# THE IMPACT OF REUSING COMMERCIAL HYDRATED LIME AS A HETEROGENEOUS CATALYST ON THE FATTY ACID METHYL ESTERS CONTENT AND ITS CHARACTERISTICS IN THE METHANOLYSIS PROCESS OF RAPESEED OIL

**ORIGINAL SCIENTIFIC ARTICLE**

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**ABSTRACT:**
**This study investigates the impact of reusing commercial hydrated lime as a heterogeneous catalyst on the content of fatty acid methyl esters (FAME) and its characteristics in the methanolysis process of rapeseed oil. The reuse evaluation was conducted by performing consecutive methanolysis reactions up to three times under optimal reaction conditions. Characterization of both unused (fresh) and used catalyst (after the first use and third reuse) was carried out using XRD and SEM/EDS methods, in order to examine structural and morphological changes associated with the catalytic activity of hydrated lime. After the first, second, and third consecutive reuses of commercial hydrated lime under optimal methanolysis conditions, FAME content of 95.46%, 93.92%, and 80.01%, respectively, were achieved. The aforementioned FAME content is high but it is below the minimum ester content requirement set by the EN 14214. Therefore, repeated use of hydrated lime reduces its catalytic activity, resulting in lower FAME content compared to the content obtained with fresh catalyst (96.7%). It was demonstrated that the extent of catalytic activity reduction is correlated with the number of reuses, but also depends on the chemical, structural, and textural properties of the catalyst. XRD analysis of the catalyst after the first use and third reuse revealed the formation of a new crystalline phase, calcium diglyceroxide ($C_6H_{14}CaO_6$), identified as the primary crystalline phase, along with $Ca(OH)_2$ and $CaCO_3$ as secondary crystalline phases.**

**KEYWORDS:** biodiesel, methanolysis, fatty acid methyl esters, heterogeneous catalyst, hydrated lime, reuse

## INTRODUCTION

The depletion of fossil fuel reserves and the growing environmental concerns related to their production and combustion have led to an exponential increase in interest in biofuels [1]. Among these, biodiesel has attracted significant attention due to its characteristics such as high biodegradability, non-toxicity and low emissions of carbon monoxide, particulate matter and unburned hydrocarbons [2], [3]. Biodiesel is a liquid biofuel composed of monoalkyl esters derived from vegetable oils, animal fats, or other lipid-based feedstocks such as used cooking oil. Since methanol is the most commonly used alcohol in biodiesel production, another name for biodiesel is fatty acid methyl esters (FAME). Due to its physicochemical properties similar to those of conventional diesel fuel, biodiesel can be used in unmodified compression-ignition engines, either in its pure form (designated as B100) or in blends with petroleum diesel [4], [5], [6], in which it also enhances fuel lubricity and increases the cetane number. Its use reduces life-cycle emissions because the carbon dioxide released during biodiesel combustion is offset by the $CO_2$ uptake during the cultivation of the plant-based feedstocks used for its production. Liquid biofuels such as biodiesel are considered as preferred alternative to petroleum-based fuels, as they entail relatively low additional infrastructure costs and provide a direct pathway for decarbonizing the transportation sector [7]. Additionally, the development of domestic biodiesel production offers developed countries an opportunity to meet their emission reduction obligations under the Kyoto Protocol [8], while for developing countries it serves as a way to reduce dependency on fuel imports by enhancing energy security. The economic impact of biodiesel is not limited to the biodiesel industry and the agricultural sector alone, but extends across the entire economy [9], due to the interconnections among the various sectors involved in its production and application. In light of this, considerable attention has been dedicated to research on its production, with the aim of making it more sustainable and cost-effective [10]. Among the existing biodiesel production methods, the conventionally applied technique is the transesterification of vegetable oils with an alcohol, typically methanol (known as methanolysis), in the presence of a catalyst. This process converts the triglycerides in the oil into alkyl esters, producing biodiesel as the main product and crude glycerol as a by-product [11], [12]. Although the transesterification reaction is reversible and primarily proceeds through the mixing of reactants, the use of a catalyst increases the solubility of alcohol in the oil, thereby accelerating the reaction [13], which enables the reaction to occur at low to moderate temperatures.

