SHAPE-SELECTIVE CATALYSIS pdf

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SHAPE-SELECTIVE CATALYSIS IN ZEOLITES Sigmund M. Csicsery Chevron Research Company, Richmond, California Introduction Zeolites have four properties that make them especially interesting for.

The ZSM-5 structure contains two intersecting channel systems of specific size and shape that restrict molecular diffusion into and through the framework. The porous material is therefore useful for catalytic reactions inside the nanopores of the material. The synthesis of ZSM-5 involves hydrothermal methods over a period of two days. Subsequent labs will involve characterization and calcination of the material, followed by an intrazeolite xylene isomerization reaction. A six-membered ring, for example, contains six "T-atoms" Si or Al and six oxygen atoms. For simplification, the latter are ignored in this nomenclature, as well as in structural representations of zeolites, where metal centers are joined by a straight line Figure 1. The MFI topology is one example of a zeolite. The dark atoms show the space-filling view. One example of a zeolite is ZSM-5 Figure 1 , which contains with two sets of perpendicular, intersecting channels, one defined by membered rings and the other by 8-membered rings. The ZSM-5 structure is a commonly occurring framework topology, where the chemical composition of the metal centers varies. As a result, there are now over 21 different names for this structure, and the material is instead commonly referred to by its assigned three-letter zeolite structure code, MFI. In this lab, the sodium form of ZSM-5 will be formed after 44 hours of heating. It will then be calcined to render the material porous and subsequently converted into the active acid form for the xylene isomerization reaction. The following preparation must be conducted in a fumehood. Solid silicic acid is a light material and is easily airborne, not unlike asbestos fibers. You must avoid breathing any small, air-suspended particles. In addition, n-propylamine is volatile and toxic. Grind up approximately 0. In a separate 50 mL beaker, prepare a solution of 0. Then add to this solution, 0. Carefully transfer the contents of the first beaker to the second and mix thoroughly. Finally, mix the contents of the 50 mL beaker thoroughly until the solution is homogeneous, and then allow to stir on a magnetic stir plate for approximately ten minutes. Transfer the majority of this mixture to a 45 mL or mL capacity Parr Stainless Steel Autoclave the former works better Seal the autoclave by first hand-tightening the top until it can turn no further. If you are using a mL capacity autoclave, first hand-tighten the screws, and then use an Allen wrench to tighten the six bolts, working your way around the circle several times. Lab 2 Remove the autoclave from the oven and place in a stream of cold water from a tap preferably in a fumehood to quickly bring the synthesis mixture to room temperature. When cooled, open the autoclave and vacuum filter the white solid on a Buchner funnel. Wash the solid on the filter paper with copious amounts of distilled water and suction dry the solid for at least 20 minutes. The dried powder should be a very fine, colorless powder. Weigh the solid, and remove a small sample for powder X-ray diffraction analysis. Calcination and Conversion of Zeolite ZSM-5 Set up the tube furnace assembly for the calcination step by placing the zeolite in an alundum or quartz boat. Spread the white powder out to maximize the surface area. The alundum boat is placed in the middle of the quartz of alumina tube. Attach a ground glass elbow at each end of the tube, with one end attached to an N2 cylinder and the other end immersed in a beaker of water or connected to a bubbler, to monitor the N2 flow rate. If the tube furnace is not programmable: It is therefore best to heat slowly at this point. If the tube furnace is programmable: Allow the materials to cool and weigh the resulting solid. Another sample is removed for X-ray analysis. The sodium form of the zeolite is converted to the acid form using the following procedure: The zeolite is collected by vacuum filtration. Repeat this process twice, following the final treatment by a thorough washing with distilled water, until the filtrate is free of sulfate anions. Test the filtrate by adding an aqueous solution of BaCl2 dropwise to the contents of the filter flask. The presence of a BaSO4 precipitate will indicate that the zeolite still contains adsorbed sulfate ions; if the solution is clear, you may proceed to the next step. Transfer the dried solid to the tube furnace by spreading it over a large surface area in the alundum boat. Do not pass N2 gas over the solid - the presence of O2 is essential for the conversion. At the end of this time period, you will notice that the zeolite has lost its brownish tinge and should appear white. Cool the material under a stream of nitrogen and store in a sealed vessel in a desiccator to preserve the acid hydrogen

