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Use of Direct Microwave Irradiation in the Synthesis of Vanadium Phosphorus Oxide Catalysts via Vanadyl Hydrogen Phosphate Sesquihydrate Precursor

ABSTRACT

Four vanadyl pyrophosphate (VPO) catalysts were prepared via sesquihydrate precursor route using direct microwave irradiation and reflux methods in Stage 1 and/or 2. The synthesised catalysts were denoted as VPOs-M_sM_s, VPOs-R_sR_s, VPOs-R_sM_s and VPOs-M_sR_s; where VPOs represents VPO catalysts prepared via sesquihydrate precursor route, Ms and Rs represent direct microwave irradiation and reflux synthesis process, respectively. Direct microwave irradiation method was found to significantly reduce the duration of each stage of the precursor synthesis from 24 h to 2 h. A distinctive secondary structure of chrysanthemum needle-like morphology which only appear in VPOs-M_sM_s has increased 35.4% of the specific surface area as compared to VPOs catalyst produced via conventional reflux synthesis method (VPOs-R_sR_s). Direct microwave irradiation has induced the removal of more than 4 times of total amount of oxygen atoms from the lattice of V⁴⁺ and V⁵⁺ phases as compared to conventional reflux method counterpart, which ultimately produced VPOs catalysts with greater catalytic performances and TON. In short, direct microwave irradiation is capable of producing VPOs catalyst with higher efficiency of improved activity and selectivity as compared to conventional reflux method.

▪ INTRODUCTION

Light hydrocarbon such as *n*-butane has gained significant attention throughout the whole world due to the mass production of maleic anhydride. Such worldwide commercial application is highly essential for modern chemistry in reducing the carbon dioxide emission [1]. The oxidation of *n*-butane to Maleic Anhydride (MA) is the only industrial process of selective oxidation of paraffin and *n*-butane selective oxidation on VPO catalyst is a much-studied reaction in heterogeneous catalysis [2]. Vanadyl pyrophosphate catalysts are the main focus of studies for selective oxidation of *n*-butane to MA [3]. A key property of (VO)₂P₂O₇ is its ability to carry out the first step, which is the activation of *n*-butane, with high selectivity [4].

n-Butane oxidation to MA production using the commercial vanadyl pyrophosphate (VPO) catalysts in the industry exhibited dissatisfaction production of 53 % to 65 % molar yield to MA and 85 % to 86% conversion of *n*-butane [5]. Therefore, intensive effort has been carried out to uplift the catalytic performances of VPO catalyst. Catalytic characteristic and behaviour of the VPO catalyst is greatly influenced by the preparation route and types of precursors, the synthesis method, the phosphorus to vanadium ratio, the calcination condition of activation procedure which include temperature, duration and environment, dopant and/or support system used in the catalyst.

The catalytic properties of the (VO)₂P₂O₇ depend on the method by which the precursor is prepared [6]. Generally, VOHPO₄·0.5H₂O is best catalyst precursor which is converted to (VO)₂P₂O₇ during activation. However, the high yield of by-products of this catalyst remains a

serious problem. The low activity from the conventional VPO catalyst cause the issue of low supply-high demand of MA in the market. The existing reducing agent, benzyl alcohol is a toxic compound which is harmful to the environment. Therefore, an alternative route via vanadyl hydrogen phosphate sesquihydrate precursor was developed by using 1-butanol as reducing agent to convert $\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$ into $\text{VOHPO}_4 \cdot 1.5\text{H}_2\text{O}$ precursor [4,7,8].

Microwave irradiation treatment is applied to facilitate a chemical reaction has become a popular and promising technique in the scientific research community [8-13]. The advantages of applying microwave irradiation in the synthesis of catalyst are shorter synthesis time and improved catalytic performances [14]. This has allowed the synthesis of VPO catalysts to be simple, low cost, and efficient with improved catalytic performances for *n*-butane oxidation to MA. The yield of MA was reported to substantially increase from 21% for conventional catalyst to 37% for VPO catalyst synthesised via microwave irradiation treatment [15]. During the microwave process, the energy interacts with materials at the molecular level and internal electric fields are generated within. This ability has resulted the conversion of electromagnetic energy to heat within the irradiated material [16,17].

Researchers have reported that the rate of chemical reactions and product selectivity were markedly improved when microwave irradiation treatment was applied during the synthesis of VPO catalysts [18,19]. In this paper, the effect of direct microwave irradiations on the physical, chemical, reactivity and catalytic performances of VPOs catalysts are studied.

