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V-Mo based catalysts for ods of diesel fuel. Part II. Catalytic performance and stability after redox cycles

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ABSTRACT

Vanadium-molybdenum oxides supported on alumina were prepared by successive wet-impregnation, in order to evaluate their activity in oxidative desulfurization (ODS) of sulfur compounds prevailing in diesel fuel. The oxidation states and reducibility of the catalysts were studied by means of reduction–oxidation cycles of temperature programmed, and chemical-quantitative analyses by permanganometric and ferrometric titrations, for fresh as well as used catalysts. Redox cycles were performed, involving TPR analyses separated by an oxidation treatment (TPO), which were carried out consecutively before or after ODS-batch cycles. ODS tests were carried out comparing oxidant performance of tert-butyl hydroperoxide and H_2O_2. TPR of the catalysts makes possible to have vanadium-oxides with different degree of reduction, which were correlated with ODS activity. According to these results vanadium-oxide species with 12.9% of reduction showed higher ODS performance.

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1. Introduction

Refineries around the world are facing the challenge of producing cleaner fuels [1,2]. To obey regulations in sulfur content is the primary focus for many years ago, the main goal is producing ultra-low-sulfur fuels (<10 ppm). Removal of sulfur-containing compounds is an important operation in petroleum refining. It is achieved by catalytic processes operated at elevated temperatures (>300 °C) and pressures (20–100 atm H_2) using Co-Mo/Al_2O_3 or Ni-Mo/Al_2O_3 catalysts [3]. Hydrodesulfurization (HDS) process is highly efficient for removing sulfur compounds, but it has problems with compounds like dibenzothiophene (DBT) and their alkyl derivatives. The least reactive derivatives are DBT with methyl groups at the 4- and 6-positions; as 4-MDBT and 4,6-DMDBT. Besides finding a new high-activity catalyst, several estimates have been made to see the necessities of HDS, showing an increase in reactor size or severe conditions of temperature and pressure [4,5]. These changes make HDS an inappropriate process to achieve the goal, although Nebula has been developed and described as a new catalyst able to solve the problem [6], it still needs high purity hydrogen. This and other reasons have not allowed the incorporation of this catalyst at refineries to solve the problem of getting low-sulfur fuels. In the meantime we have to search alternative technologies [2] such as the ODS process [7–26], a promising process where hydrogen is not used. In the ODS, DBTs compounds are easily oxidized under low temperature and pressure conditions to form the corresponding sulfones using an oxidant agent. The sulfones are highly polar compounds, easily separated from the fuel product by extraction [7]. Sulfur compounds such as disulfides are easy to hydrodesulfurize, but they are oxidized slowly. For this reason, ODS can be utilized as a second stage after HDS units, taking a low-sulfur-diesel (~500 ppm) down to ultra-low-sulfur-diesel levels.

In previous work [27], which refers to the use of spent HDS catalyst deactivated with deposited vanadium, V-Mo catalysts were evaluated in ODS reaction showing two factors that modify catalytic performance; water initially incorporated with the oxidant or produced by H_2O_2 decomposition, which produces catalytic deactivation, and reduced species of vanadium oxide, which improve ODS performance. This improvement is appreciated when TBHP is used as oxidant.

Vanadia and Molybdena have similar structure, both have three types of oxygen: oxygen type 1 when the atom is coordinated to one metal atom, oxygen type 2 when the atom is coordinated to two metal atoms and oxygen type 3 when this atom is coordinated to three atoms [28–30]. Each kind of oxygen atom can exhibit different catalytic properties [31,32]. These differences in activity and vanadium-oxides distribution (V_2O_3/VO_2/V_2O_5) in our system are important on sulfone yield as it was discussed in [27]. V_2O_3 showed better activity than VO_2 and V_2O_5, but between V_2O_3 and V_2O_5 there are a huge number of intermediary structures, which could show differences on ODS activity. Moreover, as in many

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oxidation reactions was observed, metal oxides as catalysts are more or less reduced due to activities and reaction conditions, i.e. vanadium oxide is reduced during the reaction [33]. V_2O_5 is catalytically inactive in the oxidation of hydrocarbons unless the preparation contains a certain amount of vanadium of oxidation state lower than +5 [34]. When V-Mo oxide catalysts were reduced, this reduction produced differences in ODS activity [27]. Therefore, the aim of this work is to determine the oxidation state of vanadium during the ODS reaction. In this way, vanadium-molybdenum based catalysts were tested in redox cycles (TPR-TPO-TPR), before and after ODS reaction, in order to understand the ODS performance of these catalysts.

