Modification of the Activity of Copper Phosphate in Conversion of Isopropanol by Impregnation of Vanadium Oxide

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Abstract: *In this paper we describe the synthesis and characterization of the acid–basic properties of catalysts containing varied amounts of vanadium supported on copper orthophosphate. The preparation of the copper orthophosphate was carried out using a co-precipitation method and the vanadium (2-12 wt. % of V_2O_5) was introduced by impregnation with a porous volume in several stages, followed by calcination under air at a different temperature. The techniques employed for characterization of solids were XRD, Nitrogen adsorption at -196°C and catalytic conversion of isopropanol at different reaction temperatures ranged between 180 and 400°C. The results revealed that the solids V/CuP consisted of copper phosphate $Cu_3(PO_4)_2$ as major phase together with V_2O_5 as minor phase. The degree of crystallinity of V_2O_5 increases with increasing the vanadium content. The specific area (S_{BET}) of the investigated system was found to slightly increase with the increase in V_2O_5 content. The catalytic activity in isopropanol conversion, which proceeds via dehydration yielding propene increases as a function of extent of loading.*

Keywords: *Copper orthophosphate CuP; Supported vanadium catalysts V/CuP; XRD; BET; Isopropanol decomposition.*

I. Introduction

Various supported vanadium oxide catalysts such as V_2O_5/Al_2O_3, V_2O_5/SiO_2, V_2O_5/TiO_2, V_2O_5/ZrO_2 and V_2O_5/CeO_2 are used in a wide range of industrial applications. In fact, the choice of the nature of the support depends on the reaction because the catalytic activity depends not only on the number of active sites but on their distribution on the surface and the content of the catalyst in V_2O_5. In particular, much attention has been given to the VO_x system, which has proved to be one of the most active and selective in the oxidative dehydrogenation (ODH) of other lower hydrocarbons [1–10]. So, it is well known that supported vanadium oxide is a promising catalyst for the ODH of n-butane. MgO supported vanadium was reported as a very selective catalyst in the oxidative dehydrogenation of propane and n-butane , while γ-Al_2O_3 supported vanadium catalyst was found to present a good selectivity to olefin products for ethane ODH but a poor selectivity in the ODH of n-butane. The acid–base character of the support explained this different behavior. Over MgO, a support has a basic properties, the interaction between vanadium species and the supports leads to the formation of vanadate compounds. In the case of more acid supports, such as SiO_2 or Al_2O_3 a weak interaction is expected leading to less dispersed vanadium species on the surface which, in turns favors the formation of V_2O_5 crystallites. Vanadia–based catalysts were used also in the SO_2 to SO_3 oxidation [11], a key step in the sulfuric acid synthesis process. Knowledge of the crystal structure of the respective phases is desirable, since it is a prerequisite to uncover structure– (re)activity relationships.

The transformation of isopropanol is a widely used test reaction for characterizing both the acidic and basic properties of solids [12–17]. The isopropanol reaction on solid catalysts occurs through two pathways: dehydration, which is generally catalyzed by the acidic sites or both acidic and basic sites, and dehydrogenation, which is catalyzed by basic sites.

Many different synthesis methods have been used in the preparation of supported vanadium catalysts: vapour phase grafting with $VOCl_3$ [18], Chemical vapor deposition with $VO(OC_2H_5)_3$ [19], non-aqueous impregnation with vanadium alkoxides [20], aqueous impregnation of vanadium oxalate [21,22], as well as dry impregnation with crystalline V_2O_5 [18,23]. In this work a set of copper orthophosphate supports modified by the addition of V_2O_5 in different content, have been synthesized by impregnation to porous volume. The techniques employed were XRD, adsorption–desorption of nitrogen and the catalytic activity of this solids was studied for the isopropanol reaction at 180–400°C. This reaction allows us to evaluate the acid–base properties of these catalysts.

II. Experimental

2.1. Solids synthesis

Copper orthophosphate (CuP) were synthesized upon evaporation to dryness from aqueous solutions of $Cu(NO_3)_2 .3H_2O$ and $(NH_4)_2HPO_4$. The resulting solids were dried for 12h at 110°C and were finally calcined in air at 450°C for 12h. A series of vanadium catalysts denoted V/CuP were prepared using impregnation method, over an CuP support. A mass of ammonium metavanadate NH_4VO_3 calculated, to yield the desired percentage of vanadium pentoxide on the support, was dissolved in a predetermined volume of distilled water. The impregnation catalysts V/CuP were dried at 110°C for 12h and calcined under air flow at 450°C during 10h.

