International Journal of Engineering Science Invention (IJESI)
ISSN (Online): 2319-6734, ISSN (Print): 2319-6726
www.ijesi.org ||Volume 11 Issue 9 September 2022 || PP 01-06

Influence of activation temperature of CaO catalyst on the efficiency of methanolysis of sunflower oil and the properties of the obtained biodiesel

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ABSTRACT:*In order to evaluate the influence of calcium oxide calcination temperature on the efficiency of transesterification for obtaining biodiesel, heterogeneous methanolysis of refined sunflower oil was performed using CaO catalyst which was previously thermally activated at temperatures of 500, 600 and 700 °C. Transesterification was carried out under conditions of temperature 60 °C, time 120 min, volume ratio methanol:oil of 0.25, amount of catalyst of 4 wt. % in relation to the mass of oil and mixing speed of 500 rpm. The analysis of the obtained biodiesel samples was carried out in terms of determining their physical and chemical characteristics defined by the EN 14214 standard, including the content of total fatty acid methyl esters, linolenic acid methyl ester content, density, viscosity, acid value, iodine value and flash point. Through a comparative analysis of the results, it was determined that the highest yield of fatty acid methyl esters (95.75 wt. %) was achieved using CaO pre-calcined at 600 °C, and the main characteristics of the obtained biodiesel (density, viscosity, flash point) were in accordance with values specified in the valid European standard for biodiesel.*

KEYWORDS -*Biodiesel, calcination, calcium oxide, heterogeneous catalyst, methanolysis*

Date of Submission: 01-09-2022 Date of Acceptance: 13-09-2022

I. INTRODUCTION

The growth of the human population and industrialization inevitably lead to an increase in energy requirements, especially in the transport sector [1]. Currently, fossil fuels are the most important source of energy in the world [2], but due to their non-renewability and harmful impact on the environment [3], attention has been focused on biofuels in recent decades [4]. One of them is biodiesel, which is recognized as a renewable, environmentally friendly and biodegradable fuel [5], the combustion of which can minimize gaseous pollutants such as CO, SO_2 and hydrocarbons, which are produced by the incomplete combustion of fossil fuels [6]. In addition, certain modifications to diesel engines running on biodiesel-diesel blends can make the engine even more efficient and reduce its NOx emissions. [7].

Biodiesel is defined as monoalkyl esters of vegetable oils or animal fats [8]. Triglyceride raw materials for biodiesel production can be: edible feedstocks, agricultural by-products or cellulosic materials, aquatic cultivated feedstock, i.e., algae, and bioengineered microorganisms [9],but edible oils such as rapeseed, sunflower, soybean and palm oil are mainly used in commercial production [10]. A common biodiesel synthesis procedure is the transesterification of triglycerides with methanol (also called methanolysis), which yields fatty acid methyl esters (FAME) and glycerol as a by-product [11]. Different types of catalysts such as acid and base catalysts, which are either homogeneous or heterogeneous, as well as enzymes, can catalyze the transesterification reaction [12]. Traditionally used transesterification catalysts for obtaining biodiesel are homogeneous alkaline, such as: NaOH, KOH, $NaOCH_3$ and $KOCH_3$[13]. However, due to the disadvantages of homogeneous catalysts, related to soap formation and difficulty in product separation, heterogeneous catalysts are preferred [14]. Calcium oxide (CaO), magnesium oxide (MgO), strontium oxide (SrO) and hydrotalcite ($Mg_6Al_2(OH)_{16}CO_3.4(H_2O)$) are commonly listed among the heterogeneous alkaline catalysts [15], and the efficiency of hydrated lime has also been pointed out[16][17]. Among the aforementioned catalysts, CaO is particularly used [18], whose catalytic activity can be improved if it is subjected to an activation treatment before the transesterification reaction [19].

In this paper, the influence of the temperature of thermal activation of calcium oxide on its catalytic efficiency in the methanolysis of refined sunflower oil, for the production of biodiesel was investigated.

