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Mesoporous silica SBA-15 activated by iron ions - influence of silylation process on synthesis efficiency

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The paper considers mesoporous silica SBA-15 containing propyl-iron phosphate groups. This compound has been studied to examine efficiency of the synthesis route. Mössbauer spectroscopy has shown that iron has been successfully connected to propyl-phosphate groups incorporated in the silica matrix. Nevertheless Energy Dispersive X-Ray analysis (EDX) has indicated that amount of the iron ions is much lower, than it has been assumed. In-depth analysis of spectroscopic results pointed out, that the reason of this misalignment is a side reaction between phosphate units and hydroxyl units in the silica structure, occurring during hydrolysis process. These side reactions can also be observed in Raman spectroscopy, assisted by simulations. Connections between phosphonic acid groups and hydroxyl groups on the silica surface can be identified in simulations and their presence confirmed experimentally. Moreover, hydroxyl group left by the phosphor atom, that is not bounded to the metal ion, gives clear Raman peak in the activated silica spectrum. The paper presents this analysis and proves that it can be a powerful tool for the synthesis route efficiency examination.

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