▪ EXPERIMENTAL SECTION

Materials

The materials used in this study were V_2O_5 ($\geq 98\%$ purity, Merck), $\alpha\text{-H}_3\text{PO}_4$ (85% purity, Merck), distilled water, 1-butanol ($\geq 99.9\%$ purity, Merck), 0.75% *n*-butane in air mixtures (MOX).

Instrumentation

The instrumentations used in this study were Froilabo hotplate magnetic stirrer, microwave digester (Berghof Speedwave 4), XRD-6000 Diffractometer (Shimadzu), Sorptomatic 1990 (Thermo Finnigan), Optical Emission Spectrometer Optima 2000 DV (Perkin Elmer), S3400N Scanning Electron Microscope (Hitachi) with EDAX software, TPDRO 1100 (Thermo Electron), catalytic fixed-bed microreactor with online TRACE GC Ultra TM (Thermo Scientific).

Catalyst Preparation

VPOs catalysts were synthesised in a two-step procedure, involving the synthesis of vanadyl phosphate dihydrate ($\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$) precursor in Stage 1 followed by the synthesis of vanadyl hydrogen phosphate sesquihydrate ($\text{VOHPO}_4 \cdot 1.5\text{H}_2\text{O}$) precursor in Stage 2.

In Stage 1, dihydrate precursor was prepared using two different methods, i.e. conventional reflux and direct microwave irradiation methods. In conventional reflux method, V_2O_5 (15.0 g from Merck) was suspended into 90 mL of $\text{o-H}_3\text{PO}_4$ (85% from Merck) and 360 mL of distilled water. This mixture was stirred and refluxed at 393 K for 24 h. As for direct microwave irradiation method, a mixture of 15 g V_2O_5 , 90 mL of $\text{o-H}_3\text{PO}_4$ and 360 mL of deionised water were introduced into a Teflon-lined microwave vessel. The vessel was then undergone microwave irradiation for 2 hours at 393 K using a microwave digester (Berghof Speedwave 4, at 2450 MHz and an output power of 850 W). Both mixtures from Stage 1 synthesis methods were cooled to room temperature and the resulting yellow solid ($\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$) was recovered by centrifugation and oven dried for 24 hours.

In Stage 2, both $\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$ synthesised by conventional reflux and direct microwave irradiation were divided into two equal portions. First portion of $\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$ (i.e. 7.5 g) was refluxed for 24 h with 1-butanol (116.5 ml from Merck). Second portion was introduced into a Teflon-lined microwave vessel with 7.5 g of $\text{VOPO}_4 \cdot 2\text{H}_2\text{O}$ and 116.5 ml of 1-butanol. The vessel was then undergone direct microwave irradiation for 2 hours at 393 K using the same microwave digester with the same parameter as Stage 1. The resulting whitish blue solids from both synthesis methods were then oven-dried for 24 hours. The precursors obtained were then calcined in a reaction flow of 0.75% *n*-butane in air mixtures at 733 K for 24 hours to generate the active VPOs catalysts.

The calcined VPOs catalysts were denoted as VPOs- M_sM_s , VPOs- R_sR_s , VPOs- R_sM_s and VPOs- M_sR_s , where VPOs represents VPO catalyst prepared via sesquihydrate route; M_s represents microwave irradiation synthesis process; and R_s represents the reflux synthesis process. The position of M_s/R_s represents the Stage 1/Stage 2 synthesis, respectively.

Catalysts Characterization

X-ray Diffraction (XRD) patterns were acquired using a Shimadzu model XRD-6000 diffractometer, which employed Cu K α radiation to produce diffraction patterns from crystalline powder samples at room temperature. The specific surface areas of the VPO catalysts were determined utilizing the Brunauer-Emmett-Teller (BET) method with nitrogen physisorption

performed at 77 K. This analysis was carried out using the Thermo Finnigan Sorptomatic 1990 nitrogen adsorption-desorption analyzer.

For assessing the bulk chemical composition, a sequential scanning Inductively Coupled Plasma - Optical Emission Spectrometer (ICP-OES), namely the Perkin Elmer Optical Emission Spectrometer Optima 2000 DV, was employed. Secondary structure images were captured using a Hitachi S3400N Scanning Electron Microscope (SEM). Energy-Dispersive X-ray (EDX) analyses were conducted using the EDAX software. Temperature Programmed Reduction (TPR) in H_2/N_2 was performed using the Thermo Electron TPDRO 1100 instrument, which incorporated a thermal conductivity detector (TCD).