II. EXPERIMENTAL PART

2.1. Materials

In the experimental part of this work, the following materials and chemicals were used: refined sunflower oil from the domestic producer Bimal (Brčko, Bosnia and Herzegovina), whose limit values of quality parameters are given in TableI, reagent grade calcium oxide (CaO), purchased from Sigma-Aldrich and methanol p.a. purchased from Fluka.

Table I: Physicochemical characteristics of refined sunflower oil [16]

Physicochemical characteristics	***Limit values***	***Methods***
Relative density at 20 °C	0,918-0,923 kg/dm^3	ISO 6883
Refractive index (ND 40 °C)	1,461-1,468	ISO 6320:2000
Free fatty acids (as oleic)	max. 0,2 %	ISO 660:1996
Moisture and volatile matter mass fraction	max. 0,2 %	ISO 662:1998
Iodine value	118-141	ISO 3961:2001
Saponification value	188-194	ISO 3657:1998
Peroxide value	max. 10 meqO_2/kg	ISO 3960
Soap content (as Na-oleate)	max. 50 mg/kg	DGF C-III 15 (97)
Unsaponifiable matter	max. 15 g/kg	ISO 18609:2003
Fatty acid composition		
C 14:0 C 16:0 C 16:1 C 18:0 C 18:1 C 18:2 C 18:3 C 20:0 C 20:1 C 22:0 C 22:1 C 24:0	0,0-0,2 w/w % 5,0-7,6 w/w % 0,0-0,3 w/w % 2,7-6,5 w/w % 14,0-39,4 w/w % 48,3-74,0 w/w % 0,0-0,3 w/w % 0,1-0,5 w/w % 0,0-0,3 w/w % 0,3-1,5 w/w % 0,0-0,3 w/w % 0,0-0,5 w/w %	ISO 5508:1990 ISO 5509:1990

2.2. Methods

2.2.1. Preparation of the catalyst

Calcium oxide was prepared by calcining in a laboratory furnace at temperatures of 500, 600 and 700 °C for 4 hours. After calcination, the CaO was transferred to a desiccator to cool to room temperature, then ground with an electric mill and sieved through a sieve to obtain catalyst particles up to 400 µm in size.

2.2.2. Transesterification of sunflower oil

Heterogeneous catalyzed methanolysis was carried out under laboratory conditions using an apparatus that included: a 500 ml three-necked round-bottomed flask, a heater with a thermostat, a water cooler, a mechanical stirrer and a thermometer. Transesterification procedure of the reaction mixture of 500 ml of refined oil (as received) and 125 ml of methanol p.a., with the presence of 4 wt. % of calcined CaO (in relation to the mass of oil), was performed at atmospheric pressure, temperature 60 °C and time 120 min, with constant mixing at 500 rpm.

After the specified time, the mixture was transferred to a separation funnel, in order to separate the glycerine phase from the phase of fatty acid methyl esters, i.e. of biodiesel which then was subjected to characterization.

2.2.3. Characterization of FAME

The analysis of the obtained biodiesel samples was carried out in terms of determining their basic physical and chemical characteristics, which are defined by the EN 14214 standard [20], including the total content of methyl esters, the linolenic acid methyl ester content, density, viscosity, acid number, iodine value and flash point.

The total content of methyl esters and the content of linolenic acid methyl ester of sunflower oilwere determined by the methodEN 14103 [21], using an Agilent 7890 gas chromatograph.

The density of biodiesel was measured with a densitometer DMA 35N manufactured by Anton Paar, by preheating the sample to 15 °C for 30 minutes.

The viscosity of biodiesel was determined using a capillary viscometer (Oswald's viscometer), with prior tempering at 40 °C for 30 minutes.

The acid number was determined by titration with 0.1 M KOH, the iodine value by the Wijs method [22], and the flash point was determined by the ASTM D-93 method[23].

