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Title: Bifunctional catalysts applied to produce biodiesel from waste cooking oil

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Introduction

Current energy sector depends mainly on the use of fossil fuels (Hafeez et al., 2020).

Figure 1. In the image from DNyuz we can see air pollution produced from fossil fuels.
Source: DNyuz

At least 60% of the total energy used in the world is obtained from fossil fuels use. (Mansir et al., 2018a)

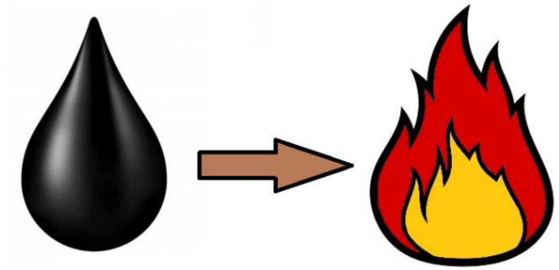


Figure 2. Use of fossil fuels
Source: author´s own creation

25% of CO_2 emissions correspond to the use of fossil fuel for transportation (Hafeez et al., 2020; Llanes Cedeño, 2017).

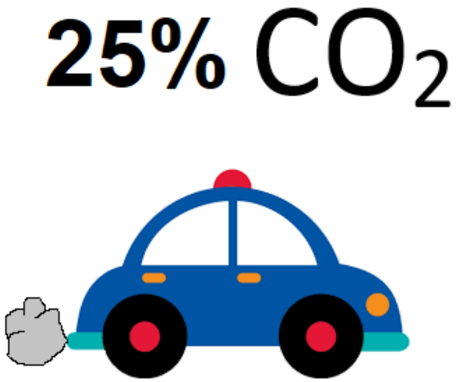


Figure 3. CO_2 emissions for transportation
Source: author´s own creation

Biodiesel has become a viable alternative fuel, mainly due to its physicochemical characteristics. It is possible to obtain it from vegetable oils (refined, edible or reused), as well as from animal fats. (Bhavani & Sharma, 2018)

Various studies have been oriented to the search for lipid raw materials that do not have a nutritional value, as the waste cooking oil. (Khodadadi et al., 2020)

Figure 4. Biodiesel
Source: author´s own creation

Figure 5. Waste cooking oil
Source: author´s own creation

Bifunctional catalysis

- Bifunctional catalysts are those that have two types of active sites.
- This allows them to carry out two processes simultaneously.
- Less soap production.
- High tolerance to free fatty acid (FFA) content.

In the synthesis of biodiesel, these catalysts can work with unrefined lipid raw materials, because they are capable of esterifying the FFAs and transesterifying the triglycerides present. (Foldvari, 2011)

Figure 6. Bifunctional catalyst.
Source: author´s own creation

Methodology

Raw material

- Recollection
- Purification.
- Characterization

Catalyst

- Cleaning/Grinding
- Impregnation of Fe
- Thermal treatment
- Catalytic characterization

Catalytic evaluation

- Transesterification of WCO.
- Reaction conditions:
  - Methanol:oil ratio 12:1
  - Catalyst weight 6%,
  - Temperature 55°C
  - Reaction time 5h.