The average oxidation number of vanadium (V_{AV}) within the VPO catalysts was determined via the redox titration method [20]. A laboratory fixed-bed microreactor on a small scale was established to conduct the oxidation of *n*-butane to Maleic Anhydride. The experimental conditions involved a gas hourly space velocity (GHSV) of 2400 h^{-1} at 673 K, using a VPO catalyst sample (250 mg) with a 1.0% *n*-butane and air mixture flowing through the reactor. The resulting gases released as products were directed into a Thermo Scientific TRACE GC Ultra TM gas chromatograph through a heated metal pipeline for gaseous analysis. This analysis aimed to generate insights into the catalytic performance, including activity and selectivity, of the catalysts.

▪ RESULTS AND DISCUSSION

X-ray diffraction (XRD)

VPOs catalysts synthesised via direct microwave irradiation and conventional oven reflux showed diffraction patterns comprising of well-crystallised $(VO)_2P_2O_7$ phase (Figure 1). Three main characteristic peaks were observed at $2\theta = 22.9^\circ$, 28.4° , and 29.9° were assigned to (0 2 0), (2 0 4), and (2 2 1) planes, respectively (JCPDS File No. 34-1381). Two V^{5+} phases were also found in the XRD profiles, i.e. β -VOPO₄ at $2\theta = 21.3^\circ$ (JCPDS File No. 27-0948) and α_{II} -VOPO₄ at $2\theta = 25.3^\circ$ and 29.3° (JCPDS File No. 34-1247) [4,6,18]. A comparative analysis of phase intensities among VPOs- M_sM_s , VPOs- R_sR_s , VPOs- R_sM_s , and VPOs- M_sR_s distinctly revealed the impact of direct microwave irradiation on the synthesis process. Notably, during stage 1 synthesis, direct microwave irradiation could induce the formation of β -VOPO₄ and α_{II} -VOPO₄. The phase composition of VPOs- M_sM_s was characterized by prominent V^{4+} and V^{5+} peaks, underscoring the enhanced catalytic performances attributed to the direct microwave irradiation synthesis. This correlation was further substantiated by the findings from the TPR in H_2/N_2 analyses and catalytic tests presented of this study.

The formula to calculate the crystallite sizes of (0 2 0) and (2 0 4) were given by Debye-Scherrer equation:

$$t = \frac{0.89\lambda}{FWHM \times \cos\theta}$$

where t is crystallite size for (h k l) phase, λ is X-ray wavelength of radiation for Cu K α , FWHM is Full Width at Half Maximum of the characteristic peak and θ is the diffraction angle for the phase. According to Debye-Scherrer's equation, the crystallite size is inversely proportional to the FWHM of the reflection plane [20].

The determination of crystallite size in VPOs catalysts was carried out by analysing the Full Width at Half Maximum (FWHM) of the (0 2 0) and (2 0 4) reflection planes using X-ray diffraction (XRD) [21]. The resulting crystallite sizes for these planes across all VPOs catalysts were calculated and presented in Table 1.

Interestingly, the adoption of direct microwave irradiation synthesis during the fabrication of VPOs catalysts yielded bigger crystallite sizes along the (0 2 0) and (2 0 4) reflection planes, in contrast to catalysts prepared through conventional reflux synthesis. This contrast has emphasized the impact of microwave irradiation in fostering a more pronounced surface morphology and contributed to an increase in the specific surface area of the catalysts.

Brunauer-Emmett-Teller (BET) Surface Area Measurements and Chemical Analyses

The specific surface areas of VPOs-M_sM_s, VPOs-R_sR_s, VPOs-R_sM_s and VPOs-M_sR_s were 24.38 m²g⁻¹, 18.01 m²g⁻¹, 17.52 m²g⁻¹ and 21.02 m²g⁻¹, respectively (Table 2). It could be observed that VPOs catalyst synthesised by direct microwave irradiation (VPOs-M_sM_s) exhibited 35.4% higher specific surface area as compared to VPOs catalyst synthesised via conventional reflux counterpart (VPOs-R_sR_s). Similar observation was also reported by Rownaghi and co-workers in 2009, whereby VPO catalyst prepared via VPD route involving microwave irradiation treatment exhibited higher specific surface area as compared to VPO catalyst prepared solely on reflux method [14,15]. Another crucial observation on VPOs catalysts prepared via direct microwave irradiation on Stage 1 (VPOs-M_sR_s and VPOs-M_sM_s) was its potential to exhibit relatively higher specific surface area as compared to catalysts synthesised via reflux on Stage 1 counterparts (VPOs-R_sM_s and VPOs-R_sR_s).

