

Synthesis and characterization of the mesoporous AIMCM-41 as catalyst for biodiesel

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Abstract-The AIMCM-41 catalysts were prepared with different SiO₂/Al₂O₃ molar ratio (25, 50, 75, and 100), for use in the transesterification reaction. The influence of the reaction time (t = 8, 10, 12, or 14 h) on the catalyst synthesis was investigated keep on temperature at 443 K. The resulting materials were characterized by X-ray diffraction (XRD), ATG/ATD, FTIR and ²⁹Si and ²⁷Al NMR. The XRD results shown formation of a single phase, with good organization of the material. ²⁷Al NMR data suggested the incorporation of aluminum in tetrahedral positions in the structure. ²⁹Si NMR data showed that the silicon was also incorporated.

The use of biodiesel represented a promising fuel alternative for the energetically demanding world. Biodiesel, which is biodegradable, is defined as monoalkyl ester derived of renewable sources, such as vegetable oils and animal fats. The conventional process of production of biodiesel consists of the transesterification of the oil or fat via homogeneous catalytic processes in either acidic or basic medium. However, homogeneous catalysis presents some inconvenience such as the difficulties associated with the reuse of the catalysts and the necessity of successive washings of the reaction product for elimination of the catalytic residue. The major interest in heterogeneous catalysis in the transesterification reaction of vegetal oils resides on the easiness of catalyst recovery (and possible reuse), elimination of the neutralization steps and simpler purification, which is, thus, a more attractive and likely efficient method for biodiesel synthesis.

In this work, the molecular sieves AIMCM-41 was synthesized hydrothermally via modifications in a recently described method [1]. The mesoporous materials were prepared from a gel with molar composition: xSiO₂: 0.2 CTABr:Al₂O₃: 0.89 H₂SO₄:120 H₂O (x= 25, 50, 75, 100). The resulting gel was autoclaved and heated at 443 K for 8, 10, 12, or 14 h. The final solid was centrifuged and dried at 353 K. A subsequent ionic exchange was carried out by using ammonium chlorate. The solid was calcinated at 813 K for 4 h. The catalysts were characterized by XRD, ²⁷Al and ²⁹Si solid NMR, FTIR, and ATG/ATD.

All samples exhibited a very strong peak at 2° (2θ), assigned to 100 reflections and associated with the p6m hexagonal symmetry. The X-ray diffraction data indicated that the best reaction times for obtaining AIMCM-41 with SiO₂/Al₂O₃ ratios 25, 50, 75, and 100 were 12, 10, 8, and 10 h, respectively (Fig. 1). The ²⁷Al-NMR spectrum showed a resonance signal at 53.5 ppm, which was assigned to aluminum with tetrahedral coordination in the framework of AIMCM-41 (Figure 2a). This same fact was confirmed by ²⁹Si-NMR data. The spectrum (Figure 2b) showed five regions in the siliceous second sphere with atoms coordinate to aluminum as indicated chemical shift assigned on range of -97 at -74 ppm and the peak at -107 ppm attributed to Q⁴ species.

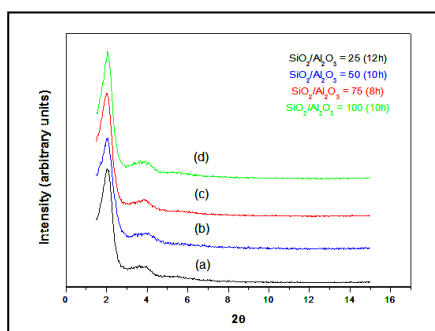


Figure 1: X-ray diffraction patterns of AIMCM-41 synthesized at SiO₂/Al₂O₃ molar ratio: (a) 25; (b) 50; (c) 75; and (d) 100.

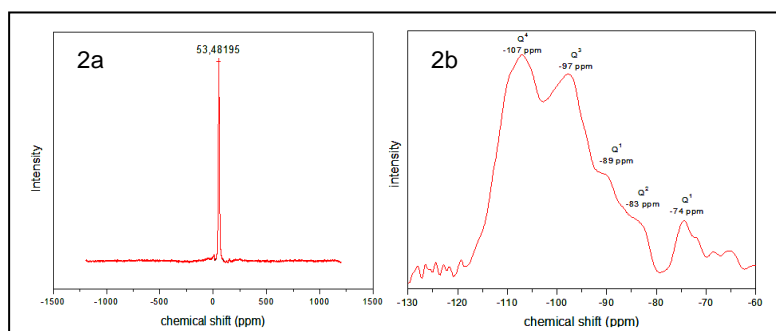


Figure 2: NMR data of (a) ²⁷Al and (b) ²⁹Si for AIMCM-41 synthesized at SiO₂/Al₂O₃ molar ratio of 50.