

Review

V-Containing Mixed Oxide Catalysts for Reduction–Oxidation-Based Reactions with Environmental Applications: A Short Review

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Abstract: V-containing mixed oxide catalytic materials are well known as active for partial oxidation reactions. Oxidation reactions are used in industrial chemistry and for the abatement of pollutants. An analysis of the literature in this field during the past few years shows a clear increase in the use of vanadium-based materials as catalysts for environmental applications. The present contribution makes a brief revision of the main applications of vanadium containing mixed oxides in environmental catalysis, analyzing the properties that present the catalysts with a better behavior that, in most cases, is related with the stabilization of reduced vanadium species (as V^{4+}/V^{3+}) during reaction.

Keywords: vanadium oxide; VO_x/TiO_2 ; SCR; H_2S ; VOCs; redox; partial oxidation

1. Introduction: Main Applications of Vanadium Oxide-Based Materials in Catalysis

The chemical reactions that involve a change in the oxidation state of products (oxidations and reductions) are of great importance in industrial chemistry and environmental engineering, since most degradation reactions of pollutants involve oxidation or reduction. To catalyze these reactions, the catalytic material must have redox sites able to reduce/oxidize the starting molecule.

Most partial oxidation reactions take place through the Mars-van-Krevelen mechanism, in which the lattice oxygen of the oxide catalysts is incorporated to the oxidation product [1]; then, such a reduced site is reoxidized by the molecular oxygen reactant, restoring the original catalyst state. Experimental evidence of such a mechanism has been found for different oxidation reactions and several oxide catalysts [2]. Effective catalysts must exchange their lattice oxygen during reaction; they must have dynamic structures capable of changing the oxidation state during the reaction cycles. The vanadium oxides are able to be with oxidation states V, IV, and III, and to interconvert with relative ease, which makes them effective oxidation catalysts [3]. However, not all redox reactions of environmental interest operate on this mechanism.

The most studied catalytic systems for partial oxidation reactions are those containing vanadium [4]. VO_x -based catalysts are efficient oxidation catalysts due to their redox properties, and during the past few decades several studies have focused on the development of mixed-oxide catalytic systems containing vanadium; in this manner, the use of another compound (dopant, component, or support) modulates the activity of the catalysts. Thus, there are several well-known mixed-oxide vanadium-containing catalytic systems that have been developed during the past few decades: For example, the VPO (Vanadium-Phosphorous-Oxides) catalyst [5–9], which is commercially used for the transformation of n-butane into maleic anhydride; Vanadium-Aimony Oxides ($VSbO$) [10–14], which is used mainly for ammoxidation reactions; or the Mo–V multioxide system [15–20], used for propane transformation into acrylic acid. Figure 1 illustrates an analysis of the literature on vanadium oxide catalysts, underlining their main uses and their evolution during the last

few decades. Figure 1A plots the total number of papers when the words “vanadium oxide catalysts”. The total number of papers published per year has remained fairly stable in the last 20 years; however, the number of these papers that also include the word “environmental” is increasing. The percentage of “environmental” with respect to the total number per year is also shown in Figure 1A; it increases continuously, from 1–5% in 1988–1991 to more than 60% during the last three years (from 2015). A deep analysis of these papers shows how, in the 1990s, most studies focused on the development of VOx-supported catalysts for partial oxidation and ammoxidation reactions; thus, they were mainly focused on chemical reactions for the production of chemical intermediates with an interest in petrochemical industry (such as butane to maleic anhydride [21], toluene to benzonitrile [22,23], methane to formaldehyde [24], propane to acrylonitrile [10,11], and alkane oxydehydrogenation (ODH) [25], among others). It is observed, therefore, in the study of the historical evolution of the bibliography as in the eighties–nineties that the interest was in the synthesis of molecules and, although these applications are still being developed, the interest in the applications in environmental catalysis is increasing. Figure 1B illustrates the main environmental applications in which these materials are used, based on the number of papers that, in addition to “Vanadium Oxide Catalysts”, also contain “SCR”, “photocatalysis”, and “total oxidation”. These data reveal that SCR (selective catalytic reduction) is the most studied environmental process in which vanadium-based catalysts have been explored.

Figure 1 clearly accounts for the significant number of review papers that focus on vanadium oxide catalytic-based materials for alkane partial (am)oxidations, including oxidative dehydrogenations [26–30]. Figure 1 also illustrates that many environmental applications have been developed with VOx catalytic materials during the past twenty years. Thus, the objective of this contribution is to review the main environmental catalytic applications for vanadium oxide catalytic materials.