2.2. Characterization techniques

The X-Ray diffraction (XRD) analysis was performed on the catalyst using Bruker-eco D8 Advance diffract meter with Cu-K$_\alpha$ radiation ($\lambda = 1.5418$Å). The 2θ were scanned from 5 to 70°.
The textural properties (i.e. specific area, pore volume) of samples were determined by nitrogen adsorption-desorption at -196°C on a 3Flex Version 3.01 system.

2.3. Catalytic test

Decomposition of isopropanol was carried out in a fixed-bed type reactor with a continuous flow system at atmospheric pressure. The catalyst (50 mg) was packed in a glass reactor placed in a furnace. Before carrying out such catalytic activity measurements, each catalyst sample was activated by heating in a stream of nitrogen for 2 h at 400°C then cooled to the catalytic reaction temperature. The reactant, 2-propanol, was diluted in nitrogen by bubbling the gas through the liquid reactant in saturator maintained at 6°C. The isopropanol decomposition was performed at a temperature between 180 and 400°C. The products were analysed by on line gas chromatography (FID) using a 10% 0 V–101 on Chromosorb-WHP (80/100 mesh) packed column (4m) maintained at 80°C. A blank test showed that insignificant thermal reaction developed in the absence of the catalyst.

III. Result And Discussion

3.1. XRD patterns

In a previous work we studied by XRD analysis the crystalline evolution of the CuP support during calcination [24]. Beyond 723 K, two crystalline materials, corresponding to $Cu_3(PO_4)_2$ and $Cu_2(PO_4)(OH)$ phases are be clearly evidenced. Progressively with the increase in temperature, diffraction lines of the copper orthophosphate are sharpen and the crystalline phase $Cu_2(PO_4)(OH)$ is converted into $Cu_3(PO_4)_2$ phase, indicating a better crystallization of $Cu_3(PO_4)_2$. As can be seen in Fig.1, the calcined solid CuP at 650°C consisted of the copper orthophosphate $Cu_3(PO_4)_2$ (PDF Index Name: Copper Phosphate ref. cod: 01-070-0494).

The solids impregnated with vanadium have been also studied. As it can be seen from Fig. 1 the calcination at 650°C of these solids lead not only to the previous phase identified for $Cu_3(PO_4)_2$ but also to a new diffraction lines at 2θ = 17°, 27° and 36°, have been assigned to crystalline phase V_2O_5 (ref. cod: 00-053-0538) which progressively increased with increasing of the vanadium load. This shows that vanadium pentoxide V_2O_5 is crystallized from low load over the surface of the copper phosphate.

3.2. Textural properties of the calcined solids

The nitrogen adsorption–desorption isotherms of the CuP support and those of representative V/CuP obtained by impregnation method are reported in the Fig. 2. All the solids exhibit a type IV isotherm corresponding to non-porous or macroporous materials. At low relative pressure formation of a monolayer of adsorbed molecules is the prevailing process, while at high relative pressure a multiplayer adsorption takes place: the adsorbate thickness progressively increases until condensation pressure has been reached, the pressure of first monlayer formation is lower if the interaction between adsorbate and adsorbent is stronger, but monolayer formation processes are always overlapped [25]. The isotherms of the CuP shows parallel adsorption and desorption branches and a well-defined hysteresis loop in the relative pressure range p/p$_0$ = 0.2–1. It corresponds to the type H3 according to the IUPAC classification (International Union of Pure and Applied Chemistry). These hysteresis (type H3) are usually found on solids consisting of aggregats or agglomerates of particles forming slit shaped pores (plates or edged particles like cubes), with uniform (type H4) or nonuniform (type H3) size and/or shape. Hysteresis is usually due to a different behavior in adsorption and desorption. For example in pore formed by parallel plates the meniscus is flat (radius =∞) during adsorption (condensation does not take place at any relative pressure) and cylindrical (radius = half the distance between plates) during desorption.