2. Experimental

All compounds were purchased from Sigma–Aldrich and used without further treatment. Hexadecane (99.8%) was used as solvent of sulfur compounds: dibenzothiophene (DBT, 98%), 4-methyl dibenzothiophene (4-MDBT, 96%) and 4,6-dimethyl dibenzothiophene (4,6-DMDBT, 97%). Acetonitrile (99.9%) was used as extraction solvent. Hydrogen peroxide (30 wt.% H_2O_2, in water) and tert-butyl peroxide (70 wt.% TBHP, in water) were used as oxidant agents. Model diesel for ODS reaction (DBTs in hexadecane) or for intrinsic activity (DBTs in acetonitrile) was prepared with 610 S ppm: 220 of DBT, 200 of 4-MDBT and 190 of 4,6-DMDBT.

2.1. Catalyst preparation and characterization

Mo and/or V-oxides were supported on powder or pellets (gamma-alumina) of cylindrical shape, 6 mm height, 5 mm diameter and internal walls thickness of 200–250 μm as in [27]. Bimetallic (V-Mo/Al) and monometallic (V/Al, V/Ap and Mo/Al) catalysts on alumina were prepared by wet-impregnation of ammonium metavanadate and/or ammonium heptamolybdate solutions. V-Mo/Al was obtained by successive impregnation of Mo/Al, with intermediate and final calcination at 500 °C. Only V/Ap was prepared on alumina powder, and all the other catalysts were supported on alumina pellet.

Permanganometric titration and titration with iron (II) ammonium sulfate were used for the evaluation of vanadium species composition ($V^{5+}/V^{4+}/V^{3+}$) in the catalysts, after dissolving the samples in a small amount of H_2SO_4 (1:1) solution, as described in [35]. Chemical analysis was followed potentiometrically with ORP Redox Meter Hanna Instruments.

Catalysts were characterized by X-ray diffraction (XRD) using Siemens D500 powder diffractometer with Cu Kα radiation. Textural properties were obtained by N_2 adsorption–desorption isotherms of the samples, with a Tristar Micromeritics apparatus. Nitrogen physisorption isotherms were analyzed using the BJH method. Prior to the textural analysis, the samples were outgassed for 8 h in vacuum at 350 °C. Average pore size was evaluated from the desorption branch of the isotherm using the BJH model. Elemental composition was determined by SEM-EDX in a Jeol JSM-5900 LV microscope equipped with an energy-dispersive X-ray (EDX) elemental analysis system.

A conventional temperature programmed reduction (TPR) apparatus was used to study the reducibility of the catalysts as well as to get V species partially reduced with a degree of reduction perfectly controlled. Also, redox cycles (TPR-TPO-TPR) were achieved carrying out three analyses in sequence: the first reduction of the oxidized sample, an oxidation and finally a second reduction. TPR of the catalysts was performed using a flow of H_2/Ar mixture (30% H_2 (v/v), 25 cc/min) at atmospheric pressure, and a heating rate of 10 °C/min. from room temperature up to 700 °C. TPO analysis was carried out for the reduced samples, using an O_2/He mixture (15.0% O_2 (v/v), 25 cc/min) at the same TPR conditions. The oxidized sample was further reduced during a second TPR analysis, under the same experimental conditions described above.

2.2. Catalytic experiments

All ODS reactions were performed in a glass-batch reactor, immersed in a thermostatically controlled water bath to carry out the reactions at 60 °C, fitted with condenser, mechanical stirrer and a thermocouple. In a typical reactive cycle, equal volumes of diesel and solvent phase were added to the reactor, then oxidant agent and catalyst were introduced and a vigorous stirring started reaction time, this is a three-phase system (L–L–S). When using pellets, catalysts were placed in a fix point. An ODS reactive cycle lasted 60 min, then spent solutions (diesel and solvent) were removed and fresh solutions were added to perform a new batch-cycle. The catalysts were submitted to four ODS cycles, after each cycle, samples of diesel and solvent phases were withdrawn and injected (auto sampler) to the GC-FID after cooling at room temperature. GC-FID analyses were performed with an HP5890 Series II Gas Chromatograph with a PONA capillary column (Methyl Silicone Gum, 50 m × 0.2 mm × 0.5 μm film thickness). Reactant and product identifications were achieved by comparing retention times in GC-FID.

TBHP or H_2O_2 was used as oxidant with an initial O/S molar ratio of 13 and it was added to the reactor gradually in small doses to reduce thermal decomposition according to previous results [7,21]. Reactions with TBHP were measured during reaction by standard permanganometric titration and GC-FID. H_2O_2 content was measured by standard iodometric titration.

2.3. TPR- TPO-TPR experiments

A redox cycle was carried out for the catalysts, after or before of ODS cycles. Fig. 1 presents the process Diagrams 1, 2, and 3 (PD1, PD2 and PD3). PD1 and PD2 were achieved to obtain information about reducibility of V and/or Mo species and ODS activity in four reactive cycles. PD3 gives information about the stability on catalytic performance. In Fig. 1, a redox cycle is summarized as TPR-TPO-TPR. The catalysts tested in a redox cycle, which were evaluated afterwards in new ODS cycles, were labeled with an additional letter "R" (V/Al-R, V-Mo/Al-R or Mo/Al-R) because they would start a new ODS cycle as reduced species.