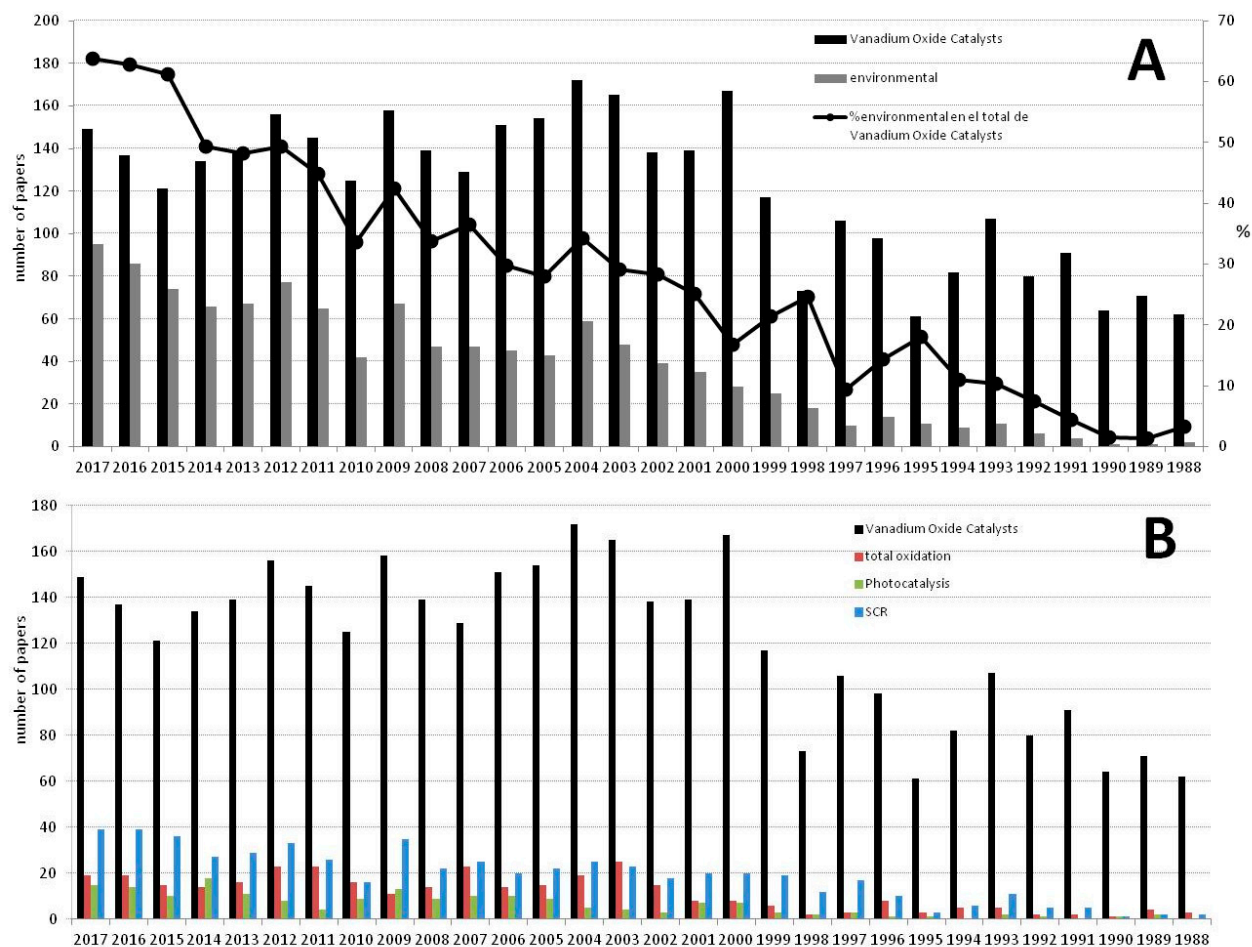
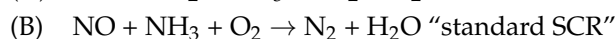
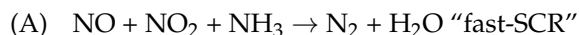


Figure 1. (A) Number of articles published by year during the last 10 years with “vanadium oxide catalysts” (black bars) and, among these, those that have “environmental” (grey bars). The lines indicate the % that grey bars represent with respect to the black ones. Note: Words in title, abstract, or keywords according to Scopus. (B) Number of articles published by year during the last 10 years with “vanadium oxide catalysts” (black bars) and, among these, those devoted to total oxidation (red), photocatalysis (green), and SCR (selective catalytic reduction) (blue).