The main drawback of using homogeneous catalysts in biodiesel production relates to the need for extensive post-treatment of the product, which includes washing and purification steps to remove the catalyst. This increases production costs and, in some cases, may lead to significant product losses. The use of heterogeneous catalysts instead of conventional homogeneous ones helps to overcome existing difficulties in separating biodiesel from the catalyst, as they are in different phases. It also enables the possibility of recycling the catalyst for reuse after the reaction, without the need for intensive washing steps.

The three main groups of catalysts used in methanolysis for biodiesel production are: acidic or alkaline homogeneous catalysts, acidic or alkaline heterogeneous catalysts and enzymatic catalysts [14].

Although significant progress has been made in biodiesel production technology, its commercialization still depends on the availability of suitable catalysts [15]. Besides high catalytic performance, important requirements for these catalysts include rapid separation and efficient recycling after the reaction [16]. Despite their high reactivity, homogeneous catalysts have serious drawbacks, such as the need for equipment resistant to strong acids and bases, inability to regenerate the catalyst and the low quality of the resulting glycerol [10], [17]. Conventional biodiesel production processes at an industrial scale face challenges not only due to the use of catalysts that cannot be regenerated but also due to difficulties in separating the resulting products [14]. Separation of the homogeneous catalyst from the medium is complicated and often not economical [18] and the multiple washing steps required to remove it from the product lead to significant consumption of water, often deionized, as well as the generation of wastewater [19]. The high energy consumption during separation and purification processes is a major drawback of using homogeneous catalytic systems [20]. Due to all these factors, the overall production costs of biodiesel based on homogeneous catalysis are still not competitive enough compared to the costs of petroleum diesel production [11]. Since the use of enzymatic catalysts is impractical on an industrial scale due to their high cost [21], [22] it has been recognized that new processes based on heterogeneous catalysts should be considered to improve the economic efficiency and competitiveness of biodiesel production [23]. The introduction of heterogeneous catalysts into the commercial biodiesel production process could reduce its cost and make it competitive with fossil diesel because there is various advantages that these catalysts offer: higher reaction rates, easy separation from the product and the possibility of reuse [24]. Generally, heterogeneously catalyzed biodiesel production processes involve fewer unit operations with simple product separation and purification steps and do not require any neutralization procedures [25].

## EXPERIMENTAL

### MATERIAL AND METHODS

#### Materials

The following materials were used in the experimental part of the research:

- commercial hydrated lime (Stamal Ltd. Kreševo),
- refined rapeseed oil (Bimal Ltd. Brčko),
- methanol p.a.,
- other materials and chemicals needed to carry out the necessary analyses.

#### Methods

Determination of structural properties of the catalyst was done using the X-ray diffraction (XRD) method. The diffractogram of the hydrated lime sample after its third reuse in the methanolysis process was recorded on an automatic X-ray powder diffractometer Philips PW1710, at an operating voltage of $U = 40kV$ and a current of $I = 30mA$. CuKα radiation with a wavelength of $\lambda = 1.54056$ Å was used, monochromatized using a graphite monochromator. Before starting the recording of the samples, the accuracy of the diffractometer device was controlled using the basic program PW-1844. The step size was 0.02º 2θ with a time delay of 1 second at each step. The angular recording interval was 2-80º 2θ. Diffraction data were collected at room temperature. Based on the obtained values of intensity I (imp) and interplanar distances d (Å), by comparison with literature data and ICDD standards, identification was made. XRD analyses of the fresh commercial hydrated lime sample and after its first use in the methanolysis process were conducted at different institutions and therefore the presentation of the diagrams varies.

Determination of morphological properties of hydrated lime was performed by scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM-EDS), using JEOL JSM 6420 LV scanning electron microscope, with an acceleration voltage of 20 kV. The sample was coated with a 15 nm layer of gold, with a density of 19.32 g/cm$^3$.

The methanolysis conditions identified as optimal according to previous studies [26], [27] were used to investigate the reuse of the catalyst. The methanolysis process was carried out under defined process conditions.

