

# Preparation, Solid-state Characteristics, and Catalytic Properties of Promoted Vanadium Phosphate Materials

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*Dedicated to Professor Manfred Meisel on the Occasion of his 65<sup>th</sup> Birthday*

**Abstract** The syntheses of transition metal promoted (M = Co, Cr, Fe, Mo) supported vanadium phosphate (VPO) catalysts (TiO<sub>2</sub> (anatase), γ-Al<sub>2</sub>O<sub>3</sub>) and their characterization by N<sub>2</sub>-adsorption, X-ray diffraction (XRD), FTIR-spectroscopy and determination of V-valence state is reported. The catalytic properties were checked in the heterogeneous catalytic ammoxidation of 2,6-dichlorotoluene to the corresponding nitrile. The catalyst samples were prepared by synthesis of the precursor compound VOHPO<sub>4</sub> · 0.5 H<sub>2</sub>O, impregnation using various metal/salt solutions and mixing with the support materials. The characterization revealed increased surface areas for all the promoted samples in comparison

to the basic materials. XRD showed the formation of (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> after calcinations as well as patterns of support materials (anatase, γ-Al<sub>2</sub>O<sub>3</sub>). The formation of crystalline proportions of mixed oxides were not observed. The catalytic ammoxidation runs revealed a significant effect of the promoter metals on the catalytic properties by an increase of yield by ca. 20 % compared to bulk VPO. Almost complete conversion of 2,6-dichlorotoluene and 81 % yield of nitrile were observed using a 25 % VPCoO/γ-Al<sub>2</sub>O<sub>3</sub> catalyst.

**Keywords:** Vanadium; Phosphoric; Catalytic; Heterogeneous catalysts

## Synthese, Charakterisierung und katalytische Eigenschaften von promotierten Vanadiumphosphaten

**Inhaltsübersicht** Es wird über die Synthese von Übergangsmetall-nienpromotierten (M = Co, Cr, Fe, Mo) Vanadiumphosphat-Trägerkatalysatoren (TiO<sub>2</sub> (Anatase), γ-Al<sub>2</sub>O<sub>3</sub>), deren Charakterisierung durch N<sub>2</sub>-Adsorption, Röntgenbeugung, FTIR-Spektroskopie und Bestimmung der V-Oxidationszahl sowie die katalytische Auswertung in der heterogenen katalysierten Ammonoxidation von 2,6-Dichlorotoluol zum entsprechenden Nitril berichtet. Die Katalysatormaterialien sind über die Synthese der Vorläuferverbindung, VOHPO<sub>4</sub> · 0.5 H<sub>2</sub>O, der Impregnierung mit den entsprechenden Metallsalzlösungen sowie der Vermischung mit den Trägerkomponenten zugänglich. Die Charakterisierung zeigte für alle Promotier-

ten Katalysatoren deutlich erhöhte Oberflächenwerte im Vergleich zu den Ausgangsverbindungen. Mit Röntgenbeugung konnten nur die Phasen von (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub>, der aus dem Vorläufer (VOHPO<sub>4</sub> · 0.5 H<sub>2</sub>O) entstehenden Verbindung, sowie die Reflexe des Trägersmaterials (Anatase) nachgewiesen werden. Kristalline Mischphasen wurden nicht beobachtet. Die katalytischen Versuche zeigten einen deutlichen Effekt der Promotierung. Ausbeuteerhöhungen um ca. 20 % konnten beobachtet werden. Mit einem 25 % VPCoO/γ-Al<sub>2</sub>O<sub>3</sub> Katalysator wurden in der Ammonoxidationsreaktion bei vollständigem Umsatz 81 % Nitril erhalten.

## Introduction

Vanadium phosphorus oxide (VPO) catalysts continue to attract considerable research attention in recent times in view of their high potentiality towards various oxidation and ammoxidation reactions such as n-butane conversion to maleic anhydride and methyl aromatics ammoxidation to their corresponding nitriles [e.g. 1, 2]. Literature survey re-

veals that the best industrial catalysts from VPO family are generally not used in the form of pure VPO phases but incorporate a broad range of promoter compounds.

The addition of chromium has surface area increasing effect [3] and enhances the presence of amorphous VPO phases [4]. Iron modifies VOPO<sub>4</sub>/(VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> dispersion [5]. The formation of solid solutions within (VO)<sub>2</sub>P<sub>2</sub>O<sub>7</sub> (VPP) is reported and it acts as an electronic promoter [6]. Cobalt addition leads to the formation of cobalt pyrophosphate structure that fills the micro pore voids and macro defects, and prevents gas phase loss of phosphorus from the catalysts [7]. Cobalt also causes an alteration of the micro structure of the catalyst and modifies VOPO<sub>4</sub>/VPP dispersion too [5]. Molybdenum facilitates easy desorption of products from the catalyst surface [3], it maintains optimal defect concentration [8] and blocks non-selective sites. Its presence leads to an increased exposure of (100) crystal plane and

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