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Biodiesel production by transesterification using choline hydroxide as catalyst

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INTRODUCTION & AIM

Biodiesel is a mixture of alkyl esters derived from vegetable oils or animal fat. It is a biodegradable and renewable fuel and does not contribute to the emission of polluting gases into the atmosphere. Still, its major disadvantage is its relatively high cost, usually regarding production and raw materials costs (Lang *et al.*, 2001). Crude oils and fats extracted from plants and animals are alternatives to highly polluting fossil fuels. They are composed of triglycerides, which can be converted into biofuels through transesterification processes. The alcoholysis of vegetable oils or animal fats may be carried out using different catalysts: acids, bases, and enzymes. Basic catalysts, sodium or potassium hydroxide, are about 4,000 times faster than acid catalysts and do not require large amounts of alcohol. On the other hand, ionic liquids (ILs) use in catalytic processes has been studied mainly in the ecological field, as it allows a high recycling efficiency. ILs based on the choline cation (2-hydroxyethyl trimethylammonium) have received much attention, mainly due to its biocompatibility characteristics and potential for various industrial applications. Choline hydroxide (ChOH – see Fig. 1) in particular represents a promising option due to its good catalytic performance in methanol solution and its successful reuse (Fan *et al.*, 2013). This work's main objective was to optimize the methyl transesterification reaction conditions using commercial and waste sunflower oil as raw material and ChOH as catalyst. The possibility of recovering the ChOH catalyst between reaction cycles was also assessed.

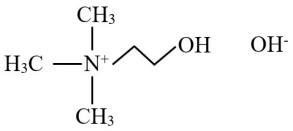

Figure 1. ChOH chemical structure

METHOD

- **Biodiesel production**

1 – reactor, 2 – paraffin bath, 3 – heating plate with automatic temperature control and magnetic stirring, 4 – reflux condenser, 5 – thermometer, 6 – temperature sensor

A – light phase (biodiesel)
B – heavy phase (glycerol)

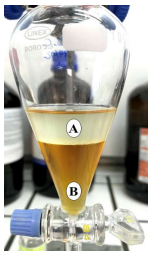

Figure 2. Biodiesel production experimental apparatus

Figure 3. Phase separation

- **Raw-materials characterization and reaction conversion assessment**

- **Acidity value** (AV) determined according to a procedure based on the European Standard EN 14104/2003.

$$AV\left(\frac{mg_{KOH}}{g_{sample}}\right) = \frac{V_{KOH} \times C_{KOH} \times MM_{KOH}}{m_{sample}}$$

- **Biodiesel FAMEs content** determined by **GC-FID**, through a procedure in accordance with the European Standard EN 14103/2003, using methyl heptadecanoate as internal standard (IS).
- Reaction conversions assessed through biodiesel FAMEs content.

$$X\,(\%) = \frac{(\sum A_{FAMEs} - A_{IS})}{A_{IS}} \times \frac{C_{IS} \times V_{IS}}{m_{sample}} \times 100$$

- **Catalyst recovery**

- **IL recovery** tests carried out by washing glycerol phase with binary system butanol (BuOH):water 1:1 (v/v), on several sample/solvent mass ratios.
- Separation assessed by **FTIR** analysis.

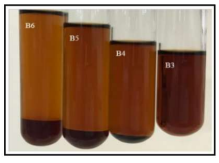

Figure 4. IL recovery tests

RESULTS & DISCUSSION

- **Biodiesel production: T = 65 °C**

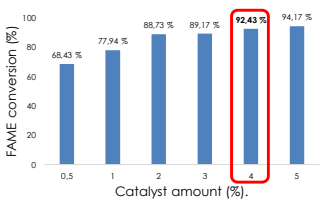

Figure 5. Effect of catalyst amount

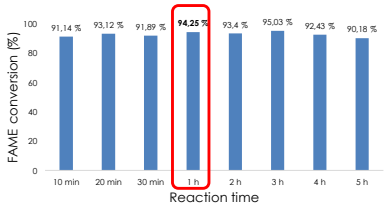

Figure 6. Effect of reaction time

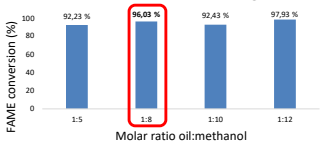

Figure 7. Effect of oil:methanol molar ratio

Table 1. Best operational conditions

Reference	Oil Acidity (mgKOH/g)	Temp. (°C)	Reaction Time (h)	Molar ratio oil:methanol	% catalyst	FAME conversion (%)
Fan et al. (2013)	± 0.3	60	2.5	1:9	4	95.0
Bessa (2015)	± 0.5	60	3	1:15	4	99.1
In this work	± 0.2	65	1	1:8	4	96.1

- **Catalyst recovery: BX test – sample:BuOH/H_2O on 1:X mass ratio**

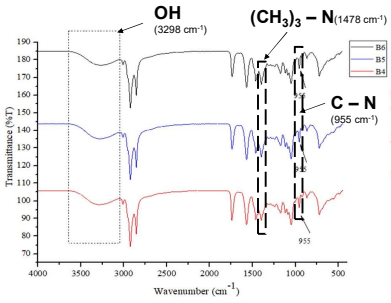

Figure 8. Light phase FTIR (solvent+ChOH)

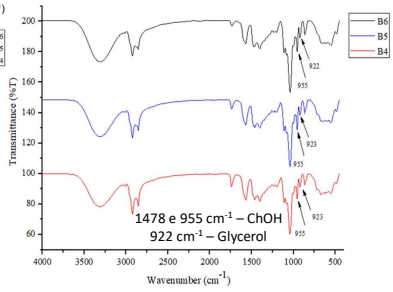

Figure 9. Heavy phase FTIR (glycerol+ChOH)

CONCLUSION

- Best conversion values were obtained using 4%wt. catalyst load, oil/methanol molar ratio of 1:8, and reaction time of 1 h, for the specified temperature of 65°C.
- Reaction products AVs for residual sunflower oil showed a significant reduction in relation to the raw material (AV – 6.14 mg KOH/g).
- Biodiesel phase AVs remained low for the reactions with the commercial sunflower oil samples (AV close to 0.20 mg KOH/g).
- Post-reaction ChOH recovery studies, performed with BuOH/water systems, proved inefficient under the conditions tested; FTIR analysis showed the presence of ChOH in both liquid-liquid extraction phases.

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