III. RESULTS AND DISCUSION

Fig. 1. gives a graphic representation of the influence of the temperature of thermal treatment of CaO on the content of methyl ester in the obtained biodiesel, where there are evident differences in the content of MEMK depending on the calcination temperature of calcium oxide. The above can be explained by the

deactivation of CaO in contact with carbon dioxide and moisture from the air, where CO_2 is the main deactivating agent [24]. Namely, it is stated that exposure of CaO to room temperature air leads to its rapid hydration and carbonation [25], but reactivation of the catalyst can be achieved by calcining it in a furnace at high temperatures [26]. After the process of activation of CaO by calcination, a significant increase in the activity of the catalyst in the production of biodiesel was achieved [27]. When pre-calcined quicklime was used as catalyst, the maximum biodiesel production rate was achieved at a methanol:oil molar ratio of 6:1 as opposed to the 12:1 ratio which was required with usage of reagent grade CaO [28]. The results obtained by [29] showed that lime calcination temperatures affected the biodiesel yield, that is, that a high calcination temperature gives a better biodiesel yield. The results obtained in this paper show that the highest content of methyl esters (95.75 wt. %) was obtained using CaO as a catalyst that was previously calcined at 600 °C, while CaO calcined at 500 °C gave a yield of methyl esters of 74.04 wt. %, and that which was calcined at 700 °C yield MEMK of 88.78 wt. %. The calcination temperature affects the amount of active sites, i.e. the basic properties of alkaline earth oxides [30], and basic strength of the catalysts have been mainly related to their activity [31]. The obtained resultsare consistent with those of [24] and [32] who came to the conclusion that the maximum yield of MEMK is obtained by performing calcination at 550 °C and at a duration of the transesterification reaction of 120 min. On the other hand, [33] achieved a methyl ester yield of 100 wt % at reaction conditions of 60 °C, 4 wt. % of catalyst and 17 vol. % of methanol by transesterification of soybean oil with CaO as a catalyst calcined at 500 °C, which can be attributed to the fact that the authors used a different type of oil, as well as different process parameters.

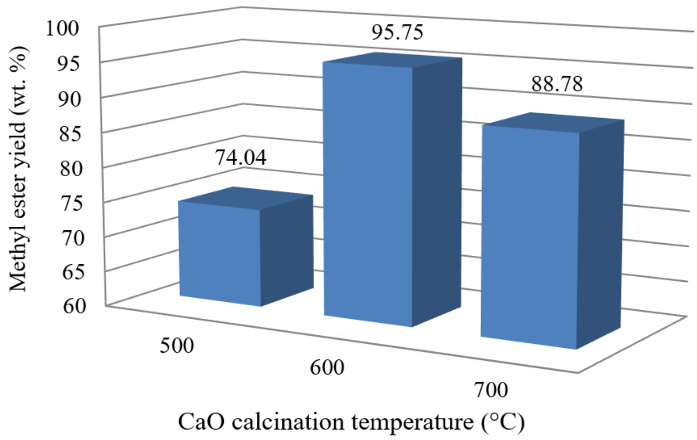

Figure 1: Effect of CaO calcination temperature onsunflower oil methyl ester yield

Fig. 2. shows the effects of calcium oxide calcination temperature on the density and viscosity of the obtained biodiesel. The mentioned properties of biodiesel are considered very important, because their unacceptable values can have a harmful effect on the performance of the engine and the lifetime of the engine system [34][35][36][37].

The determined range of densities of biodiesel samples obtained using CaO catalyst pre-calcined at temperatures of 500, 600 and 700 °C is from 0.879 to 0.88 g/ml and is in accordance with the requirement of the current European standard for biodiesel, EN 14214 [20], which prescribes a range 0.86 - 0.9 g/ml. Additionally, the obtained biodiesel densities are in the range of those obtained by other researchers by heterogeneous methanolysis of sunflower oil [38][39].

The determined viscosities of biodiesel samples obtained by methanolysis in the presence of CaO calcined at temperatures of 500, 600 and 700 °C are in the range 4.2 to 4.56 mm^2/s, which is also within the range of values prescribed by the mentioned standard (3.5 - 5.0 mm^2/s). The viscosity of biodiesel is considered a particularly important property because its high values lead to poorer fuel dispersion [40]. The results of this research show that using the CaO catalyst, previously activated at 600 °C, the lowest value of biodiesel viscosity was obtained and at the same time the highest MEMK yield was achieved, which is in accordance with the observations of other researchers that the viscosity of the produced biodiesel decreases with an increase in the yield of the transesterification reaction [41][42].

