt) and the calcination temperature (in °C). The continuous sunflower oil methanolysis was conducted in packed bed tubular reactor under the following reaction conditions: the catalyst packed bed height of 27 cm, the methanol- to-oil molar ratio of 6:1, the reaction temperature of 60 °C, the atmospheric pressure and the liquid hour space velocity (LHSV) of 0.24 h⁻¹. Leaching of Ca into the reaction mixture from the surface of the CaO(NO₃)₄₇₅ catalyst was studied in the continuous PBR with the catalyst bed height of 12 cm, the methanol-to-oil molar ratio of 6:1, the reaction temperature of 60 °C and LHSV of 0,9 h⁻¹. After the reaction was carried out, the catalyst were separated from the reaction mixture, briefly washed with methanol and without further treatment analytical characterization of the samples was carried out.

The FAME content in the reaction mixture was monitored by means of liquid chromatography (HPLC, Agilent 1100 Series and Zorbax Eclipse XDB-C18 column, the mobile phase was methanol and 2-propanol/n-hexane (5:4 v/v)). Ca concentration in the reaction mixture was analyzed by atomic emission spectroscopy (ICP-OES, axial Thermo Scientific iCAP 6500 Duo CP with closed wet microwave digestion system, ETHOS 1, Milestone). The catalysts porous texture are determined by Hg-porosimetry (Thermo Fisher, Pascal 140 and 440 series), while the changes on surface properties and structural analysis were analyzed using scanning electron microscopy (FE-SEM, Tescan MIRA3 XMU, 10 kV, 30.000x), infrared spectroscopy (ATR-FTIR, Thermo Scientific Nicolet 6700 FT-IR Smart iTR with KBr splitter and DTGS detector) and X-ray diffraction (XRPD, Philips PW-1710 automated diffractometer, *Cu-K α* radiation at 40 kV) techniques.

Results and Discussion

The obtained catalysts were tested in the methanolysis reaction performed in continuous PBR under mild conditions. One of the materials, CaO(NO₃)₄₇₅, maintained high catalyst activity over 30 h of continuous operation. During the first 24 h the FAME content was higher than

98.4%, after which the yield slowly decreased, so that after 30 h the activity of the catalyst decreased sharply. Finally, the FAME content was only 34% after 36 h of operation. In the similar reactor system, the yield reduction has been explained by clogging and channeling of the catalyst bed due to agglomeration of small quicklime bits (Miladinović et al., 2015). This negative phenomenon was not observed in the case of a relatively larger spherically-shaped catalyst particles used in the present study.