2.4. ODS with partially reduced catalysts

Experiments with partially reduced-vanadium species were performed to evaluate the appropriate oxidation states during ODS reaction. Controlled-reduced-vanadium species were obtained by means of TPR. First, V/Ap that initially contains V_2O_5 was partially or totally reduced to V_2O_3. After that, reduced catalysts were proved in ODS-two-phases system (L-S), using DBTs in acetonitrile as model diesel of extracted S-compound, and comparing oxidant agents with an O/S ratio of 4.

3. Results and discussion

First we will present the characterization results of the catalysts. Then, we are going to analyze the ODS activity of the catalysts during four reactive cycles before or after a redox cycle (TPR-TPO-TPR), according to process Diagrams 1, 2 and 3, to simulate the reactivation conditions during ODS reaction. Finally, we are going to study the totally or partially reduced V catalysts on intrinsic reactions in two-phase system.

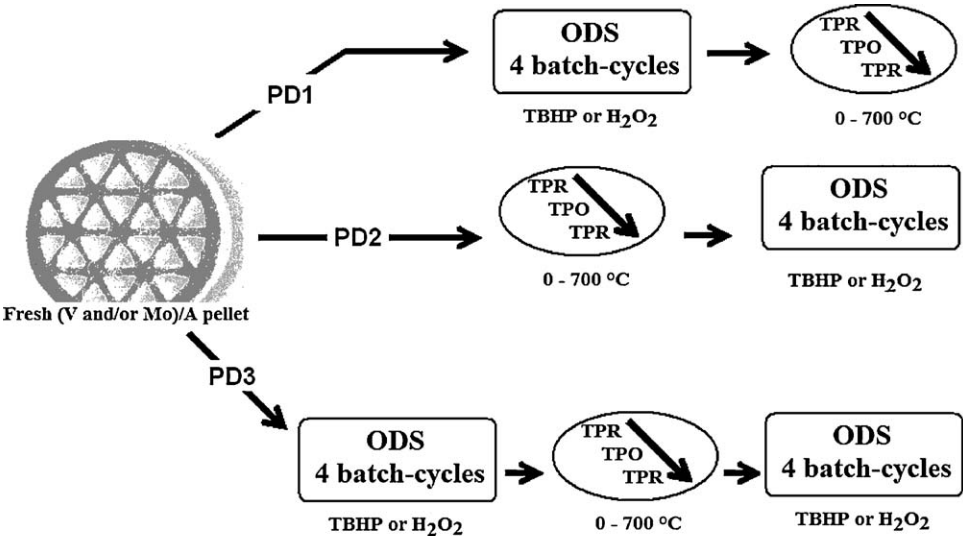

Fig. 1. Different process diagrams (PD1, PD2 or PD3).

3.1. Characterization of catalysts

Textural properties of the catalysts and support are presented in previous work [27], which shows that V and/or Mo-oxide deposition did not induce any remarkable decrease in these properties of the supported catalysts compared with alumina. Similarly, catalyst supported on alumina powder (V/Ap) did not show an important loss in textural properties with respect to the support. All catalysts, except V/Ap, showed main diffraction peaks of active phase: V_2O_5 (JCPDS 41-1426) or MoO_3 (JCPDS 89-5108) supported on gamma-Al_2O_3 (JCPDS 10-0425). Fig. 2 presents XRD patterns of V/Al, V/Ap and support, although catalysts were prepared with the same method, V/Ap has less vanadium load than V/Al (12 and 19 wt.%, respectively), this is the reason of the good dispersion of V species or absence of V_2O_5 crystals. Also, elemental mapping obtained by SEM-EDX showed a good dispersion of oxides on support surface.

3.2. ODS and redox cycles in PD1 and PD2

Fig. 3 presents initial and final distributions of vanadium species in V/Al, using H_2O_2 or TBHP. When catalyst has been used in PD1, with both oxidants, V_2O_3 is the main phase expected after process, if V species were well reduced in redox cycle. But only 84.8 (with H_2O_2) and 87.7% (with TBHP) were reduced to V_2O_3. These results suggest that during PD1, the oxidation state of vanadium changes significantly, creating species that have a strong interaction with the support being these species difficult to reduce in redox cycle.

During PD2, there were noticeable differences when catalysts were used in ODS after a redox cycle. Using H_2O_2 as oxidant, V^{4+} species are quantitatively greater than when TBHP was used. But in both cases (using H_2O_2 or TBHP), V^{4+} species were less than V^{3+} species. In contrast to PD1, during ODS V-oxides were partially reoxidized to V^{4+} species, since V species (after redox cycle) were well reduced or at least with similar level to that in PD1. In this case the amount of V-oxides reoxidized depends on the oxidant used, 37.5 and 23.3% of V^{4+} species were obtained using H_2O_2 and TBHP, respectively.