2. Removal of NO_x

One of the most important environmental issues that catalysis engineering must face is the abatement of harmful compounds, such as oxides of nitrogen (NO_x), from both stationary and mobile sources. During the last few years, more efforts have focused on the development of catalytic converter technologies for diesel-powered vehicle engines that operate at high air/fuel ratios. Several techniques seem feasible for NO_x abatement, one of the most promising being ammonia- or urea-selective catalytic reduction (SCR) [31].

Several catalytic systems have shown to be promising for this process, with active phases such as Cu- and Fe-based catalysts [31–33] or Mn–Ce–O mixed oxides [34,35]; V-based catalysts are also very promising [36]. CeO₂- and TiO₂-supported vanadia catalysts have been extensively studied for NO_x removal. Forzatti's group investigated V–Mo–O/TiO₂ catalysts [37] and they reported that the ternary V–Mo–O/TiO₂ were more active in the SCR reaction at low temperatures than the corresponding binary VO_x/TiO₂ and MoO_x/TiO₂ at a given metal oxide loading. They described that the simultaneous presence of V and Mo enhances the redox properties of the catalysts and, thus, their reactivity. The improved catalytic properties of Mo–V–O oxides have also been reported for partial oxidation reactions, such as the dehydrogenation of propane [38–40], propane ammoxidation to acrylonitrile [41–43], or the selective propane oxidation into acrylic acid [44,45]. It has been demonstrated that molybdenum favors the presence of reduced vanadium V⁴⁺ species, which are stabilized in a rutile-like structure that is responsible for the redox cycle [46]. Temperature Programmed Reduction (TPR) experiments [37] show that the addition of Mo to V/TiO₂ favors vanadium reduction, with Mo–V–O catalysts being more easily reduced with respect to the corresponding binary supported samples and their bulk counterparts. Similar results were found with W: The V–W–O/TiO₂ catalysts presented a higher SCR performance than VO_x/TiO₂ and WO_x/TiO₂ catalytic systems [47,48], since tungsten sites improve the reducibility and formation of reduced vanadium species [49]. Thus, both Mo and/or W enhance the reduction of VO_x species, this V⁴⁺/V³⁺ species being the active sites for NO₂ formation [50]. NO oxidation to NO₂ is important to the SCR reaction at low temperatures due to the so-called “fast-SCR” and “standard SCR”, according to Reactions (A) and (B), respectively [49]:



Nova et al. studied the mechanism of the fast-SCR over V-containing catalysts using transient conditions [51]. They described that the first step was the NO₂ dimerization and disproportion to nitric and nitrous acid; then, adsorbed ammonia forms ammonium nitrite (which decomposes to N₂ and water) and ammonium nitrate (only if there is no suitable reducing agent). When NO is included in the feed, it can effectively reduce ammonium nitrate to ammonium nitrite, which could decompose to N₂; thus, complete selectivity to N₂ was achieved by adding NO to the NH₃/NO₂ mixtures. The same group also investigated the redox features in this mechanism over V-based catalysts [52]. It was shown that both NO₂ and HNO₃ reoxidize the V–W–O/TiO₂ catalysts at a much lower temperature than O₂; furthermore, they significantly enhance NO + NH₃ reactivity below 250 °C via the buildup of adsorbed nitrates, which act as a reservoir of oxidizing agents. These features were not observed over a V-free WO₃/TiO₂ catalyst, pointing out the role of the vanadium redox properties, and can explain the higher efficiency of the “fast” SCR compared with the “standard” SCR. Thus, vanadium sites are the active (redox) species of these catalysts, which are reduced by the reaction between NO and NH₃ and then reoxidized either by oxygen (standard SCR) or by nitrates (fast SCR) [51,52]. Wokaun et al. also investigated this [53] through transient experiments and in situ Raman spectroscopy to study the reoxidation of the vanadium species on a W–V–O/TiO₂ sample. They found that the vanadium reduced species (V⁴⁺) formed during the reduction of NO with NH₃ are reoxidized faster by NO₂ than by oxygen, resulting in an increased reaction rate of the fast SCR reaction. Thus, reduced vanadium species appear as the most active sites and the use of a co-element (such as Mo and/or W) modulates vanadium oxide activity [54,55]. Conversely, basic elements, such as K or Ca, titrate

surface acidity and decrease the reducibility of vanadium oxide species, being detrimental to catalytic behavior [56–58]. Thus, the poisoning effects impinge both the surface acidity and reducibility of vanadium oxide species.