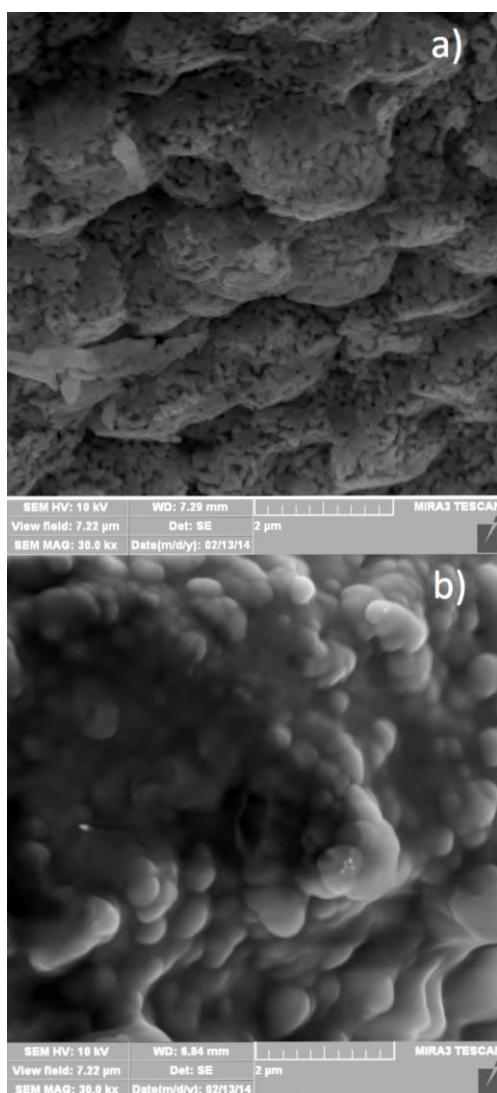


Figure 1. The SEM images of the CaO(Ac)₆₅₀ catalyst before (a) and after the reaction (b).

The porosimetry results of fresh catalysts and their changes after the reaction are presented in Table 1. All morphological characteristics of the used catalysts (pore volume, surface area, particle diameter and overall porosity) are significantly lower than for fresh materials. After the reaction, the specific surface of the most active catalyst (CaO(NO₃)₄₇₅) (Marinković et al., 2018) decreased by over 40%, the total porosity by over 36%, and the average pore diameter increased by 20%. An obviously significant reduction in total pore volume, with other parameters, is the result of deposition of organic molecules from the reaction mixture within the pores and on the surface of the catalyst.

The apparent change in the morphology of the surface of the catalyst used in the reaction is shown in Figure 1. The images from electronic microscope are in accordance with the porosimetry results. After the reaction (Figure 1b) the complete surface of the catalyst is covered by organic molecules from the reaction mixture. Fresh catalyst (Figure 1a) had the distinct particles and spherical clusters of particles on the surface, while the complete surface appears polished after the reaction. The degree of organic molecules deposition is such that surface of the catalyst after the reaction look almost unrecognizable.

Table 1. Morphological characteristics of fresh catalysts and their changes after the reaction.

Sample designation	V_{cum} , $cm^3 g^{-1}$	Relative change, %	S_{Hg} , $m^2 g^{-1}$	Relative change, %	D_{av} , nm	Relative change, %	P, %	Relative change, %
CaO(Ac) ₅₀₀	0,538	39,4	112,6	40,3	16	12,5	61,4	28,3
CaO(Ac) ₅₅₀	0,592	61,8	107,1	67,9	16	50,0	66,8	51,4
CaO(Ac) ₆₀₀	0,549	35,5	105,2	33,7	16	25,0	64,2	18,4
CaO(Ac) ₆₅₀	0,588	10,5	105,9	93,2	20	-	66,6	82,4
CaO(NO ₃) ₄₂₅	0,322	17,1	59,4	21,2	20	20,0	49,8	21,1
CaO(NO ₃) ₄₅₀	0,350	3,7	64,8	14,2	20	0,0	53,2	12,1
CaO(NO ₃) ₄₇₅	0,455	42,6	89,4	40,7	20	20,0	58,7	36,3
CaO(NO ₃) ₅₀₀	0,482	74,3	97,2	77,3	16	50,0	62,7	68,2

V_{cum} – cumulative pore volume, S_{Hg} – specific surface area, D_{av} – average particle diameter, and P – porosity.

The presence of the FAME molecules on the surface of the used catalyst was confirmed by the infrared analysis (Figure 2). The spectral lines between $1.000\text{--}1.300\text{ cm}^{-1}$ correspond to the vibration of the C-CH₂-O group, the asymmetric stretching of C-O-C bond, and the stretching of the C-C bond. An intense line at about 1.425 cm^{-1} originates from the asymmetric stretching of the C-H bond and asymmetric bending of the same functional group. The most intense spectral line, especially characteristics for esters, is present at 1.740 cm^{-1} and can be assigned to C=O vibrations. Finally, an intense dual bend between $2.850\text{--}2.920\text{ cm}^{-1}$ is correspond to the C-H vibration of methylene groups and stretches and compression of the methyl group. A wide knee line at about 3.460 cm^{-1} can be attributed to the vibrations of the hydrogen bond of the hydroxyl group, i.e. can be associated with the presence of glycerol (Salehpour and Dube, 2012). The appearance of this spectral line may be from residual glycerol due to insufficient rinsing of the samples with methanol after the reaction. All of these spectral lines, except last one, are characteristics of FAME compounds (Guerrero et al., 2013).