Biodiesel characterization

- Determination of %FAMEs content
- Acid value
- Viscosity

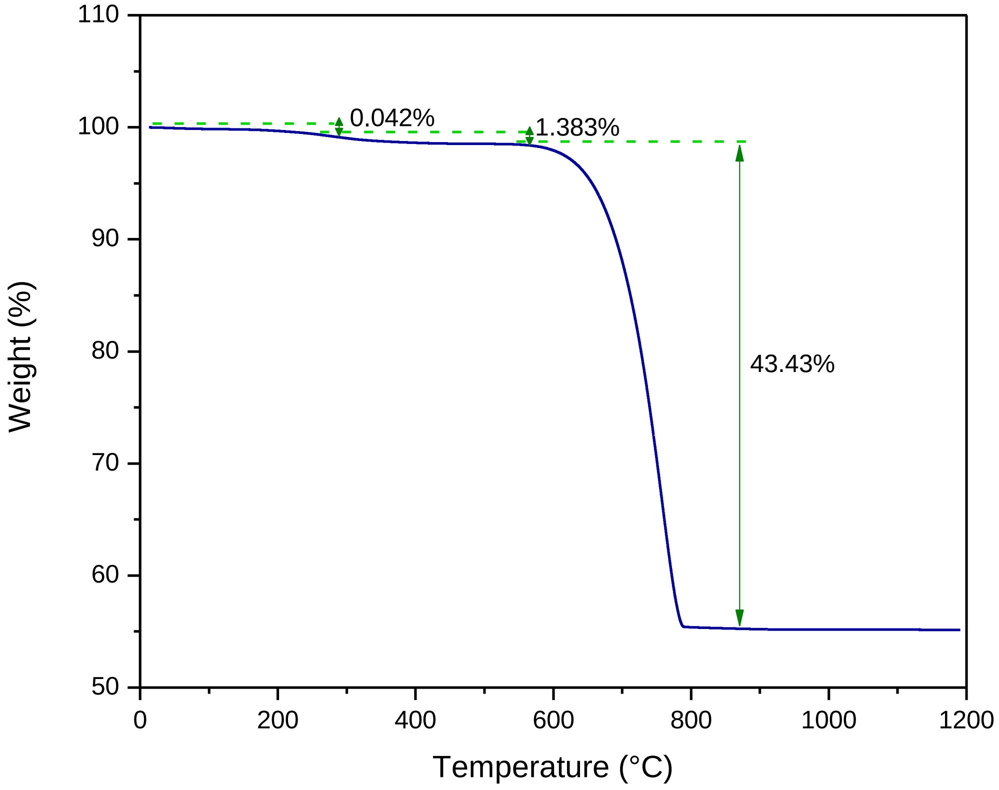
Results

Thermogravimetric analysis (TGA) and differential scanning calorimetry (DSC)

In order to establish an adequate calcination temperature, the clam shell was analyzed using TGA analysis. The results obtained, are shown in Graph 1.

It is possible to clearly observe three significant weight losses:

1st. loss: 100-180°C
2nd. loss: 250-550°C
3rd. loss: 550-850°C



Graph 1. Results of the TGA-DSC performed on the clam shell

Source: author´s own creation

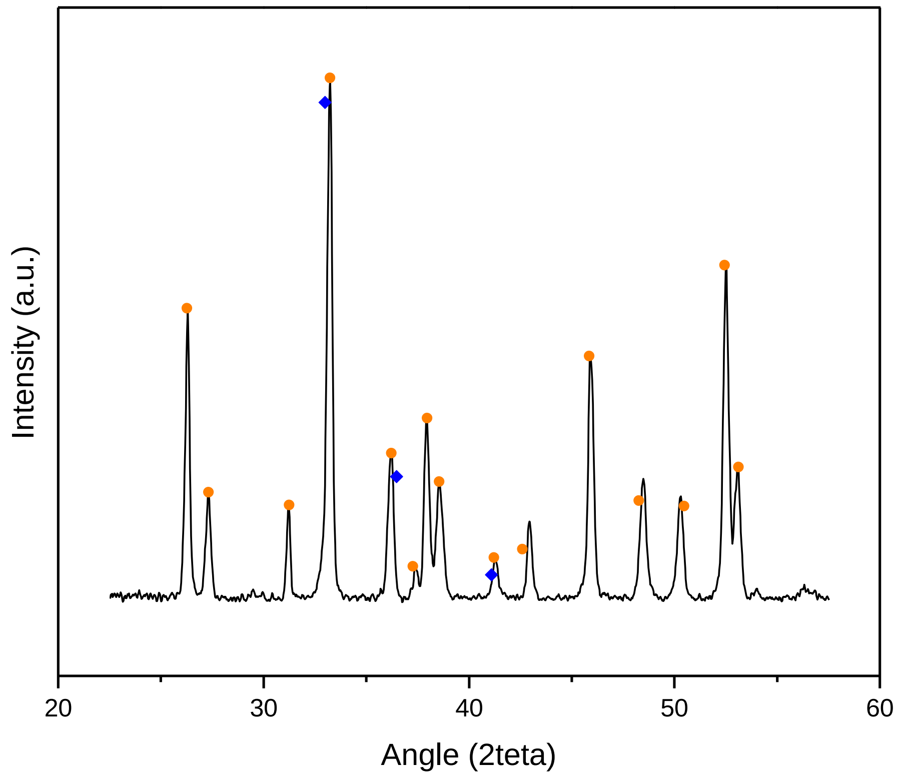
X-ray diffraction (XRD)

In order to establish the crystalline phases in the samples, the X-ray diffraction technique was applied.