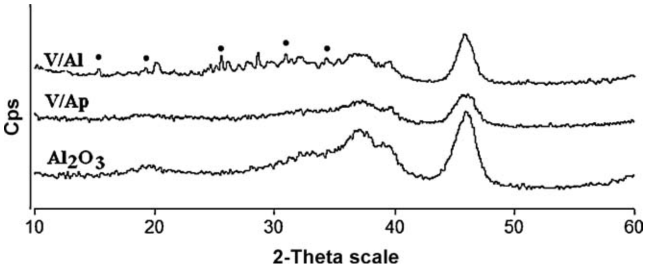

Fig. 2. XRD patterns of support and V-monometallic catalysts. (●) Main diffraction peaks of V_2O_5.

3.3. ODS and redox cycles in PD3

As it was presented in PD1 and PD2, during ODS reactions, there are important changes in predominance of vanadium species (V^{5+}/V^{4+}/V^{3+}) in presence of different oxidant agents. With the aim of knowing the vanadium-oxide distribution in the catalysts after ODS cycles and a redox cycle, chemical analysis of catalysts was carried out after each stage of PD3.

In Figs. 4 and 5 a comparison can be made between vanadium-oxides distribution in V/Al (Fig. 4) and V-Mo/Al (Fig. 5), in presence of each oxidant agent. Fig. 4 shows that V/Al has mainly V_2O_5 (93.1%) but, after first ODS reactions in which TBHP was used V^{3+} species are predominant (61.7%) and after the second ODS cycle V/Al contains 72.0% of V^{3+} species, whereas with H_2O_2 there are 16.4 and 55.6% of V^{3+} after the first and second ODS cycles, respectively. When H_2O_2 was used in the second ODS cycle (after redox cycle) V^{3+} species are oxidized close to 34% (from 84.8 to 55.6% of V^{3+}) and TBHP modifies less reduced-vanadium species, only 18% (from 87.7 to 72.0% of V^{3+}). Then TBHP modifies strongly oxidized V species and weakly reduced V species whereas H_2O_2 modifies strongly reduced V species and weakly oxidized V species.

Chemical analyses of V-Mo/Al during PD3 were tested in order to evaluate Mo effect on V/Al, Fig. 5 shows these results. In this case, only one TPR was carried out (not TPR-TPO-TPR as in PD1, PD2 and PD3 with V/Al) in order to avoid possible incorporations of metal atoms into alumina structure produced by severe temperature conditions. Results of V-oxides distribution in PD3 with V-Mo/Al are similar to PD3 with V/Al (Fig. 4). After the first ODS reaction with TBHP, V_2O_5 was appreciably reduced to V^{3+} species, in contrast with H_2O_2 where more V_2O_5 prevails. After TPR the catalysts were reduced to similar-vanadium distribution; independently of the oxidant used in previous ODS cycles. After the second stage of ODS cycles, vanadium-oxide distribution are comparable to PD3 with V/Al, therefore the reduced V species are modified strongly by H_2O_2 and weakly by TBHP.

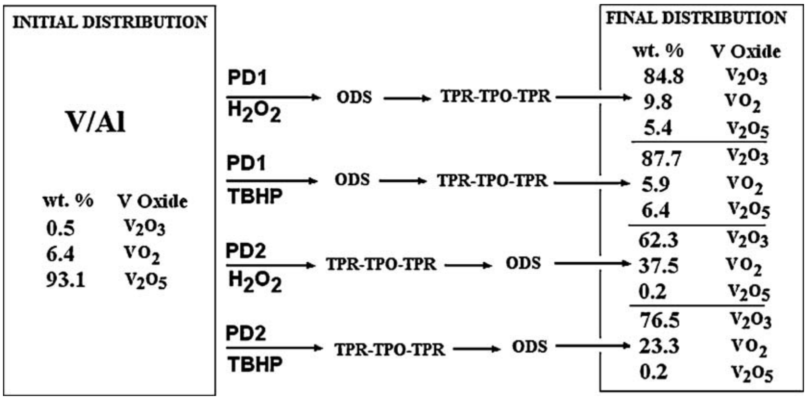

Fig. 3. V-oxides distribution in V/Al for PD1 and PD2, with different oxidant agent.

