

## Chapter 9

# Polymolybdate Supported Nano Catalyst for Desulfurization of Diesel

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### ABSTRACT

*The Catalytic oxidative desulfurization (Cat-ODS) comprises of molybdenum based catalyst, tert-butyl hydroperoxide (TBHP) as oxidant and dimethylformamide (DMF) as solvent for extraction. A series of polymolybdates supported alumina were prepared using the wet impregnation method. This potential catalyst was characterized by FTIR, FESEM-EDX and XPS for its physical properties. From catalytic testing, Fe/MoO<sub>3</sub>-Al<sub>2</sub>O<sub>3</sub> calcined at 500°C was revealed as the most potential catalyst which gave the highest sulfur removal under mild condition. The sulfur content in commercial diesel was successfully reduced from 440 ppmw to 88 ppmw under mild condition followed by solvent extraction. Response surface methodology involving Box-Behnken was employed to evaluate and optimize Fe/MoO<sub>3</sub>/Al<sub>2</sub>O<sub>3</sub> preparation parameters (calcination temperature, catalyst loading, and Fe loading) and their optimum values were found to be 550 °C, 10 g/L, and 10%. of calcination temperature, catalyst loading, and Fe loading. Based on these results, the reaction mechanisms of peroxy oxygen were proposed.*

### INTRODUCTION

Diesel can be considered the most potential fuel for transportation following gasoline. However, sulfur containing compounds in diesel are the main sources of acid rain and air pollution. In many countries around the world, environmental regulations have been introduced to reduce the sulfur content of diesel fuel and other transportation fuels to ultra-low levels (10–15 ppm)(Zongxuan et al. 2011).

Hydrodesulfurization (HDS) is the conventional method for lowering sulfur levels in diesel oil, but this technology requires severe conditions and is expensive. Hence, oxidative desulfurization (ODS) has

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been considered as an alternative technology for deep desulfurization of light oil (Wang et al., 2012). The aim of the present work was to develop alumina-supported polymolybdate oxide-based catalysts - *tert*-butyl hydroperoxide system - in oxidative desulfurization activity. Secondly, the potential catalyst was applied to be used in investigating oxidative reaction mechanism. This work also aimed to produce ultra-low sulfur diesel (< 15 ppm-S).

## **BACKGROUND**

Various studies on catalytic oxidation systems have been reported such as,  $\text{H}_2\text{O}_2/\text{Mo}/\text{Al}_2\text{O}_3$  (García Gutiérrez et al 2006) and  $\text{Fe}^{2+}\text{-CH}_3\text{COOH-H}_2\text{O}_2$  (Bhasarkar et al. 2013, Bolla et al. 2012). *Tert*-butyl hydroperoxide (TBHP) has been considered a powerful oxidant amongst sulfur compounds, and the best result is achieved when utilizing TBHP with heterogeneous catalysts (Wan Abu Bakar et al. 2012). Among heterogeneous catalysts, transition metal oxides such as Cu, Ti, Cr, Fe, Co, W and V are intensely studied in oxidation reactions (Al-Shahrani et al. 2007); (Bagiyan et al. 2004) ; (Murata et al. 2004). However, this kind of process was limited to monometallic oxides catalysts and most studies focused on model diesel. The use of alumina supported polymolybdate based catalyst for the activation of TBHP is an interesting alternative as it is the most desirable improvement of the ODS process (Abdullah et al. 2015). To date, the polymolybdate oxide catalyst doped with Fe has never been reported and explored for ODS reaction. Thus, the performance of new supported bimetallic oxide catalysts in commercial diesel will be reported. Secondly, the potential catalyst was also used to investigate the activity and oxidative reaction mechanism. This work was focused on developing a cost effective catalyst for ODS of commercial diesel under mild reaction conditions. Response surface methodology (RSM) is a set of techniques used to develop models from experimental or simulation data and can be used to obtain an optimal response (Dube et al. 2013). Compared to other RSM designs, Box-Behnken design (BBD) is the most frequently employed and offers some advantages; requiring few experimental points and high efficiency (Box et al. 1960). A Box-Behnken design was applied to determine the optimum sulfur removal, and also to explain the relationships between sulfur removal and three parameters, calcination temperature, catalyst loading and Fe loading.

## **METHOD**

### **Materials**

All solvents and chemicals were used without further purification. Phosphomolybdic acid hydrate and *tert*-butyl hydroperoxide (aqueous, 70wt%) were obtained from Across Organic. Iron(III) nitrate nonahydrate (99%), copper(II) nitrate trihydrate (99.5%) and cobalt(II) nitrate hexahydrate (99%) were obtained from QRëc.  $\lambda\text{-Al}_2\text{O}_3$  beads (3-5 mm diameter) was obtained from Sigma-Aldrich, and dimethylformamide (DMF) was purchased from MERCK. Commercial diesel fuel (440 ppm sulfur) was obtained from Petronas, Malaysia.

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