The above observations indicated that the improvement on specific surface area of VPOs catalysts could only happen when the catalyst was prepared using direct microwave irradiation synthesis method. This could be rationalised that the cavitation effect from direct microwave irradiation could increase the porosity and modify the structural morphology of VPOs catalyst platelets, which eventually led to higher specific surface area.

Table 2 showed the P/V atomic ratio for all VPOs catalysts analysed by using ICP-OES and EDX analyses. The P/V atomic ratio influences the distribution of these oxidation states, affecting the catalyst's ability to cycle between different oxidation states during the reaction. The proper redox behaviour is necessary for maintaining catalytic activity over multiple reaction cycles. The P/V atomic ratio was observed to fall within the range of 1.01 to 1.29 which was quite close with the optimum P/V atomic ratio range in producing active and selective $(VO)_2P_2O_7$ phase, i.e. 1.0-1.2. The P/V atomic ratio above unity indicated that both direct microwave irradiation and reflux synthesis method would induce phosphorus surface enrichment. This phenomenon could enhance the catalyst's ability to facilitate specific chemical transformations and increase the selectivity towards the desired products [22, 23].

The percentages of V^{4+} and V^{5+} phases were determined by using redox titration method [20] and were tabulated in Table 2. VPOs- M_sM_s exhibited the highest average oxidation state of vanadium among all VPOs catalysts. This observation has aligned well with the XRD analyses, which exhibited higher intensities in V^{5+} phases in the VPOs catalysts synthesised via direct microwave irradiation in both stages of precursor synthesis.

Secondary electron images by SEM

The secondary electron images of VPOs- M_sM_s , VPOs- R_sR_s , VPOs- R_sM_s , and VPOs- M_sR_s catalysts (Figure 2) showed rosette-shaped clusters consisting of plate-like crystals with different shapes and sizes. These rosette-shaped clusters were deemed as the characteristic morphology of $(VO)_2P_2O_7$ catalysts that preferentially exposing the (1 0 0) reflection plane [23].

VPOs- R_sR_s showed the signature rosette shape clusters which were deemed as the characteristics of VPOs catalyst synthesised via sesquihydrate precursor [4, 8]. However, the application of direct microwave irradiation could produce VPOs catalyst with chrysanthemum needle-like morphology with sharp edges. This distinctive feature could only occur when the VPOs catalyst was synthesized via direct microwave irradiation on both stages of sesquihydrate precursor synthesis. This chrysanthemum needle-like morphology had significantly contributed to higher specific surface area of VPOs catalyst as compared to conventional reflux synthesis counterpart. Therefore, VPOs- M_sM_s showed to form thinner crystal platelets as compared to other VPOs catalysts which were in agreement with BET surface area analyses. The crystal growth is usually sensitive to the initial nucleation process. In conventional reflux method, crystals tend to nucleate on container walls or impurity particles, leading to a slow growth rate due to rather few nuclei. Therefore, when direct microwave irradiation was employed for the synthesis of VPOs

catalyst, the energy of microwave irradiation has led to rapid nucleation rate and induced the formation of massive nuclei throughout the bulk solution [24].

Temperature Programmed Reduction in H₂/N₂

Temperature Programmed Reduction (TPR) in H₂/N₂ analysis is conducted to explore the redox properties of VPO catalysts. Additional insight into the nature and availability of oxidizing species from VPOs catalysts (O⁻-V⁴⁺ and O²⁻-V⁵⁺) can be obtained. The TPR profiles for VPOs catalysts in H₂/N₂ stream (5% H₂ in N₂ with a flowrate of 25 cm³ min⁻¹ at 1 bar) using fresh catalysts, with the temperature being raised from ambient to approximately 1200 K at a rate of 5 K min⁻¹ in that stream, are depicted in Figure 3. The peak maxima temperatures, the quantity of removed oxygen in each peak, and the derived reduction activation energies are listed in Table 3.

Three reduction peaks could be observed in TPR profiles for all VPOs catalysts synthesised via sesquihydrate route and similar profiles could be observed in previous findings reported by Leong and co-workers in 2004 and 2012. The O⁻-V⁴⁺ oxygen species was deemed as highly active in VPO to transform *n*-butane to maleic anhydride, while the O²⁻-V⁵⁺ species would contribute to the selectivity of this reaction [4, 8].