Methanolysis experiments were conducted in a 500 cm$^3$ three-neck round-bottomed glass flask, coupled to a water cooling recirculation system. Stirring and heating were performed using a mechanical stirrer and a hot plate. First, 250 cm$^3$ of refined rapeseed oil was added to the flask, along with the 62.5 cm$^3$ of methanol (the methanol-to-oil volume ratio was 0.25). The catalyst concentration (wt.%) relative to the mass of the rapeseed oil used was 4 wt.%. The reaction was conducted at a temperature of 60°C and the duration time was 120 minutes. The stirring speed during the reaction was kept constant at 1000 rpm.

The apparatus that was used to carry out the heterogeneous-catalyzed methanolysis of rapeseed oil shown in Figure 1 consisted of the following components:

1. electric heater with thermostat,
2. glass cup,
3. three-necked flask with a volume of 500 cm$^3$ with a round bottom,
4. mechanical mixer,
5. temperature probe,
6. water cooler.

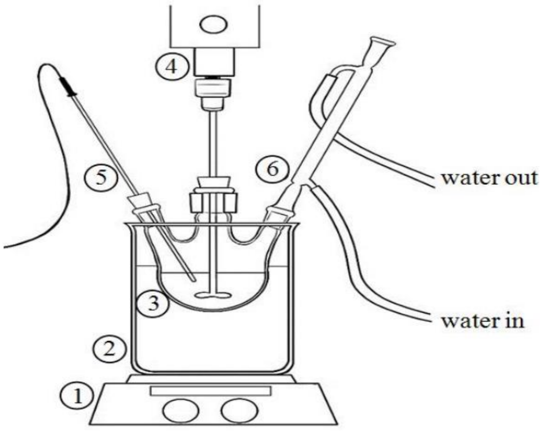

**Figure 1**. Laboratory apparatus for obtaining biodiesel

After the expiry of the set methanolysis time, the liquid phase was separated from the catalyst by a vacuum pump and left for 24 h in a funnel to separate the fractions of biodiesel (methyl ester) and glycerol based on their different specific gravity. Then the lower (glycerol) phase was discharged from the funnel, and the separation of fine catalyst particles and residual glycerol from the biodiesel fraction was performed by centrifugation at 3000 rpm (Eppendorf Centrifuge 5702) for 10 minutes at room temperature. The biodiesel thus obtained was further analyzed for fatty acid methyl esters (FAME) content. The previously used, vacuum-pump separated hydrated lime and without subsequent regeneration was added into the flask with fresh reactants and the methanolysis process was repeated under same conditions (up to 3 times).

Determination of FAME content was performed on a gas chromatograph 7890A with FID detector and automatic sampler 7683B, manufactured by Agilent Technologies, according to the standard SRPS EN 14103, April 2008, identical to EN 14103:2003. The aforementioned standard defines the determination of the ester content in fatty acid methyl esters intended for use as pure biofuel or as a component for mixing fuel oil and diesel fuel. Each obtained value of FAME content is from one experiment.

## RESULTS AND DISCUSSION

### Characterization of fresh commercial hydrated lime

The SEM images in Figure 2, as previously reported by Halilović et al., 2024 [26], show a heterogeneous morphology in the form of aggregates composed of irregularly arranged $Ca(OH)_2$ crystals of various sizes and shapes, resulting in an "open" structure of the material.