- Uncalcined clam shell

The main component $CaCO_3$ was identified in the crystalline phases:

- Aragonite (JCPDS #760606)
- Calcite (JCPDS #240027).



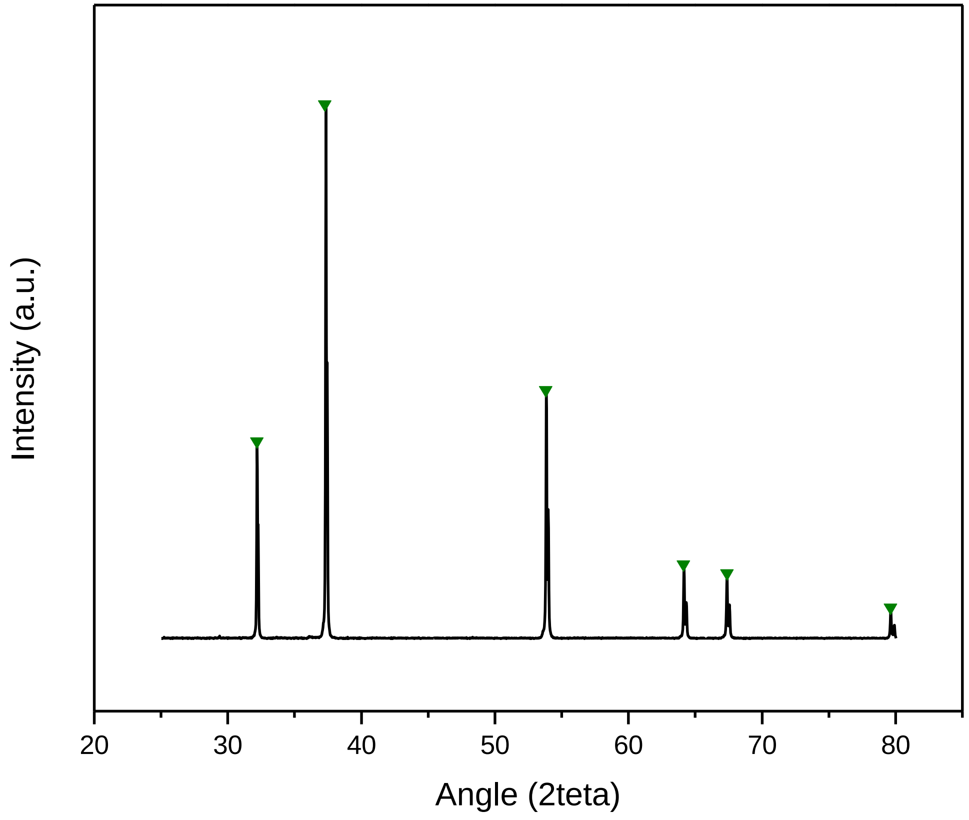
Graph 2. Uncalcined clam shell diffractogram

Source: author´s own creation

X-ray diffraction (XRD)

- Calcined clam shell for 6 h at 900°C

The peaks that are specific pattern of ▼ CaO were observed at 2θ angles: 32.2°, 37.5°, 53.8° 64.1°, 67.3° and 79.6° (JCPDS #371497).