Carbon materials are convenient catalytic supports, since they provide a high surface area and mechanical and chemical stability; the structure of vanadium oxide species on carbons has been extensively described [59,60]. The structure found is similar to that on oxide supports and surface polymeric or isolated VOx species, depending on the coverage. Lazaro et al. [61–63] have reported the use of carbon-based monoliths as supports for vanadium and tungsten oxide catalysts for the SCR process. They found some advantages with respect to the conventional titania-supported catalysts, since carbon-supported ones were not deactivated by SO₂ [64]. In addition, the higher surface area of carbons with respect to TiO₂ enables to disperse a higher amount of active vanadium species per weight of support [59]. Following this approach, they proposed a novel synthesis procedure by the sulphation of the catalysts with a mixture of SO₂ + O₂ [65,66]. They found that the pre-sulphidating catalysts duplicate conversion values. Their work showed that both ammonia adsorption and activating sites were different in the sulphated catalysts; in addition, sulphated catalysts present sulphate groups located near vanadia sites, thus facilitating vanadium reoxidation during redox cycles. In addition, sulphate groups did not contribute to the gasification of the most labile carbon groups of support.

Literature shows the promoting effect of cerium, both as a dopant and as support for the V-based catalysts during the SCR reaction. Ge et al. [67] studied the effect of adding Ce to V–W–O/TiO₂ catalysts with low vanadium loadings. Cerium was present mainly as Ce³⁺ and was beneficial for the oxidation of NO to NO₂, accelerating the SCR reaction. Huang et al. [68] also described that Ce-containing VOx/TiO₂ and VMoOx/TiO₂ catalysts exhibited high resistance to H₂O and SO₂ poisoning at low temperatures; they reported that Ce sites led to a higher concentration of chemisorbed oxygen, minimizing nonselective oxidation of ammonia to NOx. Diffuse Reflectance Infrared Fourier Transform Spectroscopy (DRIFTS) measurements [69] revealed that Ce contributes to the formation of NO₂ and monodentate nitrate species, both being reactive intermediates for the SCR reaction; the characterization also indicated that the redox cycle in the presence of Ce ($V^{4+} + Ce^{4+} \leftrightarrow V^{5+} + Ce^{3+}$) accounts for an excellent SCR catalytic performance for the Ce-doped V-based catalysts.

3. Removal of H₂S

H₂S is a major contaminant produced in most energy-related industries, such as petroleum, natural gas, fuels, and fuel cells. H₂S causes acid rain. In addition, it leads to corrosion in pipelines and acts as a poison for many catalysts, even at low levels, making it necessary to remove it in many industrial processes, such as fuel cells applications. Conventional treatments, including adsorption, or biological treatments present disadvantages, making the transformation to a useful product the better option. In this sense, the selective oxidation of H₂S to elemental sulfur with air is the most attractive process, but it requires the design of partial oxidation catalysts facilitating oxidation to S and avoiding further oxidation to SO₂. V₂O₅ oxide was reported as the most active catalyst in a study performed with 21 metal oxides for both the Claus process and partial oxidation [70]. As well as in other partial oxidation processes that have already been presented, it is easier to modulate the activity with a mixed metal oxide and/or through the use of a support. López-Nieto and coworkers [71,72] proposed the use of a mesoporous zirconium phosphate as a support for VOx species, and they reported such catalysts as active and selective for the H₂S transformation to S, reporting that the catalytic behavior was dependent on VOx loading. They reported that V₂O₅ crystals were necessary in order to have active and selective catalysts, identifying this oxide as the active phase. However, the characterization results of tested catalysts have demonstrated the formation of a partially reduced crystalline phase, V₄O₉-like, during the catalytic test. This was then confirmed by carrying out the catalyst characterization during the reaction using operando Raman-GC and XAS [73]. They demonstrated that the V₂O₅ crystalline phase was present in fresh catalysts and was transformed to V₄O₉ under H₂S partial oxidation. Furthermore, they found that the reduced V species were related

with a low selectivity to SO_2 . Following a similar approach, other groups investigated the use of supported catalysts, such as VOx/TiO_2 [74], VOx/CeO_2 [75,76], and VOx/SiO_2 [77], and of vanadium oxides bronzes [78], which were the mechanism they found to be quite similar to the one already described and demonstrated by López-Nieto's group, with the oxidation and subsequent regeneration of the vanadium active phases, from V^{5+} to V^{4+} under reaction conditions.