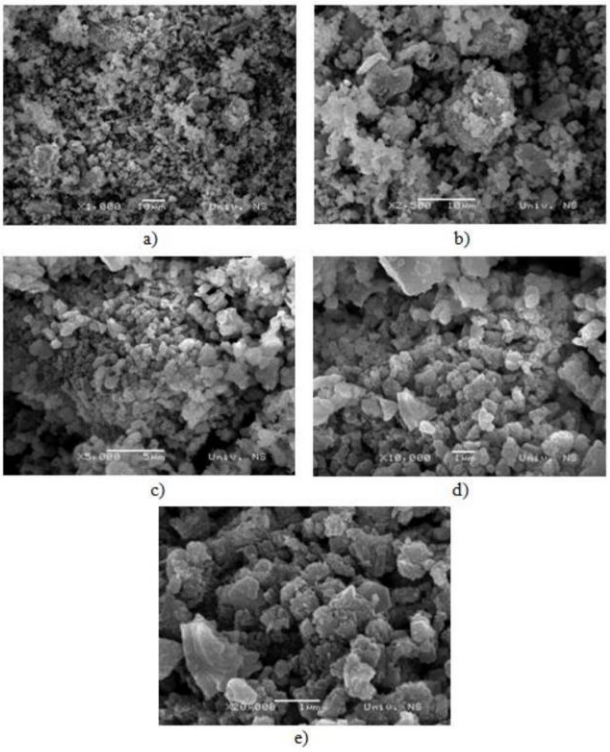

**Figure 2**. SEM images of fresh hydrated lime at magnifications of: a) 1000, b) 2500, c) 5000, d) 10000, and e) 20000.

Energy-dispersive X-ray spectroscopy (EDS), performed in combination with SEM, reveals the elemental composition (wt.%) of the hydrated lime sample and is shown in in Figure 3: 39.07% Ca, 47.68% O, 11.56% C, while Mg (0.51%), Al (0.39%), Si (0.56%) and Fe (0.24%) are present in amounts less than 1% [26]. The carbon content, which is somewhat higher in this sample compared to the carbon content in sample of commercial hydrated lime from different producer [28], may also indicate the presence of a carbonate phase ($CaCO_3$) in higher amount.

The diffractogram shown in Figure 4, as previously presented by Halilović et al., 2024 [26], displays two components of the catalyst: portlandite – $Ca(OH)_2$ (crystal structure database code: 1008781, reference code: 96-100-8782), with its two most intense peaks around 2θ values of 18° and 34°, and calcite – $CaCO_3$ (crystal structure database code: 9000968, reference code: 96-900-0969), with the most intense peak at a 2θ value between 29° and 30°. Since calcite was identified by this method, the previous assumption from the EDS results regarding its presence can be confirmed. According to the quantitative analysis from the XRD results, the sample contains 89.63 wt.% portlandite, corresponding to 67.83 wt.% CaO.

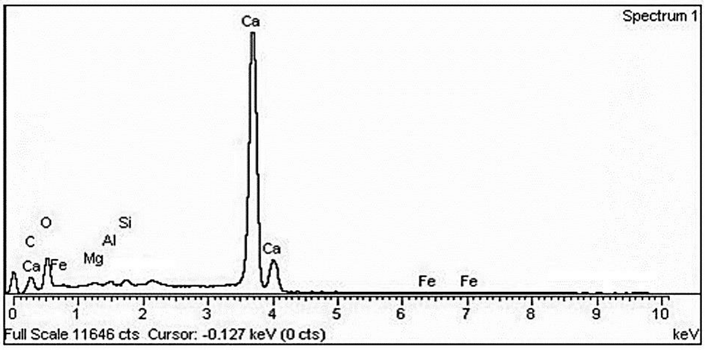

**Figure 3**. EDS spectrum of fresh hydrated lime

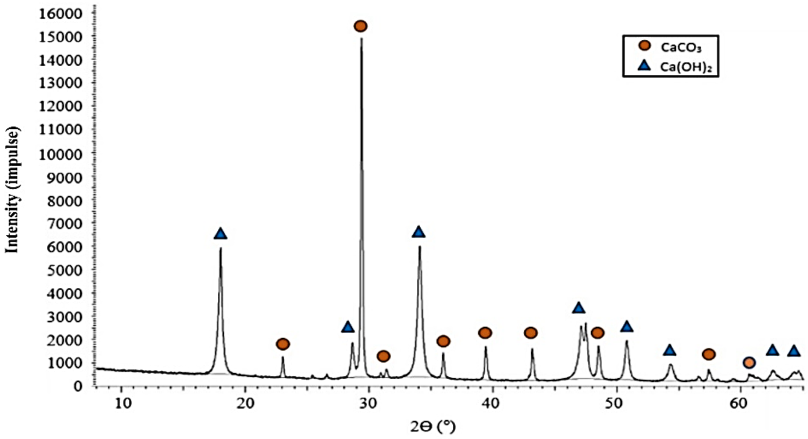

**Figure 4**. XRD pattern of fresh hydrated lime

## The impact of catalyst reuse on its properties

Figure 5 shows the comparative XRD patterns of fresh commercial hydrated lime sample and hydrated lime sample after its first use in methanolysis.