As it will be discussed below, the shape of the adsorption–desorption branches of the V/CuP not differs in function the impregnation content V_2O_5. However, given an impregnation method, whatever the content,

the samples display similar isotherms (for the sake of concision, this result is not illustrated by a figure). The N_2 isotherms corresponding to the solids V/CuP reported in Fig. 2 suggest that the macroporous structure of CuP is not altered by the V_2O_5 content introduction. However, the Content of V_2O_5 impregnation influenced significantly the pore size distribution (determined by applying the BJH method on the desorption branch) which is centered between 0.4 and 1 nm for non-impregnated solid CuP and is shifted to 0.6–1 for all impregnated samples.

The different surface parameters namely specific surface area (S_{BET}), total pore volume (V_p) and mean pore radius $\bar{r}$ were determined for pure and doped solids. The computed values of S_{BET}, V_p and $\bar{r}$ for different investigated solids are given in Table 1. This table shows that the different surface characteristics of various solids were affected by V_2O_5-doping conducted at 450°C. When comparing the CuP, it is noteworthy that the V/CuP samples exhibit slightly higher specific BET surface areas (6.5 to 6.9 m^2/g) as compared to those CuP pure (6.1 m^2/g). In contrast, the V_2O_5 impregnation does not significantly impact on the pore volume which ranges between 0.017 and 0.023 cm^3/g for all samples. The observed increase in the S_{BET} of V/CuP system due to vanadium oxide doping conducted at 450°C could be attributed to creation of new pores during the thermal treatment of the doped solids via liberation nitrogen oxides gases during thermal decomposition of ammonium metavanadate dopant added. Similar results have been reported in the case of V_2O_5/ZrO_2 [26], CuO-ZnO/Al_2O_3 [27], Cr_2O_3/Al_2O_3 [28] and NiO/Al_2O_3 systems [29]. The observed changes in the surface characteristics of the investigated system due to doping with V_2O_5 could be followed by possible changes in its catalytic activity.

3.3. Catalytic activities of various catalysts

The catalytic conversion of isopropanol was conducted over V/CuP solids containing different amounts of V_2O_5 and subjected to heat treatment at 450°C. The catalytic reaction was carried out at temperatures between 180°C and 400°C. The catalytic reaction product was propene only for all investigated catalyst samples, which acted only as dehydration catalysts (Table 2). All values shown were obtained at t = 140 min, shows that the activity remained constant over time at 240°C (fig. 3).

Fig. 4 and fig. 5 show the variation in the conversion of isopropanol as a function of the reaction temperature and of the vanadium oxide content respectively, for the catalytic reaction conducted over V/CuP solids precalcined at 450°C. Inspection of Fig. 4 and fig. 5 revealed that: (i) The rate of isopropanol conversion increased progressively as a function of the reaction temperature. (ii) The catalytic activity towards isopropanol conversion increases with increasing the V_2O_5 content. Preliminary experiments showed that CuP support material preheated at 450°C showed extremely small catalytic activity towards isopropanol conversion. So, V_2O_5 is considered, as the catalytic active constituent of the investigated system. The observed increase in the catalytic activity of the investigated samples due to increasing V_2O_5 content could be attributed to the progressive increase in the concentration of the catalytic constituents involved in the catalytic dehydration process of isopropanol. The observed decrease in the catalytic activity for V(10%)/CuP et V(12%)/CuP could be discussed in terms of a possible decrease in concentration of surface OH–groups, which participate in the catalytic reaction [30] and also to a possible effective dissolution of V_2O_5 in CuP lattice forming V/CuP solid solutions, which exhibited catalytic activity smaller than that measured for V_2O_5 solid.

The transformation of isopropanol is frequently used as a test reaction to determine acid–base properties of oxide catalysts [31]. The reaction can give as main products acetone, propene and, to a lesser extent, diisopropyl ether. Dehydration at acid sites or concerted acid–base pair sites gives propene and diisopropyl ether, and dehydrogenation at basic sites gives acetone. Ether formation must involve an intermolecular coupling reaction. Many contradictory interpretations concerning the mechanism of isopropanol dehydration have been presented and isopropanol transformation cannot be a simple test of acidity [32]. On the other hand, the isopropanol decomposition reaction cannot distinguish between the Lewis and Brönsted sites.

The presence of species with redox properties, such as the vanadium in the catalysts synthesized in this work, can be important in the isopropanol dehydration and dehydrogenation reactions. Unsupported V_2O_5 has both Lewis and Brönsted surface acidity [33]. When vanadia is supported on a metal oxide support, a decrease in the number of surface Lewis acid sites and an increase in the number of surface Brönsted acid sites has been detected [34]. The decrease in Lewis acidity would be related with the coordination of the VO_x species on the metal oxide support and the increase in Brönsted acidity is due to V-OH groups of the VOH_4^{-2} and $V_2O_7H_2^{-2}$ species on the support [35].

