p-Sulfonic acid calix[4]arene as a new reusable organocatalyst for the transesterification of vegetable oil

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INTRODUCTION

The transesterification of vegetable oils in methanol is the most common method for the production of biodiesel. The reaction is usually performed under basic homogeneous catalysis, using sodium/potassium hydroxide or alkoxides.

There is currently a great demand for organic catalysts as they are less toxic than the metal catalysts, easy available and insensitive to oxygen.[1]

The sulfonic acid calix[4]arene is a macrocyclic compound obtained via the condensation of *p*-substituted phenols with formaldehyde. It has been reported as a reusable organic catalyst for the esterification of carboxylic acids in ethanol with high conversion yields. [2]

Due to these results, the aim of the present work is to investigate the use of *p*-sulfonic acid calix[4]arene as a reusable organocatalyst for the transesterification of passion fruit oil.

RESULTS AND DISCUSSION

The influence of the transesterification parameters (Figure 1) was evaluated: heating mode (conventional, microwave or hydrothermal), determination of the minimal concentration of the catalyst), alcohol used (methanol or ethanol).

The conversion rates were calculated from the ^{1}H NMR spectra.

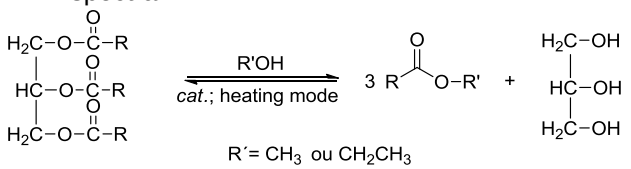

Figure 1. Transesterification reaction.

The use of hydrothermal reactor at 180 °C provided the most efficient heating method, and high rates of conversion (~100%) were obtained in 5h using 6 % w/w of the catalyst. The reaction did not occur using microwave irradiation, and the conversion rates were below 40% using the conventional heating mode.

The minimum concentration of *p*-sulfonic acid calix[4]arene to be used was determined keeping constant the mass of the oil, the volume of alcohol and the temperature (180ºC). The catalyst was used at concentrations ranging from 0-6% w/w relative to triglyceride. The most significant results were obtained using 4.5% and 3% w/w catalyst for the methanolysis and ethanolysis, respectively, with conversion rates nearly quantitative.

The optimal reaction time was determined calculating the conversion between 1h and 5h (Figure 2), using *p*-toulenesulfonic acid as standard. A total conversion was achieved in 5h for the two alcohols.

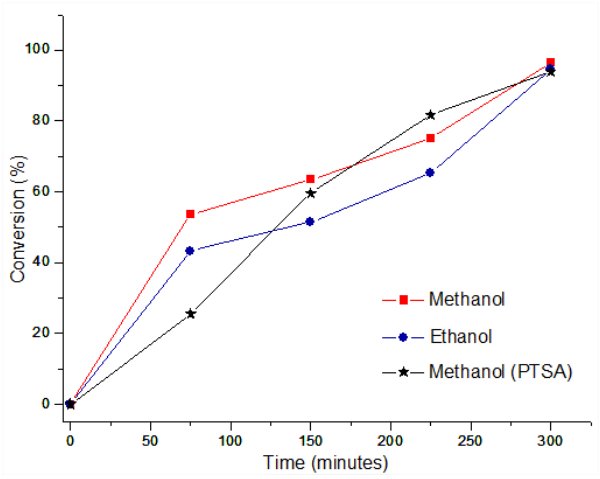

Figure 2. Conversion *x* time for the transesterification.

About 54% of the catalyst could be recovered by filtration and reused with rates of conversion of 96% and 76% in the two first cyles.

CONCLUSION

p-Sulfonic acid calix[4]arene appears to be an efficient homogeneous and recyclable organocatalyst for the transesterification of triglycerides present in vegetable oils.

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