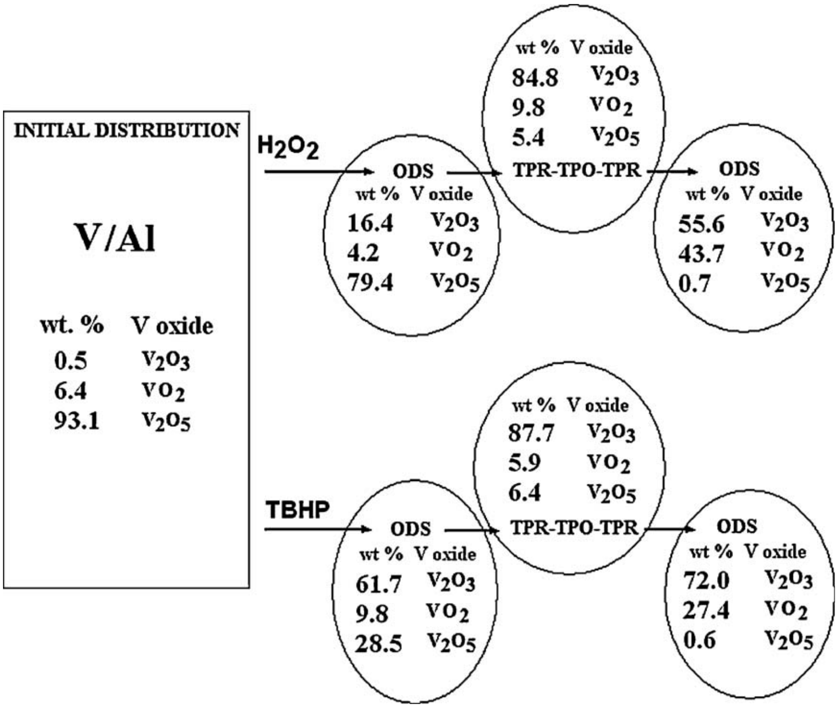

Fig. 4. V-oxides distribution in V/Al after each stage in PD3, with different oxidant agent.

Fig. 6 shows sulfone yield of DBTs obtained during four batch cycles with TBHP and oxidized catalysts (V/Al, Mo/Al and V-Mo/Al). These catalysts were also reduced (V/Al-R and Mo/Al-R and Mo/Al-R) according to PD3 and were carried out in other four batch cycles and these results are showed in Fig. 6. It is important to mention that ODS trends in PD1 and PD2 (not shown) are similar to ODS trends presented in [27], where during ODS-batch cycles with H_2O_2 activity decreases while with TBHP sulfone yield increases. In this case, ODS cycle results show a noticeable increase in sulfone yield, when TBHP is used, while with H_2O_2 the sulfone yield decreases (not shown). These trends were obtained with oxidized and reduced catalysts. Reduced-monometallic (V/Al-R and Mo/Al-R) or reduced-bimetallic catalysts (V-Mo/Al-R) increase sulfone yield, but they are affected when V-Mo form not active species due to interaction with alumina structure [27].

A comparison of the activities of oxidized catalysts (bimetallic and monometallic), suggests that the metal oxides act independently without synergistic interactions. Bimetallic catalyst presents lower activity than Mo/Al, which suggests an inhibiting effect of the Mo on V catalyst. Possibly due to Mo-alumina species, which were observed by XRD and SEM-EDX in V-Mo/Al and were not observed in Mo/Al. This activity performance of bimetallic and monometallic catalysts is according to obtained in ODS of DBT in kerosene using similar catalyst formulations [18].

During four ODS cycles, reduced catalysts have better performance than oxidized catalysts, but in reaction conditions vanadium-oxide species are partially reduced and oxidized, as previously was discussed. During each ODS cycle the sulfone yield increases, which indicates the contribution of a mixture of V species that is more active than V_2O_3, VO_2 or V_2O_5 alone. An optimal composition of these V-oxide species could be the reason to improve sulfones yield when TBHP is used. In Section 3.4 we will test the partially reduced V/Ap catalyst on ODS reaction, searching an optimal V species mixture.

3.4. Partially reduced catalysts by TPR and their intrinsic activity

The process of partial reduction of V/Ap was made as follows: first, a TPR pattern of V/Ap was carried out up to total reduction.