In all the samples the vanadium loadings varied between 2 and 12wt% of V_2O_5. The presence of vanadium on the oxides favored the formation of acid sites (Lewis and Brönsted) that would participate in the formation of the propene in all the samples. Therefore, it is important to emphasize that the lack of information about the interaction of vanadium in the structure of the CuP prevented a better interpretation of the results obtained in this study.

The activation energies determined from the Arrhenius plots for isopropanol conversion are also given between 200 and 300°C. Obviously, the highest activation energy was obtained on CuP, and it decreased when

vanadium oxide content increase. It can be presumed that the surface properties are depending on the vanadium oxide load. For instance, the highest content of V_2O_5 phase makes easier the deposition of coke on the surface catalysts. The determination of the apparent activation energy (Ea) for the catalytic conversion of isopropanol yielding propene in the presence of pure and doped V/CuP system has shed some light on the possible changes in the mechanism of the catalyzed reaction. Thus, values of k measured at different reaction temperatures ranged from 180 to 400°C over the variously treated solids have allowed (Ea) to be obtained via direct application of Arrhenius equation $K(t) = Ae^{-Ea/RT}$. The values of (Ea) thus obtained are showed in figure 6. Inspection of the figure 6, revealed that: (i) increasing the extent of V_2O_5 loading brought about a decrease in the value of E_a. These results ran parallel to the observed progressive increase in the catalytic activities of the investigated solids due to increasing the extent of loading. In other words, V/CuP did not modify the mechanism of catalytic conversion of isopropanol but alters the concentration of catalytically active constituents.

IV.   Conclusion

In this work we have reported the synthesis of a pure copper orthophosphate (CuP) and CuP modified by introduction of vanadium (2–12 wt % of V_2O_5) through an easy, cost- and timesaving route, in comparison with previous synthesis of other metal orthophosphates. The thermal treatment at high temperature under atmosphere leads to copper orthophosphate $Cu_3(PO_4)_2$, as shown by XRD analysis, and we have demonstrated also the formation of the crystalline phase V_2O_5 on the surface of support. In the catalytic tests for the isopropanol transformation reaction the catalyst with the largest concentration of copper produced the most propene. In addition the presence of vanadium in the catalysts favored the formation of new acid sites in the catalyst and resulted in the conversion of isopropanol to propene. It was found that the V(8%)/CuP sample exhibited high catalytic activity in the isopropanol decomposition than others samples.

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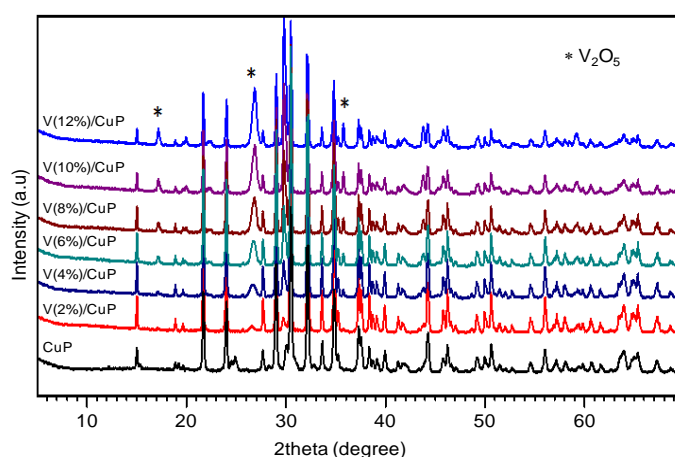
Fig. 1: XRD patterns for solids V/CuP calcined at 650°C
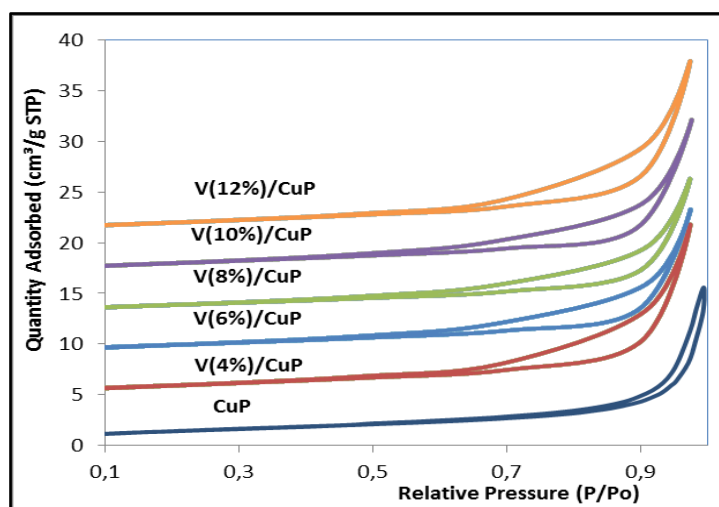