4. Removal of N-Containing Organic Pollutants

Total oxidation of organic compounds is a major topic; however, there are only few studies on the removal of nitrogen-containing organic compounds (NCOs). The design of this process is not easy, since catalysts that oxidize the organic part to CO_2 without oxidizing the nitrogen component to molecular N_2 rather than to NO_x are challenging. Metal-based catalysts with Cu and Cr or Pt have been tested for this process, but in all cases quite a high NO_x selectivity was detected [79]. Vanadium-based catalysts are widely used for selective partial oxidation processes; our group reported their use for the selective destruction of N-containing organic volatile compounds [80]. Sb–V–O catalysts were selected for such studies since they are well known as active and selective for the propane ammoxidation process [14] and, subsequently, they are able to activate nitrogen atoms [81,82], avoiding further oxidation to NO_x under ammoxidation reaction conditions. Thus, the catalytic behavior of Sb–V–O-supported catalysts on the destruction of dimethyl formamide (DMF) was evaluated [80], since DMF is commonly used as industrial solvent in the production of polyurethane products and acrylic fibers. It is also used in the pharmaceutical industry, in the formulation of pesticides, and in the manufacture of synthetic leathers, fibers films, and surface coatings. Several Sb–V–O catalysts with different supports (Al_2O_3 and Nb_2O_5) and different Sb precursors were tested [80]. The activity results are shown in Figure 2 (DMF conversion vs. total Sb+V coverage) and 3 (selectivity to N-containing products vs. total Sb + V coverage). Figure 2 shows some interesting results regarding DMF conversion. Figure 2A shows the effect of coverage on alumina support, where it can be observed that bare alumina is quite active, but selective to NO_x (Figure 3); when Sb and V are incorporated to the alumina, the conversion increases to near 100% and the selectivity to NO_x decreases. Those catalysts in which rutile VSbO_4 phase forms were able to oxidize the N-containing organic compounds, avoiding the formation of NO_x . Bare niobia support exhibits, unlike alumina, a significant production of ammonia, due to its higher acidity. The addition of V and Sb on Nb_2O_5 support covers the acidic sites and provides redox functionality, increasing the selectivity to N_2 (Figure 3).

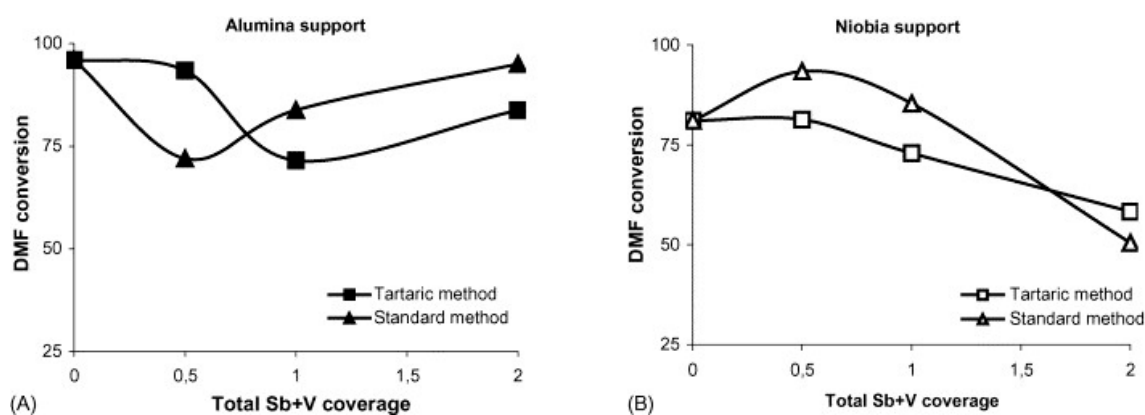


Figure 2. Dimethyl formamide (DMF) conversion values vs. total Sb + V coverage for: (A) Al_2O_3 and (B) Nb_2O_5 -supported catalysts. Reaction conditions: 1 mL of catalyst; GHSV = $10,000 \text{ h}^{-1}$; 1000 ppm of DMF; temperature reaction: 250°C . Reprinted from “Selective destruction of nitrogen-containing organic volatile compounds over Sb–V–O catalysts” *Appl. Catal. B Environ.* **2007**, 71, 85–93 [80]. Copyright ©2006 Elsevier B.V., with permission from Elsevier 4461960513380.