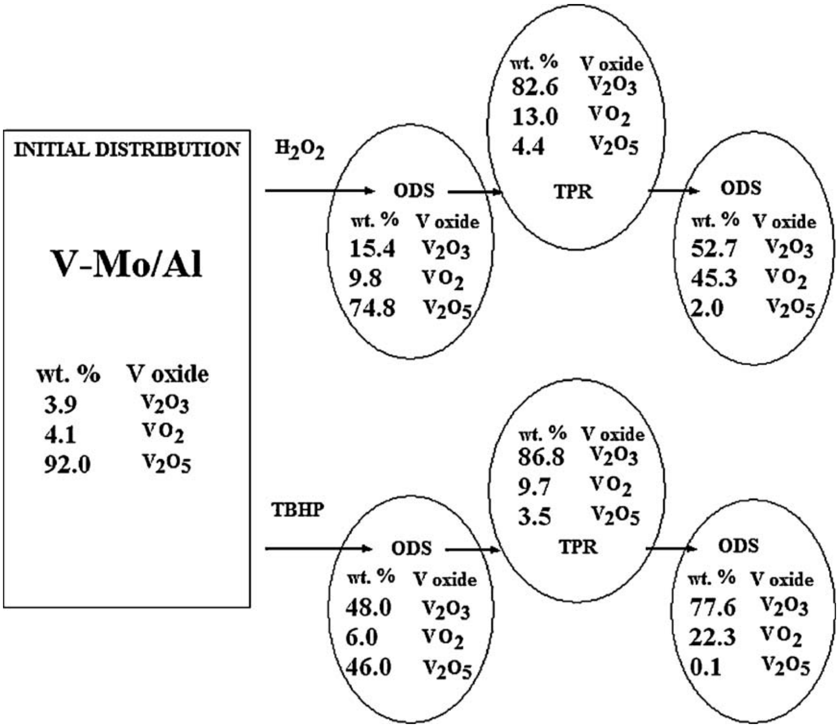

Fig. 5. V-oxides distribution in V-Mo/Al after each stage in PD3, with different oxidant agent.

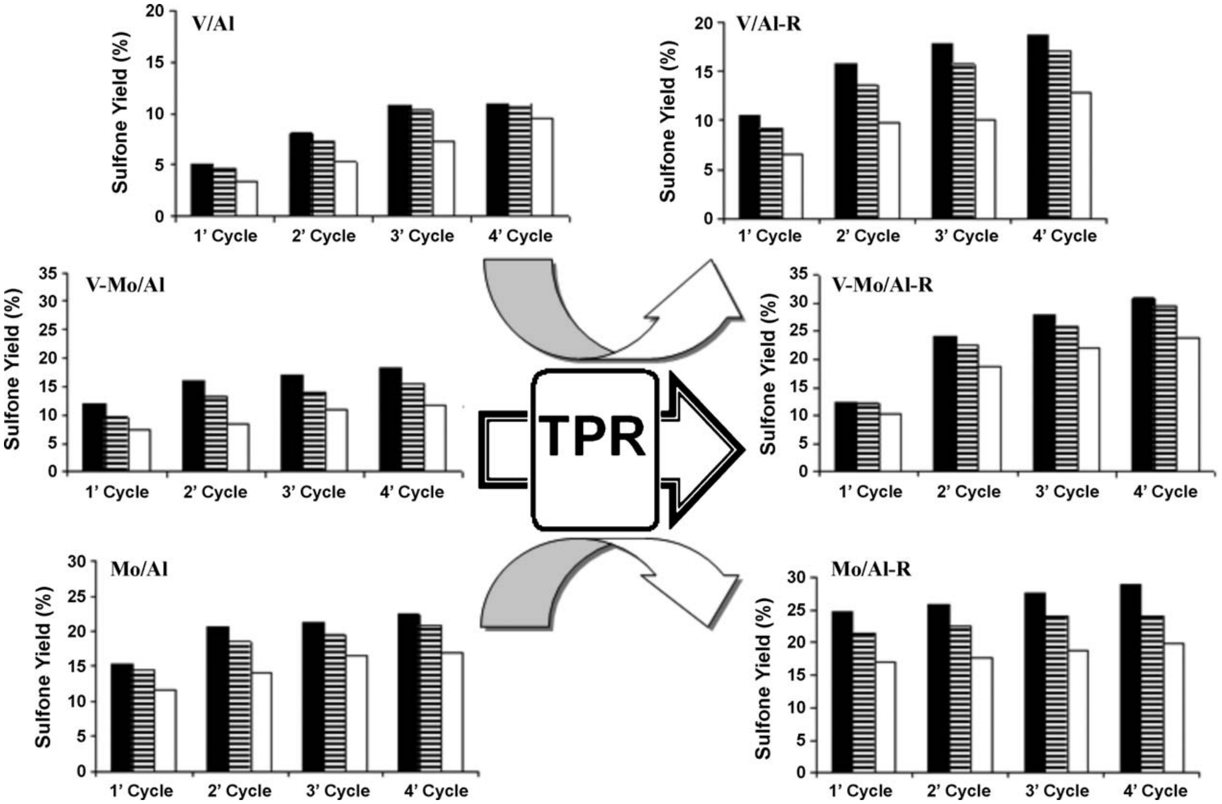

Fig. 6. Sulfone Yield of DBT (■), 4-MDBT (—) and 4,6-DMDBT (□), in four ODS cycles using TBHP as oxidant.