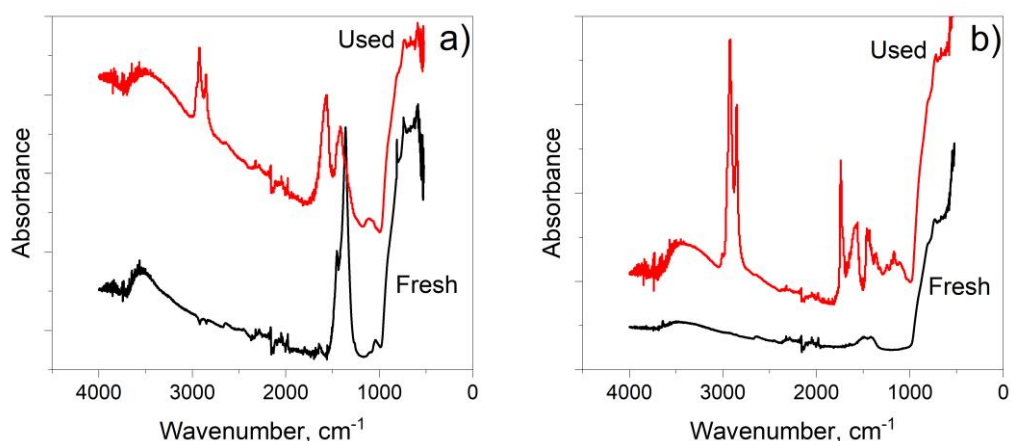


Figure 2. Vibrational spectra (ATR-FTIR) of the fresh and used catalyst, $\text{CaO}(\text{NO}_3)_{425}$ (a) and $\text{CaO}(\text{Ac})_{650}$ (b).

X-ray diffractograms of the catalysts after the methanolysis reaction are presented in Figure 3. As can be seen, diffractograms of the used catalyst are very similar to the fresh one. Only noticeable change after the reaction was the appearance of broad peak centered on $2\theta = 20^\circ$. This emerged peak could be attributed to the deposited residual glycerol or some glycerol compound (e.g. potentially formed calcium diglycerate (Gupta and Rathod, 2018)), as has already been observed at ATR-FTIR analysis.

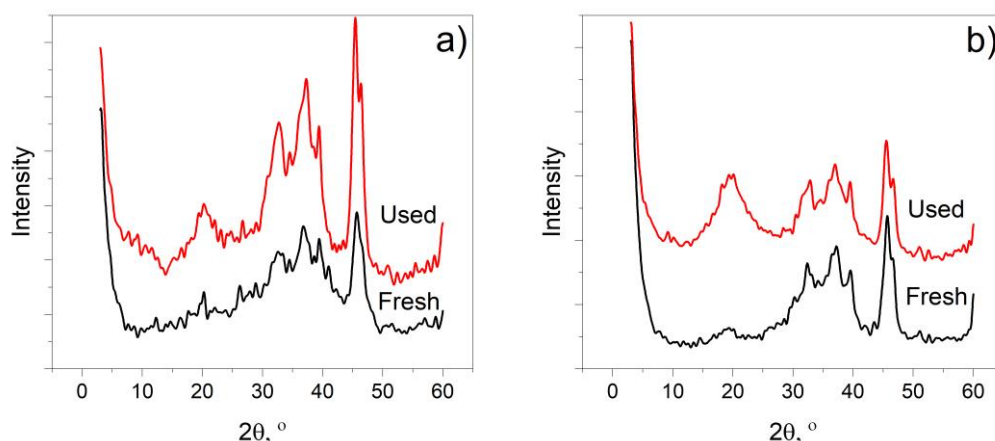


Figure 3. XRD diffractograms of the fresh and used catalyst, $\text{CaO}(\text{NO}_3)_{425}$ (a) and $\text{CaO}(\text{Ac})_{650}$ (b).