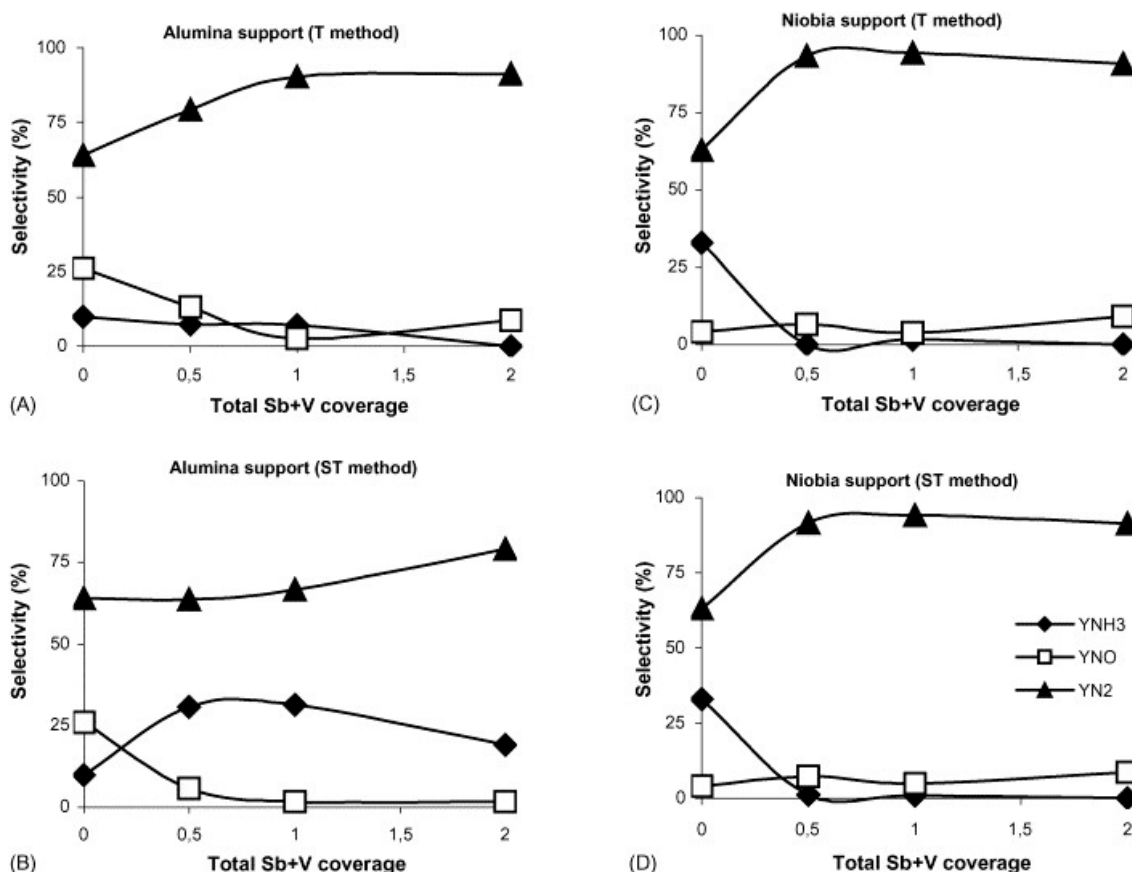


Figure 3. Selectivity to N-products vs. total Sb + V coverage at 400 °C for: (A) Tartaric (T) and (B) standard (ST) alumina-supported catalysts; (C) tartaric (T) and (D) standard (ST) Nb₂O₅-supported catalysts. Reaction conditions: 1 ml of catalyst; GHSV = 10,000 h⁻¹; 1000 ppm of DMF. Reprinted from “Selective destruction of nitrogen-containing organic volatile compounds over Sb–V–O catalysts” *Appl. Catal. B Environ.* **2007**, *71*, 85–93. Copyright ©2006 Elsevier B.V., with permission from Elsevier 4461960513380.

Figure 4 shows the conversion and selectivity profiles versus temperature at high Sb + V coverage on both Al₂O₃ and Nb₂O₅. Above 400 °C, the selectivity to N₂ decreases at the expense of the formation of NO_x. In addition, the selectivity to CO decreases with temperature, in this case at the expense of CO₂ formation. The catalysts with high coverage afford promising results; selectivities to NO_x and organics are low (at moderate temperatures), and the system could be used with a CO to CO₂ converter in a second bed. The presence of a rutile VSbO₄ active phase eliminates the overoxidation to NO_x and appears to be responsible for better selectivity on the alumina series. During the catalytic cycle, the surface V⁵⁺ species undergo reduction and they become stabilized as V³⁺/V⁴⁺ in the VSbO₄ structure [83,84]. The better selectivity to N-containing products was subsequently related to the presence of VSbO₄.