Second, partial reduction of fresh V/Ap (quenching at different temperatures, together with switching of control valve of H_2/Ar and Ar) was carried out to obtain several reduction degrees as showed in Fig. 7. The heating was stopped and cooled slowly from switch-temperature (ST) to room temperature, with the aim of stopping structural changes or mobility on species due to temperature. Reduction degree was obtained comparing TPR areas between total reduced V/Ap area and that of catalyst partially reduced. Fig. 7 shows partial reductions of V/Ap catalyst, their ST and reduction degree.

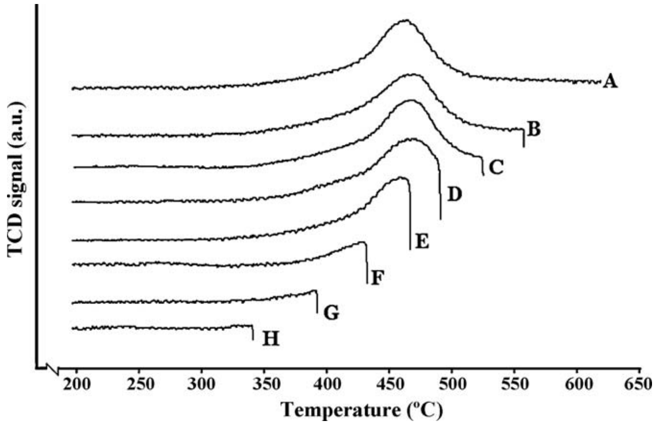

Fig. 7. TPR patterns of V/Ap catalyst up to different switch temperatures: (A) 700 °C —100% reduced, (B) 550 °C —97.1% reduced, (C) 520 °C —93.9% reduced, (D) 495 °C —90.9% reduced, (E) 460 °C —71.2% reduced, (F) 430 °C —39.3% reduced, (G) 380 °C —12.9% reduced and (H) 330 °C —5.3% reduced.

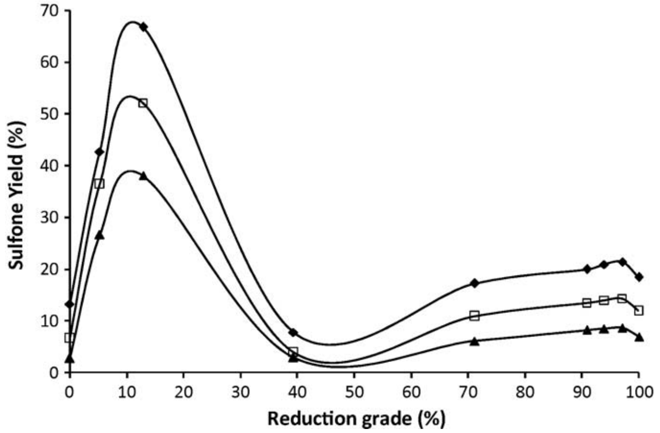

Fig. 8. Yield to (◆) DBT-sulfone, (☐) 4-MDBT-sulfone and (▲) 4,6-DMDBT-sulfone, in function of reduction grade of V/Ap catalyst, using H_2O_2.

All samples with different ST (700, 550, 510, 495, 460, 430, 380 and 330 °C) and original V/Ap (with 91.9% of V^{5+} and 5.6% of V^{4+}) were carried out in an ODS-batch reaction for 60 min at the same conditions previously explained.

Figs. 8 and 9 show the results of sulfone yield of DBTs obtained with V/Ap with different reduction degrees, using H_2O_2 (Fig. 8) or TBHP (Fig. 9). In both cases two maxima were obtained in sulfones yield, near to the catalyst with V^{5+} or V^{3+} (ST = 380 and 520/550 °C, respectively) and the sulfones yield has a minimum in ST between 430 and 450 °C. For both oxidants the behavior is similar showing the highest activity when V/Ap was reduced 12.9%. The original catalyst shows lower ODS activities: using H_2O_2 the sulfones yields were 13.2, 6.7 and 2.8% for DBT-sulfone, 4-MDBT-sulfone and 4,6-DMDBT, respectively, whereas with TBHP sulfones yields were 10.8, 4.7 and 1.3%, respectively. Results comparing V/Ap and totally reduced V/Ap are in agreement with ODS activities showed in [27], reduced V species are more active than oxidized V species, but being 12.9% the optimum degree of reduction for ODS reaction.

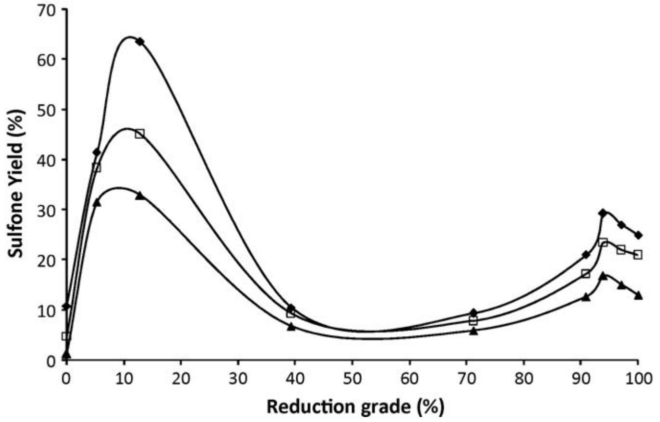

Fig. 9. Yield to (◆) DBT-sulfone, (☐) 4-MDBT-sulfone and (▲) 4,6-DMDBT-sulfone, in function of reduction grade of V/Ap catalyst, using TBHP.