VPOs-M_sM_s catalyst prepared via direct microwave-synthesised catalyst exhibited the removal of highest total amount of oxygen atoms associated to both V⁴⁺ (O⁻-V⁴⁺) and V⁵⁺ (O²⁻-V⁵⁺) phases, i.e. 3.69 x 10²¹ atom/g. This significant value was found to be equivalent to more than 4 times the total amount of oxygen atoms being removed from conventional reflux counterpart (VPOs-R_sR_s), i.e. 7.19 x 10²⁰ atom/g. These results were well-agreed with the XRD analyses whereby both V⁴⁺ and V⁵⁺ phases were found to be more prominent in VPOs-M_sM_s as compared to VPOs-R_sR_s. Furthermore, the formation of chrysanthemum needle-like morphology with the highest specific surface area had contributed the greatest oxygen mobility in the lattice of VPOs-M_sM_s as compared to other VPOs catalysts (VPOs-R_sM_s, VPOs-M_sR_s and VPOs-R_sR_s) in this study.

It could also be noticed that all VPOs catalysts prepared via direct microwave irradiation synthesis method either in Stage 1 (VPOs-M_sR_s) or Stage 2 (VPOs-R_sM_s) showed significant improvement in the amount of oxygen atoms removed from the VPOs catalysts as compared to the conventional reflux synthesis method (VPOs-R_sR_s). The total amounts of oxygen atoms being removed from VPOs-R_sM_s and VPOs-M_sR_s were 2.39 x 10²¹ atom/g and 2.98 x 10²¹ atom/g, which were more than 2 and 3 times higher than VPOs-R_sR_s catalyst, respectively. This phenomenon

has indicated that direct microwave irradiation synthesis method could produce VPO catalyst with greater catalytic performances and will be discussed in the next section.

***n*-Butane conversion, product selectivity and turnover number of VPOs catalysts**

The catalytic performances of the VPOs catalysts for *n*-butane oxidation to maleic anhydride (MA) were performed using a laboratory fixed-bed microreactor at 673 K, which is a standard operating temperature for VPO catalysts. The details of the catalytic performance data for all VPOs catalysts are presented in Table 4.

The activity to *n*-butane conversion for VPOs-M_sM_s, VPOs-R_sR_s, VPOs-R_sM_s and VPOs-M_sR_s were 34%, 16%, 19% and 23%, respectively, while the selectivity to maleic anhydride of the same series of VPOs catalysts were 95 %, 59 %, 88 % and 93 %, respectively. In comparison to VPOs-R_sR_s catalyst, the activity and selectivity of VPOs-M_sM_s were significantly improved by 18% and 36%, respectively. These results were in consistent with the results obtained in BET surface area measurements and TPR in H₂/N₂ analyses, whereby direct microwave irradiation could produce VPOs catalyst with 35.4% higher specific surface area and more than 4 times higher removable oxygen atoms as compared to VPOs-R_sR_s catalyst. Furthermore, the same phenomenon could also be observed on VPOs catalysts prepared via direct microwave irradiation synthesis method either in Stage 1 (VPOs-M_sR_s) or Stage 2 (VPOs-R_sM_s).

Besides that, VPOs-M_sM_s catalyst showed the highest Turnover Number (TON), i.e. 1.39, as compared to conventional reflux synthesis method (VPOs-R_sR_s), i.e. 0.89. VPOs-M_sR_s and VPOs-R_sM_s gave about the similar TON numbers of 1.09 and 1.08, respectively. These results had indicated that direct microwave irradiation synthesis method would generate VPOs catalyst with higher efficiency in butane oxidation process.

▪ CONCLUSION

Direct microwave irradiation showed a promising synthesis method in the preparation of VPOs catalyst (VPOs-M_sM_s) via sesquihydrate precursor. This synthesis method has produced VPOs catalyst with a distinctive secondary structure of chrysanthemum needle-like morphology, which could induce higher specific surface area and higher removable active and selective lattice oxygen as compared to conventional reflux synthesis method (VPOs-R_sR_s). As a result, VPOs-M_sM_s could exhibit the highest activity (34%), selectivity (95%) and TON (1.39) for the reaction to convert *n*-butane to maleic anhydride. In addition, the sesquihydrate precursor synthesis duration could be notably reduced from 48 hours to 4 hours through the application of direct microwave irradiation. Ultimately, the overall production cost of VPO catalysts will be lowered.

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