ZSM-5 fast synthesis employing soluble starch as template

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Solid catalysts with a zeolitic framework were synthesized in the ZSM-5 (MFI) topology employing tetrapropyl ammonium hydroxide (TPAOH) as structure directing agent and soluble starch as template in order to be employed in biomass revalorization reactions. It should be noted that starch is a soluble, biodegradable, low cost and nontoxic polymeric carbohydrate obtained from agricultural raw materials. Starch is employed in this synthesis process for pores generation and crystal connection to produce a material with MFI morphology type.

ZSM-5 is a medium pore material from the pentasil zeolites group. The traditional synthesis procedure [1] takes around 10 days to obtain the MFI structure. In this new procedure, ZSM-5 type material is obtained by a hydrothermal treatment but in a shorter crystallization time. The effect of time on structure and cristallinity was studied.

Zeolite synthesis was done employing sodium aluminate ($NaAlO_2$) as aluminum source, tetraethyl orthosilicate (TEOS) as silicon source, TPAOH and distilled water. The solution was aged at 80 °C for 210 minutes under vigorous stirring. Ethanol produced from TEOS hydrolysis was evaporated and a clear gel was obtained. Then, different starch quantities dissolved in aqueous solutions were incorporated and further stirred for 30 minutes at the same temperature. The result suspension was hydrothermally treated at 140 °C in a Teflon lined stainless steel autoclave under static conditions. Crystallization time was varied between 96, 48 and 24 hours and the obtained material was washed with distilled water and dried at 110 °C for 24 hours. Finally, the solid were desorbed under N_2 flow (20 ml/min) at 500 °C and further calcined at the same temperature for 8 hours in order to remove the carbonized starch and the organic structure directing agent trapped in the crystals. According to the crystallization time, samples were named as MFI-96, MFI-48 and MFI-24, respectively.

The obtained samples were characterized by X Ray Diffraction (XRD), BET and Scanning Electron Microscopy to confirm structure, crystallinity and surface area. From XRD patterns high crystallinity and MFI structure were confirmed, indicating the successful crystallization in short time when soluble starch was added in the synthesis gel. Surface areas calculated from BET were similar to that obtained for traditional ZSM-5 (approximately 360 m^2/g).

The so far synthesized and characterized materials were tested as catalysts for peanut shells valorization reactions to obtain platform molecules for the fine chemical industry.

Reference:

[1] J. Dedecek, V. Balgova, V. Pashkova, P. Klein, B. Wichterlova, Chem. Mater. 2012, 24, 3231−3239