化学学报 1998 56 (4): 405-416 ISSN: 0567-7351 CN: 31-1320/06

FER型沸石的合成、结构与吸附性质

马明华,姜慧文,龙英才,孙尧俊

复旦大学化学系;复旦大学分析测试中心

收稿日期 修回日期 网络版发布日期 接受日期

摘要 在TMEDA(四甲基乙基二胺)-Na2O-SiO2-Al2O3-H2O体系(I),Na2O-K2O-Al2O3-SiO2-H2O-HCO3^--CO3^2^-体系(II)及Py(吡啶)-PrNH2(正丙胺)-HF-SiO2-H2O体系(III)中,
分别合成了纯相FER沸石及FER硅沸石。用粉末XRD, FT-IR, 29Si MAS NMR及TG/DTA等表征其结构性质,
并用超微量电子真空吸附天平测定这些沸石样品对正己烷, 甲醇和水的吸附等温线。结果表明:
各体系合成的样品虽然结晶度高, 呈现出FER沸石的典型结构特征, 但由于它们的组成和晶格微结构不同,
热稳定性与吸附性质有明显的差异。在(I)体系中合成的FER沸石层错缺陷少, 晶格完美,
正己烷与甲醇的吸附量可达到理论值, 结构破坏温度为1190℃。红外精细谱及29Si MAS
NMR高分辨谱证明FER硅沸石具有十分完美的骨架结构。由于晶胞收缩,
它对正己烷与甲醇吸附量略低于理论值, 并呈现出高度的疏水性。它的结构破坏温度高于1300℃。在(II)
体系中合成的FER型沸石结构缺陷多, 沸石孔中的钾离子不易被质子完全交换。它的正己烷与甲醇吸附量均较低,
而水的吸附量相对较高。吸附现象表明, 正己烷和甲醇都被吸附于FER沸石的十元环主孔道中, 分压较高时,
甲醇可通过八元环进入小笼, 而水的吸附性质则主要与各样品的Si-OH缺陷及骨架中的阳离子含量有关。

关键词 红外分光光度法 水 氧化铝 沸石 X射线衍射分析 吸附 氧化硅 吡啶 氧化钠 氧化钾 热重量分析 付里叶变换 四甲基乙基二胺

分类号 0612

Synthesis, structural characterization and adsorption of FER-type zeolites

MA MINGHUA,JIANG HUIWEN,LONG YINGCAI,SUN YAOJUN

Abstract Pure phase FER zeolite and siliceous FER zeolite have been synthesized in the systems of tetramethyl ethylene diamine-Na2O-SiO2-Al2O3-H2O(I), Na2O-K2O-Al2O3-SiO2-H2O-HCO3^--CO3^2^-(II), and pyridine-propylamine-HF-SiO2-H2O(III) respectively. All the samples prepared were characterized by chemical composition, SEM, XRD, FT-IR, 29Si and 27Al MAS NMR, and TG/DTA methods. Adsorption isotherms of hexane, methanol and water on these samples were measured using super micro-adsorption-electron balance in vacuum. It is found that all the samples are of high-crystallinity, whereas their thermal stability and adsorption property are remarkably varied because of the differences in composition and micro-defects in the structure of these zeolites. Zeolite FER synthesized in system (I) has less stacking faults and perfect framework. Its adsorption capacities of hexane and methanol are in good agreement with calculated values. The temperature of the zeolite structure damage is at 1190℃. Siliceous FER zeolite possesses perfect framework as revealed by FT-IR and high resolution 29Si MAS NMR spectra. Its adsorption capacities of hexane and methanol were slightly lower than calculated values because of the unit cell contration. It has high hydrophobic property. The temperature of the zeolite framework collapse is higher than 1300℃. More stacking faults are found in FER zeolite obtained from system (II). Potassium cations in the channels are difficult to be ion exchanged with protons. Its adsorption capacities of hexane and methanol are obviously low, whereas the capacity of water is relatively high. The temperature of the zeolite structure damage is at 1169℃. Accroding to the sorption isotherms, hexane and methanol are adsorbed in the ten-membered ring channel. Methanol molecules can enter the sjmall cage through the eight-membered ring window at jhigh partial pressure. The adsorption of water correlates to Si-OH defect and concentration of cations in the zeolites.

Key words INFRARED SPECTROPHOTOMETRY WATER ALUMINIUM OXIDE ZEOLITE X-RAY DIFFRACTION ANALYSIS ADSORPTION SILICON OXIDE PYRIDINE SODIUM OXIDE POTASSIUM OXIDE THERMOGRAVIMETRY FOURIER TRANSFORM

DOI:

通讯作者