Graph 3. Calcined clam shell diffractogram

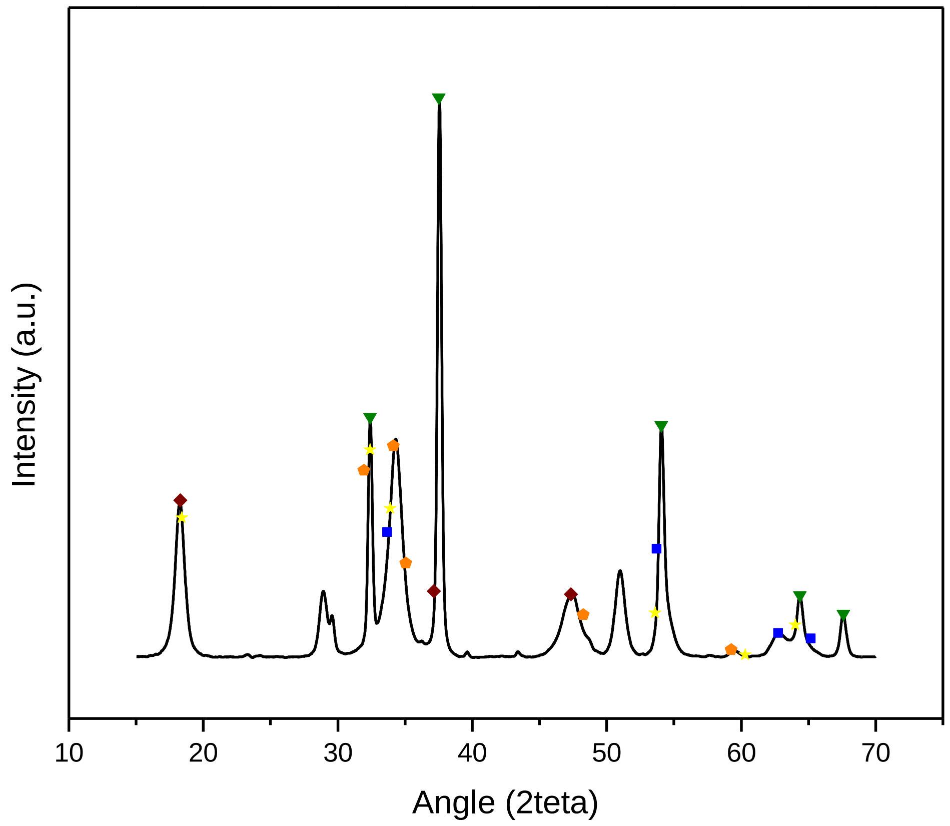
Source: author´s own creation

X-Ray Diffraction (XRD)

- Catalyst Fe_2O_3/CaO

Through X-ray diffraction analysis the following phases were identified:

▼ CaO (JCPDS #371497)
◆ Fe_2O_3 (JCPDS #330664)
■ Fe_3O_4 (JCPDS #890688)
⬟ $Ca_2Fe_2O_5$ (JCPDS #712264)
☆ $Ca_2Fe_{15.57}O_{25.56}$ (JCPDS #722346)