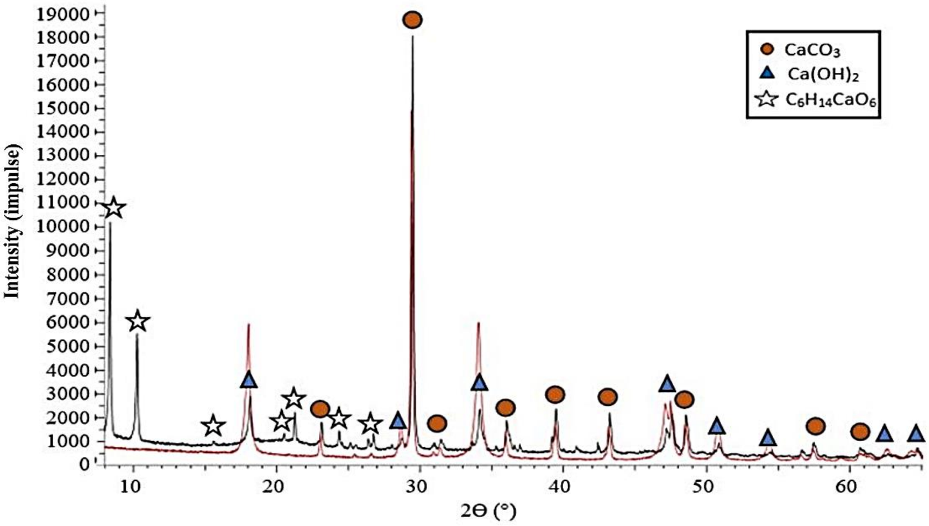

**Figure 5**. Comparative XRD patterns of hydrated lime samples: fresh commercial hydrated lime (red curve) and hydrated lime after first use in methanolysis (black curve)

The diffractogram of the hydrated lime sample, recorded after its first use as a catalyst, shows previously identified components present in fresh samples: portlandite – $Ca(OH)_2$, with the most intense peak around at 2θ value of 18°, and calcite – $CaCO_3$, with the most intense peak at a 2θ value between 29° and 30°. According to the XRD quantitative analysis results, the content of portlandite in the used hydrated lime is slightly lower compared to that in the fresh sample, amounting to 59.23 wt.%, but its presence indicates the possibility of reusing the used hydrated lime sample in the methanolysis of rapeseed oil. However, in the hydrated lime sample after its first use in methanolysis, an important change was detected. It is the presence of a new crystalline phase, calcium diglyceroxide – $C_6H_{14}CaO_6$, which was identified as the main crystalline phase, alongside $Ca(OH)_2$ and $CaCO_3$ as secondary crystalline phases. The formation of calcium diglyceroxide (Ca-DG) from calcium hydroxide was previously reported by Sanchez-Cantu et al. (2013) [29], who stated that two glycerol molecules first react with the surface of $Ca(OH)_2$, where oxygen from calcium hydroxide abstracts a proton from each glycerol molecule, which can then react with calcium ions to form calcium diglyceroxide and water. The authors also emphasized that when glycerol reacts with hydrated lime, Ca-DG is clearly identified as the main crystalline phase with only traces of $Ca(OH)_2$ and calcite.

Figure 6 shows comparative SEM images (at appropriate magnifications) of fresh commercial hydrated lime sample and hydrated lime sample after its first use as a catalyst, where only very slight morphological changes were observed, further indicating the possibility of its reuse in the methanolysis of rapeseed oil.

Figure 7 shows the XRD diagram of a commercial hydrated lime sample after its third reuse as a catalyst in the methanolysis of rapeseed oil.