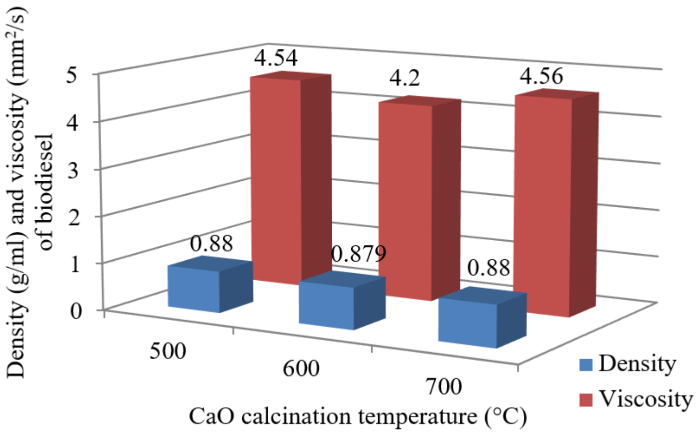

Figure 2:Effect of CaO calcination temperature onsunflower oil methyl ester yield

Table II shows the results of the analysis of other properties of biodiesel obtained by methanolysis of sunflower oil in the presence of 4 mass% CaO catalyst that was previously calcined at different temperatures.

Linolenic acid is one of the common fatty acids that exist in the triglyceride molecule [43], and the content of its methyl esters determined in biodiesel obtained using CaO calcined at temperatures of 500, 600 and 700 °C corresponds to the content commonly seen in sunflower oil biodiesel [44] and complies with the requirements of the EN 14214 standard for biodiesel [20].

The acid value (A_v) and flash point (F_p) of the obtained biodiesel samples are in accordance with those in the mentioned European standard for biodiesel, while the iodine value (I_v) is slightly higher. It is stated that, although high iodine values are associated with higher linolenic acid content in biodiesel raw materials, high I_v are not necessarily based on high linolenic acid methyl ester content [45], which is confirmed by their values determined in this paper. Considering that the iodine number of methyl esters is almost identical to that of the triacylglycerols from which they are derived [46], the higher I_v of biodiesel is a consequence of relatively high limit values of the iodine number of refined sunflower oil from which the methyl esters were obtained.

TableII. Properties of biodiesel obtained using calcium oxide catalyst previouslu activated at differentcalcination temperatures

Biodiesel properties	***CaO calcination temperature (°C)***			***EN 14214***
	500	*600*	*700*	
Content of linolenic acid methyl esters (wt %)	0.122	0,121	0,117	max. 12,0
Acid value, A_v (mg KOH/g)	0.12	0.05	0,03	max. 0,5
Iodine value, I_v (g/100 g)	135,5	135,58	132	max. 120
Flash point,F_p (°C)	179	164	172	min. 101

IV. CONCLUSION

Reagent grade calcium oxide was calcined at temperatures of 500, 600 and 700 °C and used as a heterogeneous catalyst for the methanolysis of refined sunflower oil. By analyzing the physical and chemical characteristics of the obtained biodiesel samples, the optimal temperature of the thermal activation of CaO was estimated. It was concluded that an increase in the calcination temperature of calcium oxide to a certain value leads to an increase in its catalytic activity, where by using a CaO catalyst calcined at 600 °C it is possible to achieve a yield of methyl esters of fatty acids greater than 95 wt %, i.e. about 22 % greater than that obtained by CaO calcined at 500 °C and about 7% higher than that obtained by CaO calcined at 700 °C. By methanolysis of sunflower oil with calcium oxide previously activated at 600 °C, the lowest values of density, viscosity and flash point of biodiesel were achieved compared to those obtained using CaO activated at 500 and 700 °C, which are within the values prescribed by the European standard for biodiesel.

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Amir Fazlić, et. al. "Influence of activation temperature of CaO catalyst on the efficiency of methanolysis of sunflower oil and the properties of the obtained biodiesel." *International Journal of Engineering Science Invention (IJESI)*, Vol. 11(09), 2022, PP 01-06. Journal DOI- 10.35629/6734