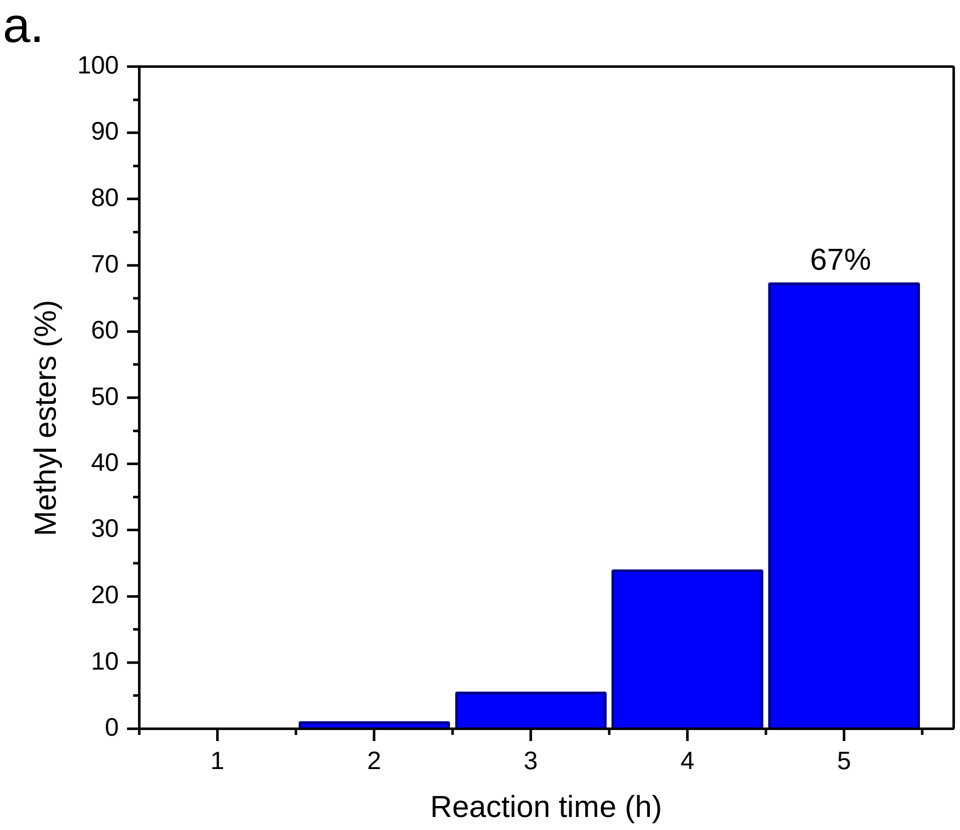


Graph 4. Catalyst Fe_2O_3/CaO diffractogram

Source: author´s own creation

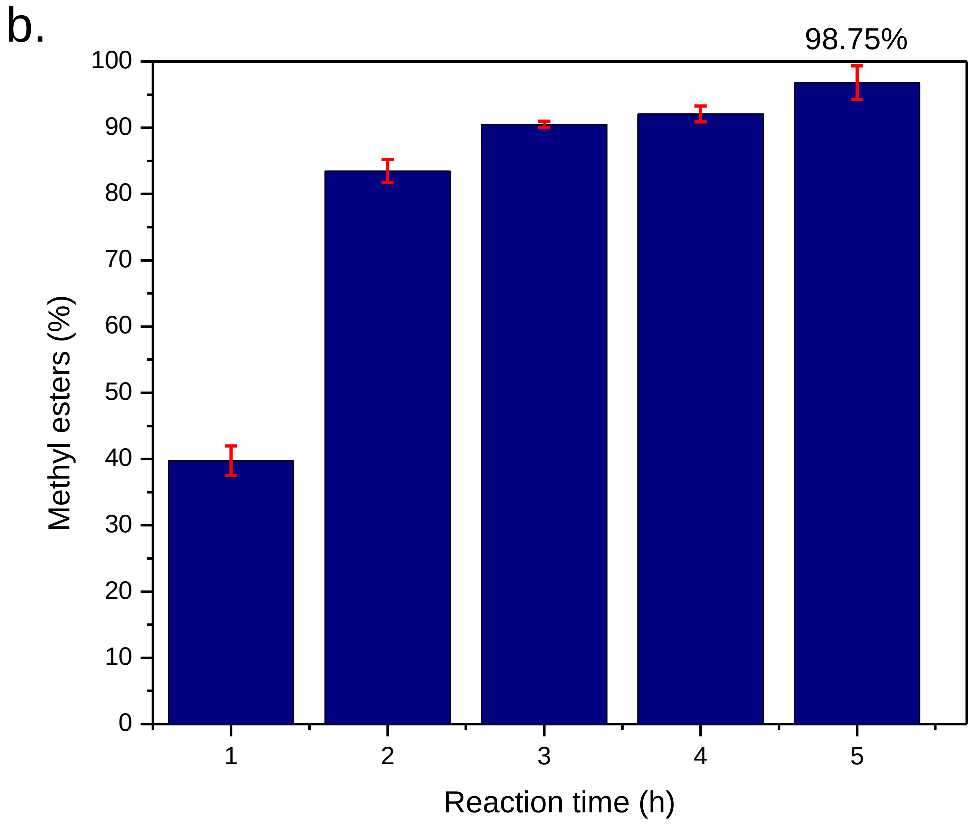
Catalytic evaluation

a. Influence of the type of catalyst with waste cooking oil



Graph 5. WCO transesterification catalyzed with CaO.