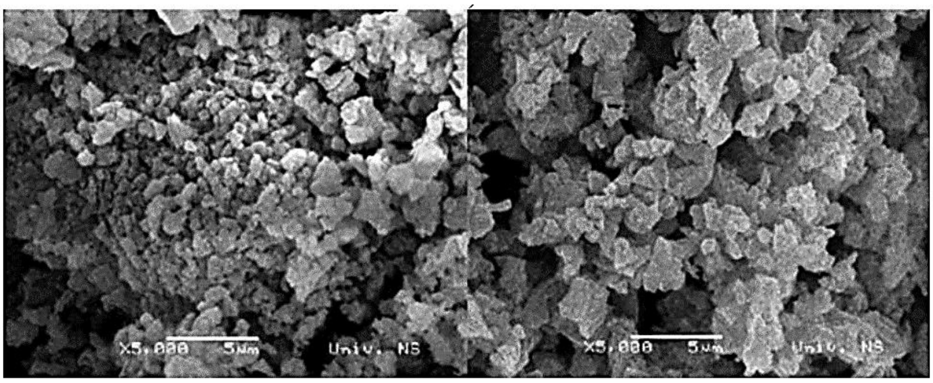

**Figure 6**. Comparative SEM images at corresponding magnifications of fresh commercial hydrated lime and hiydrated lime sample after its first use in methanolysis

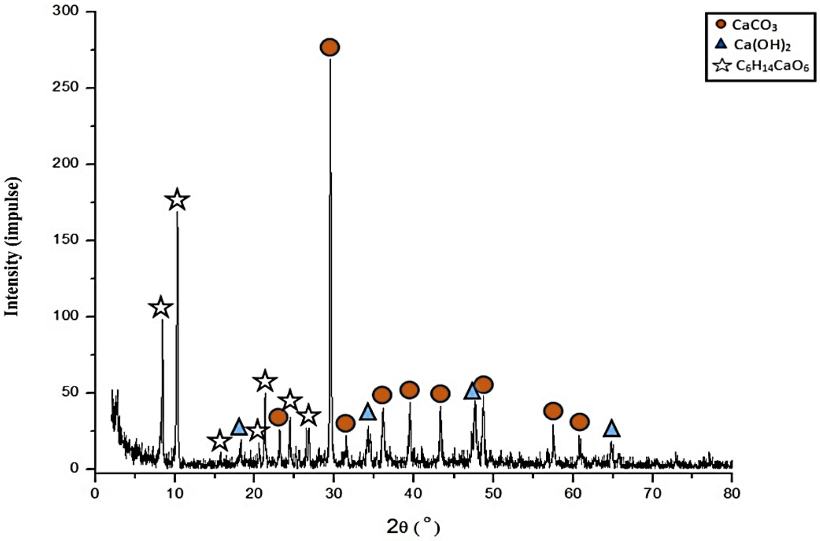

**Figure 7**. XRD diagram of the commercial hydrated lime sample after the third reuse in methanolysis

In the hydrated lime sample after the third reuse, the presence of crystalline phases of calcium diglyceroxide ($C_6H_{14}CaO_6$), calcium carbonate ($CaCO_3$) and calcium hydroxide ($Ca(OH)_2$) was detected. The sample is characterized by a higher content of calcium carbonate ($CaCO_3$), amounting to 28.2 wt.%.

### The impact of catalyst reuse on FAME content

Figure 8 shows the effect of successive use of commercial hydrated lime under optimal methanolysis conditions on the content of methyl esters of rapeseed oil.

