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FLUORIDE-MEDIATED SYNTHESIS OF TON AND MFI ZEOLITES USING 1-BUTYL-3-METHYLIMIDAZOLIUM AS STRUCTURE-DIRECTING AGENT

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Abstract: In this work, 1-butyl-3-methylimidazolium (1B3MI) cation has been employed as a structure-directing agent for the synthesis of pure silica and Ti-containing zeolites in fluoride media. The 1B3MI OSDA exhibited distinct phase selectivity depending on the synthesis conditions, and particularly the concentration of the synthesis gel. Thus, under more diluted conditions ($H_2O/SiO_2=14$), the 1B3MI cation yielded pure silica TON zeolite with high crystallinity. Attempts to obtain Ti-containing zeolites, however, failed at this diluted condition even after 15 days of synthesis. In contrast, highly crystalline Ti-MFI was the only phase obtained upon reducing the H_2O/SiO_2 molar ratio from 14 to 7. The presence of Ti in the MFI zeolite framework was confirmed by both infrared and DRS UV-vis spectroscopies. The presence of intact OSDA molecules within the micropores was inferred from elemental analysis and ^{13}C CP-MAS NMR spectroscopy. On the other hand, thermogravimetric analyses showed a different packing of the 1B3MI cation in the pure silica TON and Ti-MFI samples. Moreover, the environments for Si and F species in the as-made materials were investigated by means of ^{29}Si and ^{19}F MAS NMR, respectively. Finally, molecular simulations showed that the most stable arrangement of the 1B3MI cations involves the location of the imidazolium rings on the channel intersections and of the butyl chains on the sinusoidal channels.

Keywords: zeolite synthesis; fluoride media; TON; MFI; ionic liquid; 1-butyl-3-methylimidazolium; titanium

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