Source: author´s own creation



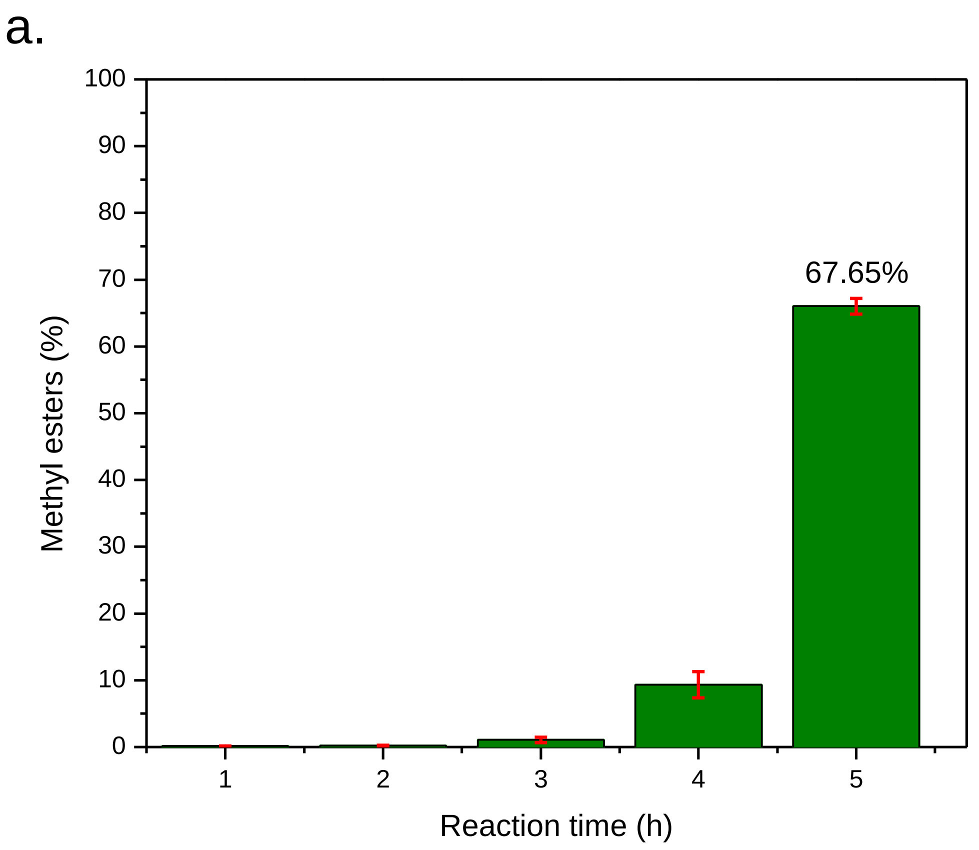
Graph 6. WCO transesterification catalyzed with Fe_2O_3/CaO

Source: author´s own creation

Reaction conditions: T= 55°C, W_{cat}= 6%, Vol_{rxn}=135ml, methanol:oil molar ratio=12:1