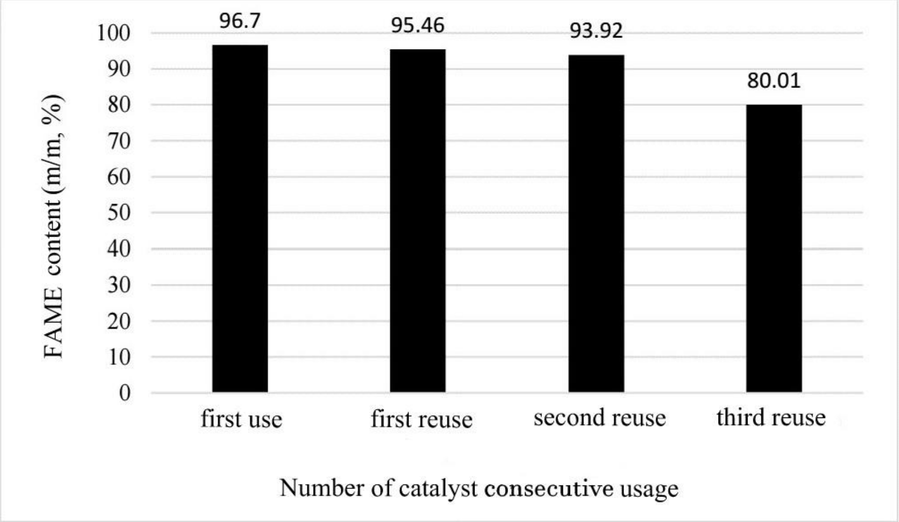

**Figure 8**. The impact of consecutive use of commercial hydrated lime as a catalyst on FAME content (catalyst concentration 4 wt.%, methanol:oil volume ratio 0.25, reaction temperature 60 °C, reaction time 120 min)

Based on Figure 8, it can be concluded that the FAME content after the first and second reuse exceeds 93%, while after the third reuse it decreases to 80.01%. Taking into account that the use of fresh commercial hydrated lime produced by Stamal Ltd. under optimal methanolysis conditions also resulted in a lower FAME content (96.7%) compared to commercial hydrated lime from another manufacturer (98.76%) used in the study by Halilović et al. (2021) [28], the obtained results indicate its lower catalytic activity.

The reduced activity of the commercial hydrated lime from Stamal Ltd. as a catalyst can be attributed to its lower portlandite content, detected in the fresh hydrated lime based on XRD analysis (89.63 wt.%) compared to the commercial hydrated lime from Ingram Ltd., which showed a portlandite content of 96.16 wt.% [28]. Additionally, after the third reuse of the Stamal hydrated lime, a higher $CaCO_3$ content was detected (28.2 wt.%). In the study on the efficiency of hydrated lime in the methanolysis of waste soybean oil by Sanchez-Cantu et al. (2011) [30], the reduced FAME content was primarily attributed to calcite, which was identified as a secondary phase in fresh lime but became the dominant crystalline phase after multiple uses of the catalyst, thereby reducing the catalytically active phase.

The FAME content values after the first, second, and third reuse of hydrated lime: 95.46%, 93.92% and 80.01%, respectively are lower compared to those obtained using fresh catalysts (96.7%), indicating a decline in catalytic activity. A clear correlation was observed between the number of catalyst reuses and FAME content, showing a decreasing trend in FAME content with an increasing number of reuses. A similar trend has also been reported with other heterogeneous catalysts [31], [32], which is attributed to catalyst deactivation. Possible causes of deactivation include leaching of the active phase, pore blockage, poisoning of surface active sites, and structural changes in the catalyst [33], [34], [17], [35].

In terms of the methyl ester content of biodiesel obtained through the reuse of commercial hydrated lime, the ester content does not meet the requirements of the European biodiesel specification EN 14214 [36]. However, obtained FAME content fall within the upper range of ester content (79–99%) typically achieved using heterogeneous catalysts [11].

## CONCLUSION

Commercial hydrated lime can be used successively as a catalyst in the methanolysis of rapeseed oil without the need for intermediate reactivation steps, which opens up the possibility for its application in a continuous biodiesel production process. After the first, second and third successive reuse of hydrated lime under optimal methanolysis conditions, FAME content above 80% was achieved. These values of ester content are high but the obtained values fall below the minimum threshold prescribed by the European biodiesel standard EN 14214 [36]. FAME content is high, but full standard testing according to EN 14214 [36] and American biodiesel standard ASTM D6751 for biodiesel fuel blends [37] was not performed in this study.

The reuse of hydrated lime results in reduced catalytic activity and a corresponding decrease in FAME content compared to that obtained using fresh catalyst. The degree of catalytic performance loss is correlated with the number of reuses of the catalyst, but its chemical, structural and textural properties must also be taken into consideration. Successive use of hydrated lime leads to the formation of a new phase, calcium diglyceroxide, which further contributes to the progression of the methanolysis reaction.

The content of pure calcium hydroxide and calcium carbonate as impurity in commercial hydrated lime is correlated with its catalytic ability in the transesterification reaction.

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