form of the zeolite. Powder X-ray Diffraction Analysis Run powder X-ray diffraction spectra on your as-synthesized and calcined samples to confirm its structure, purity and crystallinity. Mix your sample with a very small amount of petroleum jelly just enough to make a paste , and "paint" the mixture onto the center of a glass slide. Your material should display a pattern similar to the characteristic X-ray diffraction pattern of ZSM-5 Figure 2, 3. A comparison includes the relative intensity of the peaks, peak width, and the 2q values also see Appendix. How do the d-spacings compare? Calcination of ZSM-5 changes the powder X-ray diffraction pattern from that of the as-synthesized material. Do not use too much glass wool or sample or the xylene will be unable to flow! Using vacuum grease, connect this central piece to glass tubes with ground joints, such that glass tubes are protruding from each end of the tube furnace. Connect the rest of the pieces shown in Figure 4, and fill the reagent flask with 8 mL of o-xylene. A catalytic reactor is used for the isomerization reaction. Flow N2 gas through the hot o-xylene at a low to moderate flow rate indicated by the bubbler to act as a carrier gas. Place the collection flask in an ice bath, to ensure condensation of the xylenes, which should occur within 10 minutes. Cool the product mixture to ambient temperature and take a 1H NMR spectrum of both o-xylene and the product. Integrate the two peaks to determine the extend of conversion to p-xylene Figure 5. Also analyze the product by gas chromatography GC. The mobile phase, or carrier gas, is an inert gas helium. The separation process occurs as a result of repeated sorption-desorption acts, that arise from the differences in the distribution coefficients of the individual sample components, during the movement of the sample components across the stationary phase. At the end of the column, the components should emerge at different times. They are then detected and the resulting signal is displayed on a recorder or printer. The peak area is used to determine the relative amounts of the individual components present in the sample. The retention times of these components are calculated and the peak area is determined by cutting them out and weighing them. Is the column capable of separating o-,m-, and p-xylenes? Keeping in mind that gas chromatography separates components on the basis of their boiling points and polarity, are your experimental results supported by theory? Adsorption Analysis Run the physisorption isotherm for the as-synthesized and hydrogen form of your zeolite. Print out the final data and compare with typical data of any zeolite in the literature. Construct o-, m-, and p-xylenes. Drive these molecules into the membered rings of the three dimensional channels. How does the zeolite pore size compare with the molecular diameters of the various xylenes? What do you observe and how does it relate to the catalytic results? Keep in mind that rates of diffusion and absorption of organic molecules is influenced both by the size and shape of the channel system and by the location of extraframework cations. The issue is far more complex than that provided by the CAChe component. For Your Report This experiment was a small-scale version of a major industrial process, as the size- and shape-selective zeolite ZSM-5 is an industrially important catalyst. X-ray powder diffraction spectra have been obtained on the initially synthesized zeolite and the sodium form. Compare the experimental spectra to literature spectra. Isomerization of o-xylene to p-xylene was performed and analyzed by a 1H NMR assay and gas chromatography. The zeolite and xylenes were modeled on CAChe and the results compared to the experimental catalytic method. In your report, propose a possible mechanism for the catalysis of o- to p-xylenes, and be sure to support this mechanism with literature references. Nature , , Structure, Chemistry, and Use; New York: Science , ,

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For example, catalysis by zeolites is a shape-selective catalysis. The pore size present in the zeolites ranges from pm. Thus, molecules having a pore size more than this cannot enter the zeolite and undergo the reaction.

3: Shape Selective Catalysis in Industrial Applications, Second Edition, - CRC Press Book

Shape-selective catalysis inzeolites Sigmund M. Csicsery Chevron Research Company, Richmond, California , USA (Received 1 September ; revised 10 FebruaTy) Small and uniform pores characterize zeolite catalysts.

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Shape selective catalysis differentiates between reactants, products, or reaction intermediates according to their shape and size. Only molecules whose dimensions are less than a critical size can enter the pores, have access to internal catalytic sites, and react there.

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