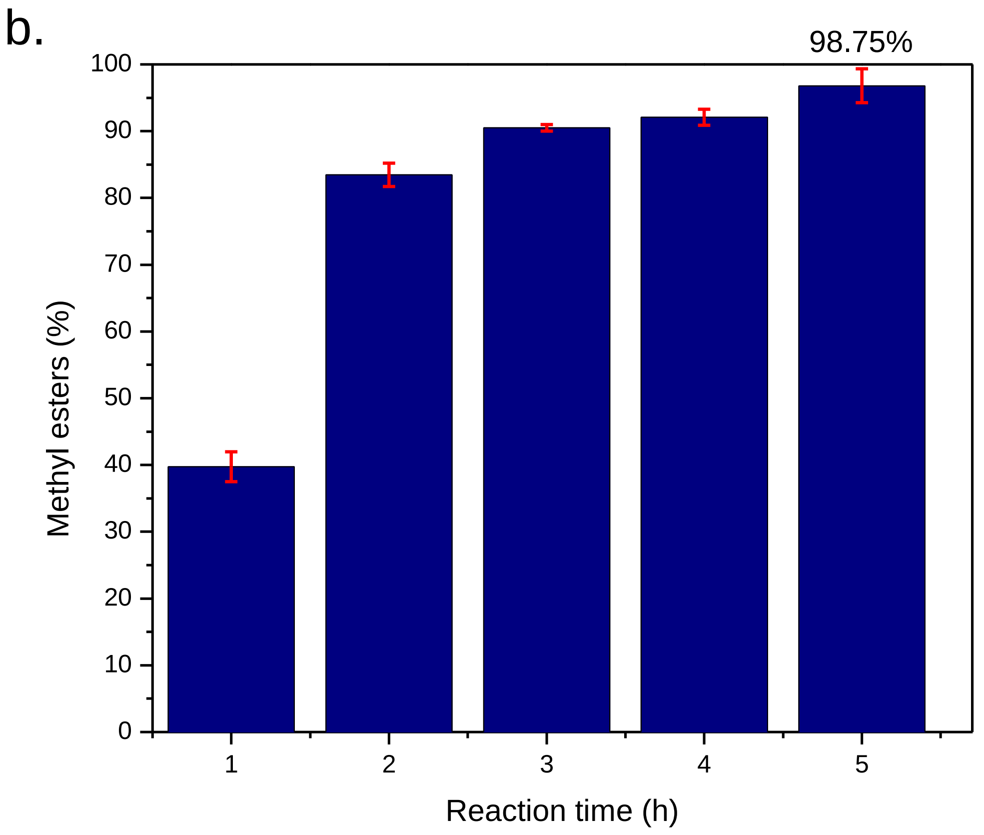
Catalytic evaluation

b. Effect of the amount of iron salt on the bifunctional catalyst (Fe_2O_3/CaO)



Graph 7. WCO reaction with catalyst 5% by weight of $Fe(NO_3)_3$

Source: author´s own creation



Graph 8. WCO reaction with catalyst 7% by weight of $Fe(NO_3)_3$

Source: author´s own creation

Reaction conditions: T= 55°C, W_{cat}= 6%, Vol_{rxn}=135ml, methanol:oil molar ratio=12:1

Biodiesel Characterization

To characterize the biodiesel obtained from the previous transesterification reactions, the content of fatty acid methyl esters (FAMEs), viscosity and acid value was determined by means of the standard methods shown in UNE-EN 14103, ISO 3104 and UNE-EN 14104.

The obtained results are shown in Table 1:

Table 1. Characterization of the biodiesel obtained with the bifunctional catalyst

Raw material	Methyl esters content (%)	Viscosity (mm^2/s)	Acid value (mgKOH/g)
Waste cooking oil	98.8	4.6	0.052
Refined oil	100.0	4.8	0.05
EN 14214	96.5	3.5 – 5.0	Máx. 0.50

Source: author´s own creation

Conclusions

- A bifunctional catalyst, Fe_2O_3/CaO, was synthesized. This catalyst has basic active sites (provided by CaO) and acid sites (provided by iron). This allows to process raw materials with higher content of free fatty acids than those present in refined oils.

- Biodiesel was obtained through a simultaneous reaction of esterification and transesterification, using 6% by weight of catalyst, a content of 1% by weight of iron, methanol: oil molar ratio of 12:1, with a reaction time of 5h, obtaining a content of methyl esters higher than 98%.

Conclusions

- It was determined that 7% by weight of iron precursor salt in the synthesis of the catalyst is the optimal amount of precursor salt to obtain biodiesel that complies with EN-14214 regulations.

- The development of efficient heterogeneous catalysts in the use of raw materials with a higher FFA content represents a significant area of opportunity in reducing the cost of biodiesel production. However, the efficiency of a heterogeneous catalyst depends on several variables, such as the type of raw material, the type of alcohol used, the molar ratio of alcohol:oil, reaction temperature, stirring speed, type of reactor, and type of catalyst used.

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