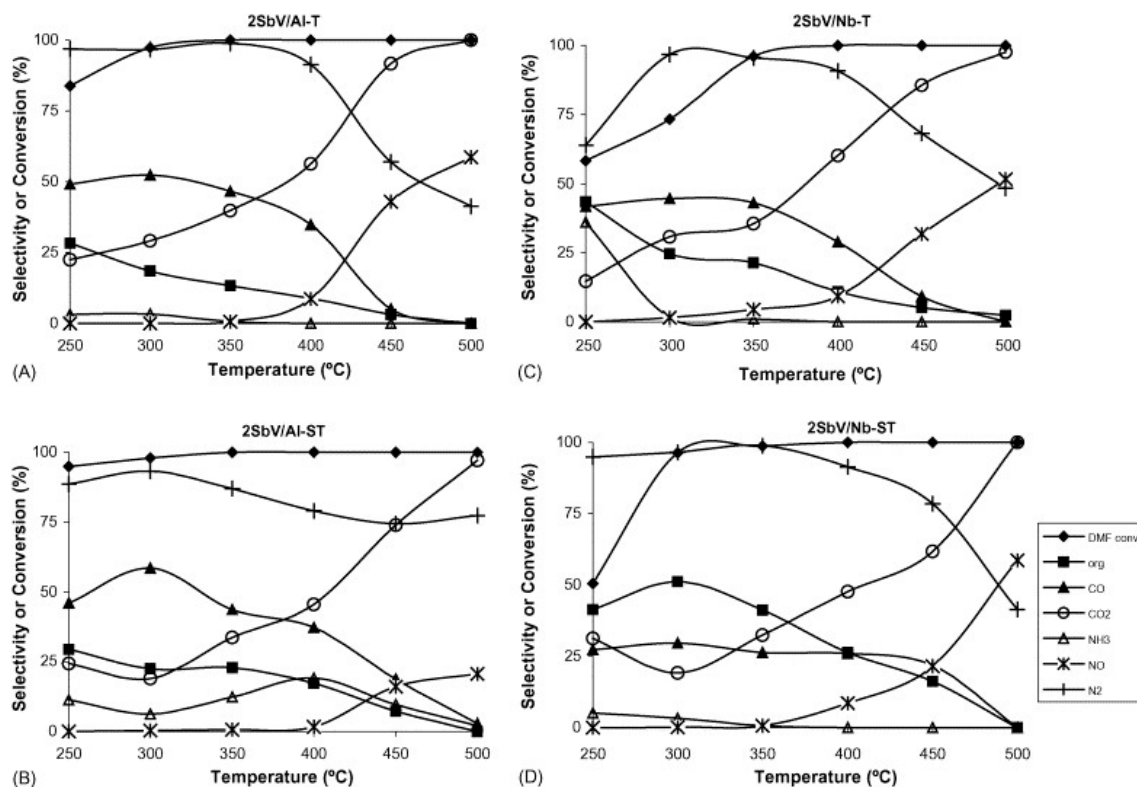


Figure 4. DMF conversion and selectivity profiles for principal reaction products vs. temperature for: (A) 2Sb₅V/Al-T; (B) 2Sb₅V/Al-ST; (C) 2Sb₅V/Nb-T; (D) 2Sb₅V/Nb-ST. Reaction conditions: 1 ml of catalyst; GHSV = 10,000 h⁻¹; 1000 ppm of DMF. Reprinted from “Selective destruction of nitrogen-containing organic volatile compounds over Sb–V–O catalysts” *Appl. Catal. B Environ.* **2007**, *71*, 85–93 [80]. Copyright ©2006 Elsevier B.V., with permission from Elsevier 4461960513380.

5. Total Oxidation Process

VOCs (Volatile Organic Compounds) are recognized as hazardous molecules that contribute to photochemical smog and are toxic, since some of them may cause cancer and other diseases. They present a high vapor pressure and low water solubility and can be found in the air both indoors (due to furniture, cleaners, pesticides, air fresheners, cosmetics, tobacco smoke, adhesives, flooring, carpets, varnishes, paint strippers, sealants, etc.) and outdoors (oil and gas extraction and processing, wood/coal combustion, diesel and gasoline emissions, etc.). The nature of these molecules depends on the source, including alkanes, alcohols, ketones, aldehydes, aromatics, paraffins, olefins, and halogenated hydrocarbons. Among the most common and toxic not halogenated compounds are phenol, acetone, formaldehyde, benzene, propylene, toluene, and styrene. There are a few techniques available to reduce VOC emission when they are highly diluted, catalytic complete or “deep” oxidation being one of the most suitable techniques [85]. Thermal incineration (nuncatalytic) requires higher temperatures, with the corresponding emissions of undesirable products, such as NO_x and dioxins, and it is a quite high-energy-demanding process. Thus, there are many studies that have focused on the best catalytic formulation for the VOCs’ catalytic total oxidation, which is not an easy task, due to the large variety of organic molecules present in VOC mixtures. Both noble metals and transition metal oxides have been reported as promising catalysts. Noble-metal-based catalysts [86], especially Pt and Pd, present very good activity performances, but they are quite expensive materials and can be deactivated by sintering or poisoning. Due to that, transition and rare earth metal oxide catalysts have been explored. Although V-based catalysts possess redox active sites, vanadium is not commonly used in these formulations; other transition metals, such as copper oxide, manganese dioxide, iron oxide, nickel oxide, chromium oxide, and cobalt oxide, are used instead [87].

