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Surface properties of Na-doped V_2O_5/TiO_2 systems

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Abstract. V_2O_5/TiO_2 catalysts doped with sodium and calcined at different temperatures have been prepared and characterised by XRD, N_2 adsorption and v -UV/diffuse reflectance and IR spectroscopies. Changes in the anatase content and in the specific surface area have been related to the formation of V–Na–O ternary compounds.

A series of V_2O_5/TiO_2 systems, currently used as oxidation catalysts, have been characterised after doping with sodium and calcination at different temperatures, in order to check their effect on the texture properties of the catalysts, as it is well known [1–3] that the addition of dopants, mainly alkaline metals, and catalyst pre-treatment play an important role in the selectivity of these systems.

The support (TiO_2 P-25 Degussa, 50% anatase, $\approx 50 \text{ m}^2 \text{ g}^{-1}$) was doped with sodium (0, 1, 3%) by impregnation with NaOH(aq), drying and calcination at 773 K in O_2 . Vanadia was incorporated by dry impregnation (series I) with aqueous oxalic solutions of NH_4VO_3 and calcination at 773 or 973 K in O_2 ; for comparison, another set of samples (series M) was prepared by melting vanadia on titania at 973 K. Characterisation was carried out by XRD (Philips 1730), v -UV/DR (Shimadzu UV-240) and IR (Perkin–Elmer FT-1730) spectroscopies, and N_2 adsorption measurements at 77 K.

Samples I-973 consist solely of rutile, while in samples M-973 the content of anatase decreases (up to 7–16%) as the content of Na increases. Simultaneously, S_{BET} decreases sharply, being $\approx 1 \text{ m}^2 \text{ g}^{-1}$ for samples containing 3% Na. Vanadium species are well dispersed in the Na-free I samples, while the XRD diagrams of Na-free M samples show peaks due to V_2O_5 . In contrast, in the presence of Na, peaks due to stoichiometric ($Na_4V_2O_7$, Na_3VO_4 , $NaVO_3$) and non-stoichiometric ($Na_xV_2O_5$ bronzes) compounds are recorded, the content of the former increasing in the samples that were richest in Na and that were calcined at 973 K.

In the v -UV/diffuse reflectance spectra, samples M with low ($\leq 1\%$) Na content show an absorption band at 500–430 nm due to V_2O_5 , which is absent in the corresponding I samples. However, the two sets of samples display similar spectra when the calcination temperature increases up to 973 K, because the melting and dispersion of V_2O_5 leads to the samples becoming fairly similar in the two cases (see table 1).

The IR spectra of the Na-free samples show a shoulder at 1000 cm^{-1} (series I) due to $\nu_{V=O}$ in crystalline V_2O_5 [4], that shifts to 1025 cm^{-1} as the crystallinity improves (series M). In contrast, samples containing Na–V–O compounds show only a band at 900 cm^{-1} due to the presence of bronzes [5].

Table 1

Preparation method Calcination temperature (K)	I						M					
	773			973			773			973		
%Na	0	1	3	0	1	3	0	1	3	0	1	3
%anatase	55	45	47	0	0	0	55	55	51	16	0	7
S_{BET} ($\text{m}^2 \text{g}^{-1}$)	46	24	32	6	5	<1	50	38	34	16	3	<1

N_2 adsorption–desorption isotherms are quite similar in all cases, the adsorption capacity decreasing (because of ‘rutilisation’) as the Na content and/or the calcination temperature increases.

It can be concluded that both the anatase content and the specific surface area depend mainly on the calcination temperature and that, for a given calcination temperature, they both decrease as the Na content increases.

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