Solvothermal synthesis of vanadium phosphate catalysts for n-butane oxidation

ABSTRACT

In this paper, we have developed a simple, low-cost, template-free and surfactant-free solvothermal process for synthesis of vanadyl hydrogen phosphate hemihydrate ($VOHPO_4 \cdot 0.5H_2O$) with well defined crystal size. The synthesis was performed by reaction of $VPO_4 \cdot 2H_2O$ with an aliphatic alcohol (isobutyl alcohol, 1-pentanol, 1-hexanol, 1-heptanol or 1-decanol). This afforded well crystallized $VOHPO_4 \cdot 0.5H_2O$ by solvothermal methods at 120 °C temperature. This new method significantly reduced the preparation time and lowered production temperature (50%) of catalyst precursor ($VOHPO_4 \cdot 0.5H_2O$) when compared to conventional hydrothermal synthesis methods. By varying the reducing agent, the solvothermal evolution process from layered tetragonal phase $VOPO_4 \cdot 2H_2O$ to orthorhombic phase $VOHPO_4 \cdot 0.5H_2O$ was observed. It was found that the length of carbon chain in an alcohol in the solvothermal condition had a great impact on chemical and physical properties of resulting catalysts. Interestingly, there was no trace of $VO(H_2PO_4)_2$ an impurity noted to be readily formed under solvothermal preparation condition. Therefore, this study introduces a more facile synthetic pathway to V(III) compounds. In addition, the microwave-synthesized catalysts exhibited some properties superior to those of conventionally synthesized catalyst such as better stability, crystallinity, and catalytic activity in the production of maleic anhydride. The characterization of both precursors and calcined catalysts was carried out using X-ray diffraction (XRD), inductively coupled plasma-atomic emission spectrometer (ICP-AES), N_2 physisorption, temperature programmed reduction (H_2-TPR) and scanning electron microscopy (SEM). The XRD pattern of the active catalyst prepared by this solvothermal method confirmed the presence of smaller crystal size (between 6 and 13 nm along 0 2 0 planes) of vanadiumphosphatecatalyst with higher specific surface area. Finally, the yield of maleic anhydride was significantly increased from 29% for conventional catalyst to 44% for the new solvothermalcatalyst

Keyword: Vanadium phosphate; Vanadyl hydrogen phosphate hemihydrate; Solvothermal synthesis; High pressure autoclave; Microwave heating; Primary alcohol; n-Butaneoxidation.