V–W–O-supported catalysts, used for the SCR process as has been discussed, have also been studied for VOC oxidation [88] and have been reported as active for the abatement of several polychlorinated VOCs, showing that this catalytic system was useful for the combined reduction of NO_x and decomposition of these halogenated (dioxins), even in the presence of sulfur dioxide [89]. This is an important issue in this case, since SO₂ is usually present with VOC pollutants and it is well known that it is able to poison noble-metal-based catalysts. However, at low temperatures (150 °C), polychlorinated molecules remain adsorbed on the catalysts without being oxidized [88]. Similar results were reported over VO_x/TiO₂ catalysts [90,91]. The total oxidation of nonchlorinated VOCs over vanadium-supported catalysts have also been studied by Debecker and coworkers [92], who reported very promising results where oligomeric polyvanadates would be the active site. This group also reported [93] the promoting effect of W and Mo on VO_x/TiO₂ catalysts for the total oxidation reaction for both halogenated and nonhalogenated molecules, obtaining similar results to those already exposed for the SCR process. They attributed this synergy between V and Mo/W oxides to the additional Brønsted acid sites brought by these promoters, since such acid sites would favor the adsorption of the VOC molecule.

6. Photocatalysis

Photocatalysts use the energy of impinging photons and may carry out a wide variety of important chemical reactions, such as (i) environmental remediation, (ii) the destruction of organic pollutants for water or air purification, (iii) solar fuels production, (iv) production of fuels, (v) and H₂ from water or methane/methanol from CO₂, among other applications. TiO₂ [94] is commonly used as a photocatalyst since it is an available and safe semiconductor, which is stable in solution; only 5% of the incident Sun radiation can be used by TiO₂, due to its bandwidth (3.2 eV = 390 nm). Subsequently, other photocatalytic systems, including the use of doped TiO₂, in order to modify the bandwidth, have been extensively studied during the last few decades. Doping TiO₂ with transition metal ions, such as V, has been one of the most important approaches [95–98]; however, this increases carrier-recombination centers, decreasing the quantum efficiency of doped TiO₂ photocatalytic materials [95,96]. Another approach is doping with nonmetals, such as N [99]. Gu et al. [100] investigated the effect of doping TiO₂ nanocrystal catalysts with vanadium and nitrogen, reporting an enhanced photocatalytic activity for the co-doped catalytic materials compared to V-doped TiO₂ and N-doped TiO₂. Here [100], a co-doped TiO₂ nanocrystal had a narrower band gap (2.76 eV) than mono-doped TiO₂ (2.91 eV for V–TiO₂ and 2.92 eV for N–TiO₂). They demonstrated that the existence of V ions in the lattice facilitates the incorporation of N atoms into the anatase lattice, enhancing absorption in the visible light region and quantum efficiency.

Bismuth vanadate, BiVO₄, has also been extensively studied, since it is a semiconductor material with some properties that make it an attractive photocatalyst, such as low toxicity, low production cost, resistance to corrosion, high photostability, and narrow band gap (≈2.4 eV) with a good response to visible light. Recently, the synthesis methods and properties of these materials have been reviewed by Madhavan and coworkers [101], who showed how the photocatalytic efficiency of these materials could be improved by the control of the morphology and composition during synthesis. Nanostructured VO₂ dioxide is also a semiconductor material that, with the appropriate synthesis procedure, can have a band gap of ≈2.7 eV and that shows a promising photocatalytic activity for hydrogen production [102].

7. Conclusions

This review underlines the possibilities of vanadium compounds in redox catalysis, which stand on the possibility of vanadium oxides to have different oxidation states during reaction. Additives that foster phases with reduced vanadium sites during reaction enhance redox cycles, facilitating changes in oxidation states. These cycles also modify the capability of the catalysts to absorb/desorb reactants and other intermediates. Vanadium-based materials are very promising catalysts in partial

oxidation reactions, which are useful for industrial chemical processes (petroleumchemistry), as well as for environmental applications.

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