

XD107: Abstract to poster contribution

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Synthesis of high surface area aluminophosphate and -phosphonate xerogels by non-hydrolytic sol-gel reactions

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We disclose the preparation of high-surface-area mesoporous aluminophosphates and aluminophosphonates by the non-hydrolytic sol-gel reactions (NHSG) of $\text{Al}(\text{NMe}_2)_3$ with trimethylsilylated phosphate $\text{OP}(\text{OSiMe}_3)_3$, phosphonates $\text{RP}(\text{O})(\text{OSiMe}_3)_2$ ($\text{R} = \text{Me}, t\text{Bu}, \text{Ph}$), and bis-phosphonates $(\text{Me}_3\text{SiO})_2(\text{O})\text{P}-\text{X}-\text{P}(\text{O})(\text{OSiMe}_3)_2$ ($\text{X} = \text{C}_6\text{H}_4, (\text{C}_6\text{H}_4)_2$) in dry toluene. The reactions proceed by silylamine elimination of $\text{Me}_3\text{SiNMe}_2$ to provide organic-inorganic hybrid xerogels with properties influenced by organic substituents and the Al:P ratio of the precursors. Dried xerogels exhibit large surface areas (up to $1000 \text{ m}^2 \text{ g}^{-1}$) and matrices based on condensed Al–O–P networks. They stay stable under relatively harsh thermal conditions. ^{27}Al , ^{13}C , and ^{29}Si solid-state NMR spectroscopy was employed to characterize the aluminum coordination and the residual amido and trimethylsilyl groups. The catalytic performance of NHSG prepared material was examined in gas-phase dehydration of ethanol to ethylene exhibiting conversion and selectivity comparable to weak solid acid benchmark catalysts. The number of unreacted surface groups was determined by gravimetric measurements and by thermal analysis (TG-DSC). These residual groups have the potential to be used in post-synthetic grafting of catalytically active metal centers.