



Studies on the synthesis of biodiesel catalyzed by $\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ in transesterification reactions of cotton, coconut and flaxseed oils

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Keywords: Niobic acid, Biodiesel, Transesterification.

INTRODUCTION

Transesterification is the predominant process for converting vegetable oils into biodiesel.¹

$\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ is known to have strong surface acidity and it reportedly² increases Lewis acid sites at temperatures higher than 500 °C, whereas Bronsted acid sites are more abundant at temperatures between 100 and 300 °C.

This study, which is a continuation of other investigations of this research group³, aims to implement a methodology for preparing methylic biodiesel through transesterification process of cotton, coconut and flaxseed oils, monitoring heat treatment of $\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ catalyst ratios and different proportions of catalyst mass/oil mass.

RESULTS AND DISCUSSION

The reactions were made with the addition of methanol (1.0 ml), cotton seed, coconut and linseed oils (0.5 g), DMSO (2.5 ml) as solvent to increase the boiling point of the mixture, and the catalyst in the proportions and treatment conditions shown in Table 1. For each vegetable oil, a reaction was made without the use of catalysts.

All reactions were made under heating conditions of 170 °C in a reflux system, and 0.5 mL of methanol was added every 8 hours. Reaction time was 48h. After the end of the reaction time, the products were extracted through multiple extractions.

Through the use of ^1H NMR spectroscopy, the products obtained were analyzed and quantified by the following expression:

$$\text{Conversion rates (\%)} = \frac{3 \text{ Ab}}{3 \text{ Ab} + 2 \text{ Ao}} \times 100\%$$

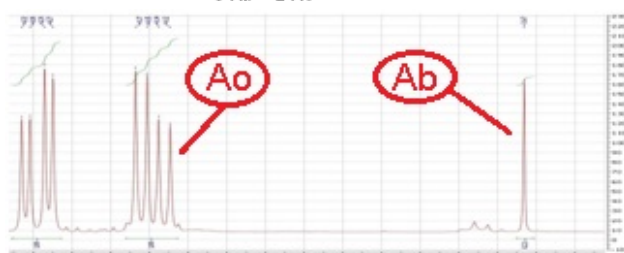


Figure 1. Methylene hydrogen's of the triglyceride (Ao) and methoxy hydrogen's ester (Ab) of biodiesel.

This expression relates the area of integration of the signal referring to the methoxy hydrogen's ester (Ab) and the area of methylene hydrogen's of the glyceridic of triglyceride (Ao).

Table 1. Proportions of catalyst/oil, calcinations temperature and conversion rates.

Oils	Catalyst/oil (m/m) %	Calcination T°C	Conversion rates (%)
Cotton seed	20	115 °C	6.2
		300 °C	9.5
	100	115 °C	12
		300 °C	17.4
Coconut	0	--	0
	20	115 °C	8.3
		300 °C	13.1
	100	115 °C	5.7
		300 °C	24.0
	0	--	0
Linseed	20	115 °C	5.7
		300 °C	18.4
	100	115 °C	5.7
		300 °C	21.0
	0	--	0

We observed an increase in conversion rates with the increase of the mass ratio of $\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$. This behavior was already expected, because with the increased amount of catalyst, there is increased availability in the reaction.

CONCLUSION

We found that the use of $\text{Nb}_2\text{O}_5 \cdot n\text{H}_2\text{O}$ as a catalyst promotes conversions in the transesterification of vegetable oils studied, but not in very satisfactory yields.

ACKNOWLEDGEMENTS

The authors thanks to CBMM, CAPES, CNPq, and LabPetro-DQUI/UFES.

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