Incipient wetness impregnation of HSiW on zeolite Y to butyl acetate production

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Abstract

HSiW supported on zeolite Y by incipient wetness showed no leaching on acetic acid esterification reaction with butanol.

Introduction

The development of new catalytic materials supported on solid matrices has been increasingly driven to enhance classical chemical processes.[1] The combination of heteropolyacids and zeolites improves catalytic performance and minimizes costs.[2] In this sense, the objective of this study was to prepare, characterize and evaluate the catalytic activity of $H_4SiW_{12}O_{40}$ (HSiW) supported on zeolite Y for acetic acid esterification reaction with n-butanol.

Results and Discussion

The catalysts prepared by incipient wetness impregnation[3] were characterized by XRF/EDX to determine the amount of supported HSiW. FT-IR results (Figure 1-I) confirmed the presence and maintenance of HSiW (increasing band at 921 cm^{-1}, related to the Keggin anion structure).[4] In addition, changes in the profile bands of pure zeolite suggest that there was an interaction with the HSiW. XRD (Figure 1-II) using aluminum sample holder enabled to quantify the crystallinity of the samples (Table 1), according to the method described in the literature.[5] Even after impregnation, the crystallinity remained high, indicating that the zeolitic structure had few modifications. The observed loss may be related to aluminum removal of the zeolite structure by HSiW that generates ideal environments for its deposition. Since it was not possible to identify characteristic peaks of HSiW in the XRD patterns, it is inferred a good dispersion on the zeolite Y. In order to obtain information about the stability of the catalyst, a leaching test using n-butanol and measurements of UV-Vis indicated no leaching of HSiW and a high interaction with the support. For the esterification reaction, the catalysts were previously activated at 300 °C/1 h on 5 mL microreactors. It was used 10% (m/m) of the catalyst in relation to the mass of acetic acid; molar ratio of acetic acid: n-butanol 1:2; 100 °C and variable reaction time. Conversion values obtained from GC/FID measurements (Table 1) indicated distinct activities, an increase with reaction time, and some catalysts were very active, exceeding 60% conversion after 2 h.

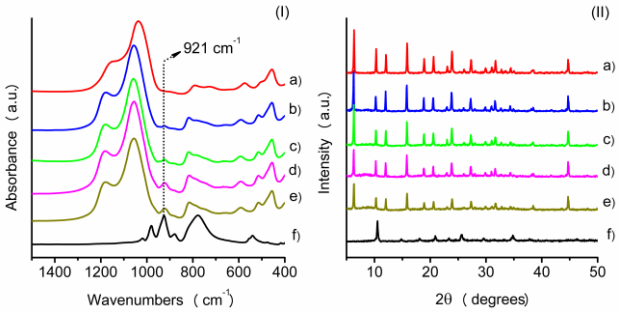

Figure 1. Left (I) FT-IR spectra: a) pure zeolite Y; (b, c, d, e) HSiW/Y (5, 7, 9, 12%); f) pure HSiW. Right (II) XRD patterns a) pure zeolite Y; (b, c, d, e) HSiW/Y (5, 7, 9, 12%); f) pure HSiW.

Table 1. Crystallinities values for the prepared catalysts and related conversions for different reaction times of esterification reaction.

Samples	Crystallinity (%)	Conversions (%)			
		1 h	2 h	3 h	4 h
5%HSiW/Y	98	20	37	55	58
7%HSIW/Y	87	25	47	65	68
9%HSIW/Y	81	33	57	63	71
12%HSiW/Y	71	36	58	72	73

Conclusions

There was a good stability in the interaction between the support and HSiW and promising conversion results for acetic acid esterification reaction.

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