Fig. 2: Nitrogen adsorption-desorption isotherms of raw CuP and V/CuP catalysts: (For a better visibility, the isotherms are shifted along the Y-axis).
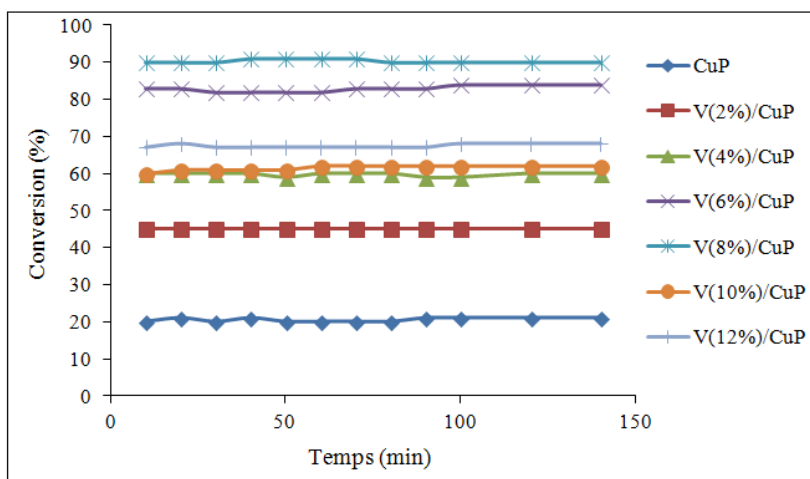
Fig. 3: Isopropanol decomposition as function of the time reaction on V/CuP catalysts at 240°C.
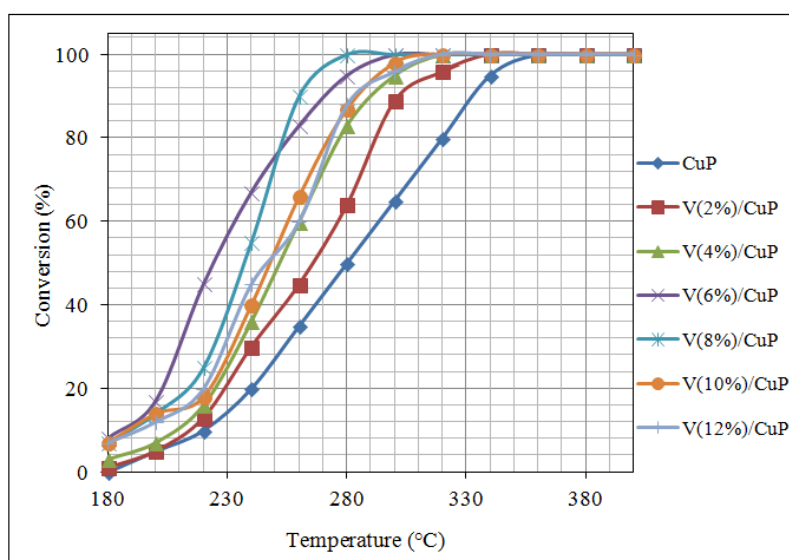

