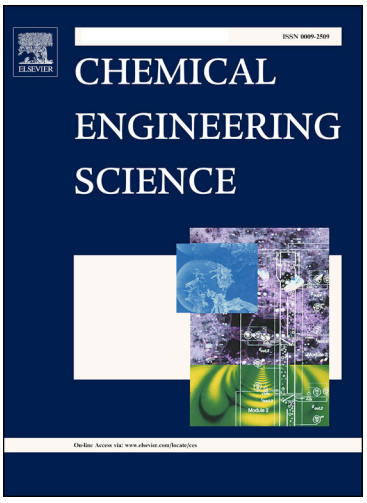

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Cyclohexanone ammoximation over TS-1 catalyst without organic solvent in a microreaction system

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Abstract

We herein propose a highly efficient technology for the synthesis of cyclohexanone oxime, a key intermediate for the production of caprolactam. The classical cyclohexanone ammoximation over TS-1 catalyst needs *t*-butanol as the organic solvent to change the reaction system from liquid-liquid-solid to liquid-solid for much better reaction performances, which greatly increases the reactor size and the downstream separation cost. In this work, a solvent-free liquid-liquid-solid heterogeneous reaction was successfully implemented in a microreaction system containing two T-junction microreactors. The effects of temperature, pressure, residence time, concentrations of reactants and catalyst on the reaction performance were systematically investigated. This developed technology resulted in a 99.87% conversion of cyclohexanone and nearly 100% selectivity to cyclohexanone oxime within 3 minutes. These results indicate that the new process could provide a more environmental friendly and economic approach to caprolactam industry.

Keywords: Liquid-Liquid-Solid heterogeneous reaction; Cyclohexanone ammoximation; TS-1; Microreactor

1. Introduction

Ammoximation of cyclohexanone is an important process to synthesize cyclohexanone oxime, which is a key intermediate in the production of caprolactam, the monomer for nylon-6.(Dahlhoff et al., 2001) In 1987, a single-step ammoximation process was developed, in which cyclohexanone reacts with ammonia and hydrogen peroxide to form cyclohexanone oxime with the catalysis of titanium silicalite-1 (TS-1). It has been demonstrated that the process on an industrial scale could result in a high yield up to 99%.(Lin et al., 2014; Thangaraj et al., 1991) In the industrial process, the organic solvent *t*-butanol is used to improve the reaction performance.(Yip and Hu, 2011; Zhao et al., 2011) However, the large amount of solvent results in much larger size reactor, environmental drawbacks and very high separation cost. Moreover, it is

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