Leaching of a catalytically active species in the reaction mixture is one of the most important problems in heterogeneously catalyzed transesterification, because it irreversibly reduces the possibility for repeated and prolonged use of catalyst. Additionally, the reaction products are contaminated by the leached species so purification step need to be applied in order to achieve

the required product quality. Data on CaO leaching in methanol, glycerol, biodiesel, or entire reaction mixture are often inconsistent in literatures, and the reason for this should be found in the specifics of each investigated reaction system individually.

In present study, as shown in Figure 4, the leached Ca concentration in the biodiesel phase increased over time from 9.2 mg/kg after 3 h to 50.9 mg/kg after 10 h. The Ca leaching rate slowed down with time, which was attributed to the decrease of the concentration gradient. The obtained leached Ca concentrations were in accordance with the literature data for the CaO-

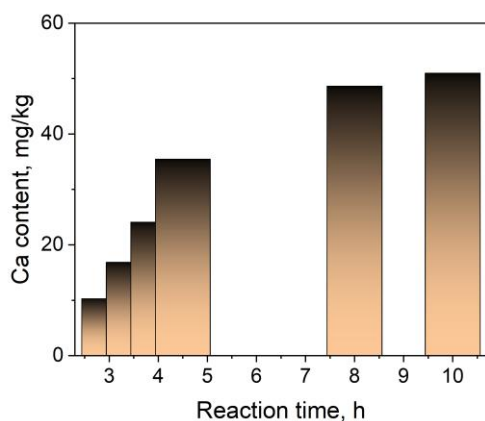


Figure 4. Ca leaching in the biodiesel with time in continuous reaction conditions.

based catalysts. In the case of the palm oil methanolysis catalyzed by CaO/Al₂O₃ catalyst, the Ca concentration in the reaction mixture was 31 mg/kg (Zabeti et al., 2009). Crude sunflower oil-based biodiesel obtained in a PBR with a quicklime bits contained 38 mg/kg of Ca (Miladinović et al., 2015). In the batch systems with CaO·ZnO and CaO catalysts, the leached Ca concentration in methanol were 62 mg/kg and 91 mg/kg, respectively (Lukić et al., 2013). However, some authors also obtained a significantly higher Ca lixiviation in the reaction mixture, although for different reaction conditions (Yoosuk et al., 2010) (Verziu et al., 2011). It should be noted that Ca is more soluble in the reaction mixture (methanol + biodiesel + glycerol) than in methanol or biodiesel alone (Marinković et al., 2016).

Conclusions

The deactivation mechanism was studied for the methanolysis reaction catalyzed by the CaO-based catalyst under the mild reaction conditions in a continuous tubular PBR. After 30 h of high activity of the catalyst in the continuous process, the activity began to decline sharply. The catalytic material was analyzed before and after the reaction using analytical methods of Hg-porosity, XRD, FTIR, and SEM. All applied analytical methods showed significant changes in the morphology and texture on the surface of the catalyst as well as the porous structure before and after the reaction. The following observed phenomenon, which led to a decrease in the catalytic activity in continuous process, was massive deposition of organic molecules from the reaction mixture on the catalyst surface. Analytically proved significant deposition of organic molecules from the reaction mixture on the surface of the catalyst resulted in a reduction of the total specific surface area of the catalyst, by reducing the pore diameter and their completely obstruction. On the other hand, the deposited organic molecules blocked and shelter the catalytically active sites on the catalyst surface. The loss of catalytic activity through leaching of CaO was negligible, because for 10 h of continuous reaction only about 50 mg/kg is leached into reaction mixture.

Acknowledgments

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