Acetalization of Furfuraldehyde with argan nut shells carbon catalysts

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Several biomass waste derived carbons are widely used in catalysis applications, either as supports or as catalysts on their own (Figueiredo 2013; Li et al. 2020). Due to their diverse porous structure, surface chemistry, low density, low cost and easy accessibility (De et al. 2015; Matos, Bernardo, and Fonseca 2017), the waste based porous carbons have attracted much attention as catalysts for various organic reactions such as oxidative esterification reactions (Feng et al. 2020; Liu et al. 2016).

In this context, the main purpose of the present research was to evaluate argan nut shells as precursors of porous carbon and to test the obtained carbon material as catalyst in the acetalization of furfuraldehyde with alcohol at different reaction conditions.

The porous carbon was prepared from argan nut shells as raw materials via chemical activation using phosphoric acid H_3PO_4 85% (w/w) as activating agent, at a mass ratio of 1:1.5 (biomass/H_3PO_4). The impregnated biomass was subsequently submitted to carbonization/activation in a furnace, at 400 ° for 2 h under N_2 atmosphere. The obtained carbon was washed, then was oven dried at 70 °C over night. The dried carbon was named as ACH. A second carbon catalyst was prepared from ACH carbon after modification by acidic treatment (HNO_3). It was denoted as N-ACH.

The characterization of the obtained catalysts was performed by N_2 adsorption–desorption isotherms, Fourier transform infrared (FTIR) spectroscopy, elemental analyses, and scanning electron microscopy (SEM).

The textural analysis indicated that the porous carbon before acidic treatment presented a well developed porosity when compared to the treated carbon.

The catalytic acetalization experiments were carried out at 45 ºC, with a mixture of 100 mg of catalyst, 0.250 ml of undecane (internal standard) and 12 ml of ethanol; then the addition of 0.210 ml of 2-furfuraldehyde 99% (v/v) started the reaction. Samples were stirred in high-rate values for 7 hours. Blank experiment without catalyst was also performed in the same conditions. Samples were taken periodically and analysed by GC-FID (KONIC HRGC-5000B).

This work does not only open a way to valorize argan nut shells but also presents a simple and sustainable approach to synthesize catalysts. The effectiveness of the porous carbons, ACH and N-ACH, as catalysts are being conducted.

Table 1. Elemental composition of biomass, ACH and N-ACH samples.

	ANS	ACH	N-ACH
Elemental analysis			
C (wt. %)	47.8	59.1	54.65
H (wt. %)	6.54	4.39	2.06
N (wt. %)	0.33	<0.2	1.70
S (wt. %)	<0.03	<0.03	0.02
O* (wt. %)	45.2	35.7	41.57
Ashes (wt. %)	0.065	0.875	n.d.

*Calculated by difference (wt. %): 100 - (C + H + N + S + Ash).; n.d. – not determined.

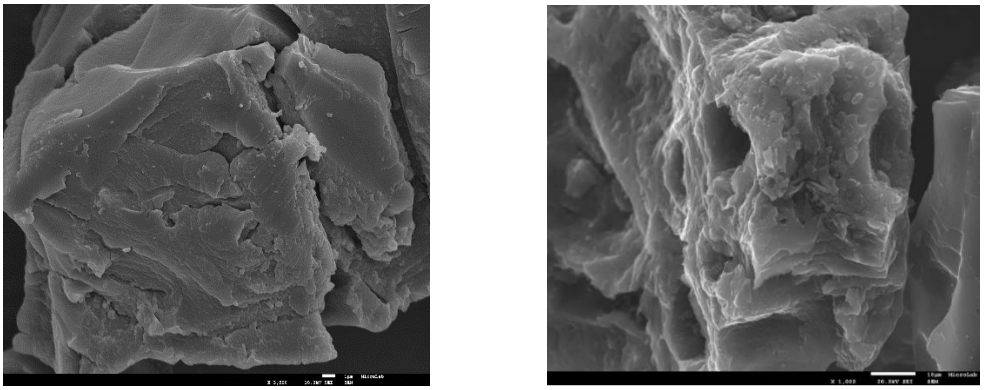

Figure 1. SEM images of ACH sample with magnification of 1000x (right) and of N-ACH sample with magnification of 3500x (left)

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