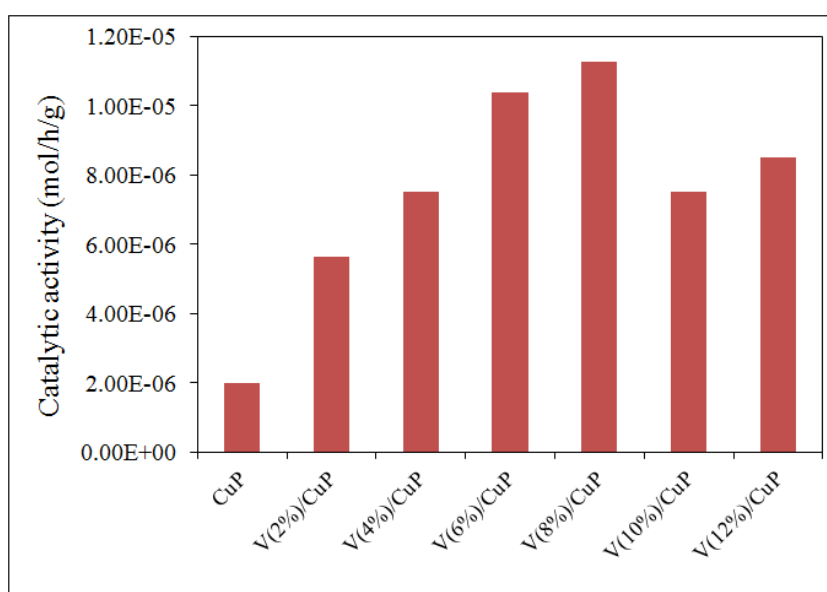**Fig. 4:** Isopropanol decomposition as function of the reaction temperature on V/CuP catalysts.

Fig. 5: Catalytic activity of V/CuP catalysts in reaction of isopropanol decomposition at 240°C.
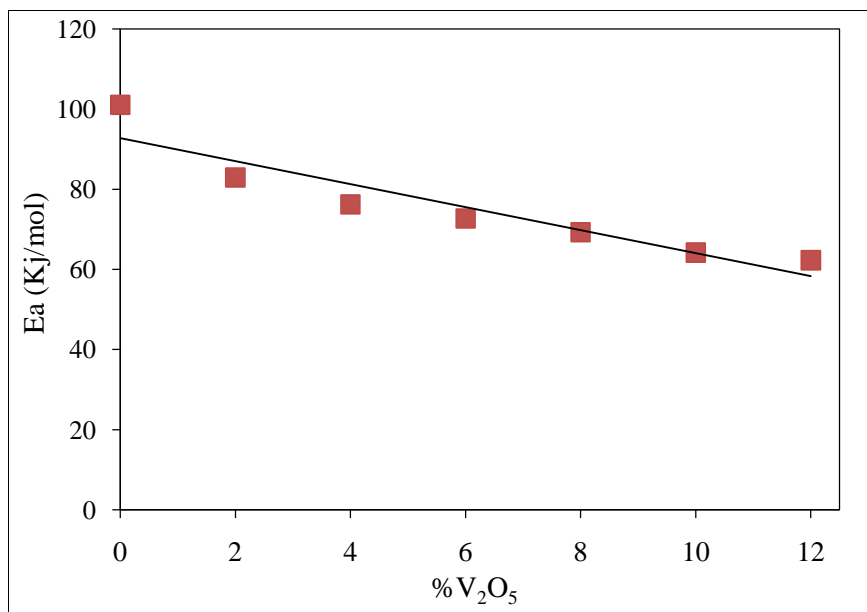
Fig. 6: Activation energy as function of the vanadium oxide content for various V/CuP solids calcined at 450°C.

Table 1: Specific surface area (S_{BET}), cumulative pore volume (V_p), and average pore diameter (d_p) for the solids calcined at 450°C.

Catalysts	S_{BET} (m²/g)	V_p (cm³/g)	d_p (nm)
CuP	6.1	0.017	11
V(4%)/CuP	6.6	0.022	13
V(6%)/CuP	6.7	0.019	12
V(8%)/CuP	6.5	0.018	11
V(10%)/CuP	6.9	0.020	11
V(12%)/CuP	6.9	0.023	13

Table 2: Isopropanol conversion (%) and selectivity towards propene, acetone at 240°C over V/CuP catalysts calcined at 450°C.

Solids	specific area (m²/g)	Conversion (%)	$S_{propene}$ (%)	$S_{acetone}$ (%)	Catalytic activity 10^{-6} mmol/h/g	10^{-6} mmol/h/m²
CuP	6.1	20	100	0	2,50	0,41
V(2%)/CuP	–	45	99	1	5,63	–
V(4%)/CuP	6.6	60	97	3	7,50	1,14
V(6%)/CuP	6.7	83	97	3	10,40	1,55
V(8%)/CuP	6.5	90	98	2	11,30	1,74
V(10%)/CuP	6.9	60	97	3	7,50	1,09
V(12%)/CuP	6.9	68	97	3	8,50	1,23