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Effect of flow field distribution on the synthesis of sub-micron ZSM-5 molecular sieve in a quasi-solid system

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Abstract: Scale-up synthesis of sub-micron ZSM-5 molecular sieve in a quasi-solid system was investigated. Compared with traditional hydrothermal synthesis, the synthesis in a quasi-solid system has the advantages of high yield, short crystallization time, low energy consumption as well as low emissions. However, the high solid content in the quasi-solid system can cause the mass and heat transfer problems and make scalable production difficult. In order to solve the problem, we have developed a method for the optimization of the mass and heat transfer. By this method one can vary the flow field in the reactor by changing the stirrer speed. Scale-up synthesis of the sub-micron ZSM-5 molecular sieve in a quasi-solid system was carried out in a 5 L reactor with double propeller-type agitators. The process was investigated with product characterization using X-ray diffraction (XRD) and scanning electron microscopy (SEM) and the flow field information was collected using laser Doppler velocimetry (LDV). The results showed that the flow field patterns can be tuned by using different stirrer speeds, the morphology and size of as-synthesized of ZSM-5 can be effectively controlled.

Key words: Quasi-solid system, sub-micron ZSM-5, scale-up, stirrer speed, flow field characteristics

1 Introduction

ZSM-5 zeolite had been widely investigated by many researchers since it was first reported by the Mobil group in 1978 (Kokotailo et al, 1978). It possesses a unique two-dimensional 10-ring channel system and shows favorable thermal stability, acidity, and shape-selectivity. Therefore, much effort has been devoted to its synthesis and application (Yamamura et al, 1994; Sang et al, 2004; Wang et al, 2007; Mohamed et al, 2005; Minkee et al, 2009). ZSM-5 zeolite had been successfully used in catalysis (Chem and Garwood,1978; Wallenstein and Harding, 2001), sorption-separation as well as ion-exchange applications. Its extensive application potential greatly promoted its production in a commercial scale (Casci, 2005).

Our group described the synthesis of a molecular sieve in a quasi-solid system for the first time (Dou et al, 2008). The synthesis of the molecular sieve in the quasi-solid system has the advantages of high yield, short crystallization time, low energy consumption as well as lower emission than conventional hydrothermal synthesis. However, the high solid content in a quasi-solid system makes the mass transfer and heat transfer more difficult than in the conventional hydrothermal synthesis system. Therefore, the scale-up in the quasi-solid system is more difficult. The scale-up effect is mainly caused by the temperature gradient, concentration gradient, and residence time of the reactant in different scale reactors. To solve scale-up problem in the synthesis of molecular sieves, many studies have been conducted aiming at changing reactants and crystallization time (Xie et al, 2006). Laser Doppler velocimetry (LDV) has proven to be more accurate in the measurement of the flow field in the stirred tank reactor than any other technique, since it can provide flow information even in the unsteady and highly turbulent flow regions as well as in the return flow area of the tank without fluid contact (Yianneskis et al, 1987; Chen et al, 2007; Wang and Chen, 2009; Schwingshackl et al, 2009). In this work, scale-up synthesis of sub-micron ZSM-5 molecular sieve in a quasi-solid system was carried out in a 5 L reactor with double propeller-type agitators. For the first time, the crystallization process was controlled by tuning the flow field pattern in the reactor by changing the stirrer speed. The process of the scale-up synthesis in the quasi-solid system was investigated with product characterization using X-ray diffraction (XRD) and scanning electron microscopy (SEM) and the flow field information was collected by laser Doppler velocimetry (LDV).

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