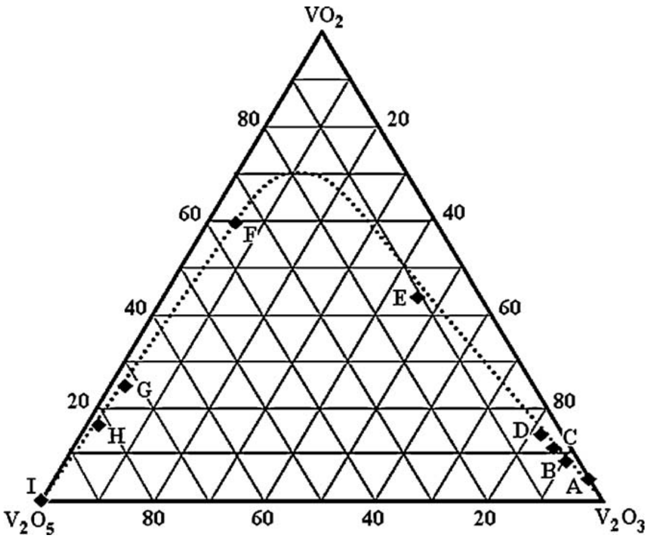

Fig. 10. V-oxides distribution in V/Ap with different degrees of reduction. A, B, . . ., and H are the reduced catalysts (see Fig. 7) and I is the fresh catalyst.

Chemical analyses of V/Ap after TPR with different ST were tested and the results are shown on a triangular diagram (Fig. 10). Vanadium-oxides distribution, obtained during partial reduction of V/Ap, shows a maximum in VO_2 according to a consecutive-reaction scheme where V_2O_3 is the final product, but these results show that V_2O_5 is not totally reduced.

The optimal catalyst (reduced in 12.9%) has an average-oxidation state of 4.7, which would be represented as V_3O_7 with an average-oxidation state of 4.67, V_4O_9 with an oxidation state of 4.5 or V_6O_{13} with an oxidation state of 4.33. This catalyst, with a small amount of reduced vanadium, shows an activity five times higher with respect to the totally oxidized catalyst.

The ability of vanadium atoms to possess multiple stable oxidation states results in the easy conversion between oxides of different stoichiometry by oxidation or reduction and is believed to be an important factor for the oxide to function as catalyst in selective oxidation [28,36]. This property of V-oxides would be improved in order to design catalysts with better performance for ODS of fuels with ultra-low-sulfur content.

4. Conclusions

Reducibility of bimetallic and monometallic catalysts, of Mo and/or V-oxides on powder or pellets of gamma-alumina, was evaluated by a redox cycle (TPR-TPO-TPR). Then, the oxidized or reduced catalysts were tested in ODS-batch cycles of a model diesel containing sulfur compounds prevailing in diesel fuel, as DBT, 4-MDBT and 4,6-DMDBT. During ODS cycles, significant changes occur in the predominance of vanadium species ($V^{5+}/V^{4+}/V^{3+}$) in presence of different oxidant agents. Using TBHP, the vanadium-oxidation state changes creating reduced species more significantly than when H_2O_2 is used.

Results of V-oxides distribution in V-Mo/Al obtained by chemical analysis are similar to V/Al catalyst. After the first ODS cycles with TBHP, V_2O_5 was appreciably reduced to V^{3+} species, in contrast to H_2O_2 where more V_2O_5 prevails. After the second stage of ODS cycles, vanadium-oxide distribution in V-Mo/Al is comparable to V/Al, the reduced V species are modified strongly by H_2O_2 and weakly by TBHP. This V-oxide distribution in the catalysts leads to different ODS performance, which depends on the V-oxide species present on the catalyst. The difference in the oxidation state of vanadium with H_2O_2 and TBHP is related to the ability to reoxidize reduced species. Low ability to reoxidize and high ability to reduce the catalyst with TBHP is the reason of the increase in sulfones yield during ODS cycles. Although reduced species of V-oxides are more active than oxidized species, it is necessary that V-oxide species on the catalysts contain a certain amount of vanadium of oxidation state lower than +5, in order to improve ODS performance. ODS activities of V-oxides on powder alumina with different reduction degrees show a maximum in sulfone yield of DBTs for the catalyst reduced up to 12.9%. This catalyst has an average-oxidation state of 4.7.

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