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Structure - Catalytic Activity Correlation in Aluminium Hydroxide Supported Vanadia Catalysts

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Abstract

Aluminium hydroxide samples containing vanadium in the range 5 - 25 wt % have been prepared by incipient wetness impregnation method. The catalysts have been characterized through % composition of vanadium, BET surface area, FTIR, PXRD, SEM and solid state V-NMR. Liquid phase conversion of benzyl alcohol over the above materials into various products has been used as a chemical probe to investigate the activity of these catalysts. Benzyl alcohol has been converted into benzaldehyde, toluene and 1, 2-diphenyl ethane (dibenzyl). Formation of various products at different loadings of vanadium is found to be related to structural features of vanadium oxo-species. Oxidation- reduction products and 1, 2-diphenyl ethane have been formed selectively, at lower loadings of vanadium, which was in the tetrahedral environment of oxygen and at higher loading when vanadium was in square pyramidal environment catalytic activity increased but with low selectivity. When aluminum hydroxide was used as a support monolayer coverage was completed only at higher loading indicating better dispersion of metal - oxo species over this support.

Key words*:* $Al(OH)_3$ support, $Al(OH)_3$ supported vanadia, 1, 2-diphenyl ethane, benzyl alcohol.

1. Introduction.

Supported- vanadia catalysts are known for a number of organic conversions, for instance, partial oxidation of methanol to formaldehyde [1], oxidation of *o*- xylene to phthalic anhydride [2], ammoxidation of aromatic hydrocarbon [3], oxidation of SO_2 to SO_3 [4,5] selective catalytic reduction of sulphur trioxide [6-8], selective oxidation of aromatics [9-12], olefins [13], selective catalytic reduction of NOx by NH_3 [14] as well as for the oxydehydrogenation of short chain paraffins. Catalytic properties of supported- vanadia depend on both the V loading and the acid- base nature of the oxide support [15].

It was earlier reported by various authors that the catalytic activity of a supported catalyst depends on the metal oxide- support composition [16-18]. In many cases it was proved that catalytic activity increases with increase in the loading level of metal-oxo species and reaches a maximum, beyond which there is either decrease or no change in activity. Usually, when the metal oxide is well dispersed on the support the catalytic activity found to be more. Beyond monolayer coverage, catalytic activity decreases due to the crystalline metal

oxide formation on the support. The monolayer capacity depends on the nature of the support. Earlier it was reported that higher the interaction of hydroxyl groups of support with the metal oxo species, higher will be the dispersion and hence higher catalytic activity [19-21]. Al_2O_3 is very well known for its role as a support. Support –metal ion interaction at a given pH on Al_2O_3 depends on its acid-base properties, which in turn depend on the surface –OH group concentration which is a function of calcination temperature [22, 23].

Recently, we have been interested in evaluating the merits of using aluminum hydroxide as a support for oxospecies of Mo, Cr and V and determine their catalytic performance in benzyl alcohol conversion. Interestingly we have observed different metal-oxo species exhibited different catalytic activity when compared to pure $Al(OH)_3$. 5 wt % Mo containing support enhanced the formation of dibenzyl ether [24], where as supported V catalyst resulted an additional product dibenzyl as one of the major products. Chromium catalysts did not show any activity neither towards the formation of high DBE nor towards dibenzyl (unpublished results).

Keeping these interesting observation as a starting point we have extended our work to check the effect of different loadings of vanadium on $Al(OH)_3$ and their structure and activity correlation in the structure sensitive benzyl alcohol reaction.

Inorder to materialize the said objectives, V- $Al(OH)_3$ system containing 5-25 % wt of V were prepared, characterized and catalytic performance was evaluated in liquid phase benzyl alcohol conversion.

2. Experimental.

2.1. Preparation of aluminum hydroxide supported vanadia catalyst.

The supported catalysts have been prepared by incipient wetness impregnation method using uncalcined $Al(OH)_3$ support obtained commercially. The aluminum hydroxide sample was dried at 120 ºC for 12 hrs. In a typical procedure a known amount of 120 ºC dried aluminum hydroxide was mixed with required amount of a solution of ammonium metavanadate obtained by dissolving the salt in 2M oxalic acid (higher amount of ammonium metavanadate is insoluble in water) to obtain finally 5, 9, 15, 20 and 25 wt % vanadium containing samples. After an hour of thorough mixing the contents were heated at 120 ºC overnight in an air oven and finally calcined at 550 ºC for 5 hours. Pure support was also calcined separately at 550 ºC for 5 hours to compare its physico- chemical properties with the calcined supported vanadium catalysts.

2.2. Characterization.

Percentage composition of vanadium in the supported catalysts was determined by colorimetric method. By trial and error method, the entire catalyst was found to be soluble on heating with conc.H_2SO_4, followed by addition of water in the ratio 4:1. Further vanadium was converted to a reddish brown complex of $(VO_2)_2(SO_4)_3$ by adding H_2O_2 in acidic medium. The absorbance of the solution was measured at 450mμ [25]. Surface areas of the support and supported catalysts calcined at 550 ºC were measured by N_2 adsorption at 77 K by the BET method using a NOVA-1000 (VER: 3.7) instrument. The crystallographic phases present in the catalysts were determined by recording powder X-ray diffraction

patterns on a Philip analytical X-ray diffractometer type PW 3710 using Cu Kα radiation (1.54nm) with a Ni filter**.** To check the formation of any V_2O_5 crystallites and the nature of bonding of metal-oxo species with the support IR absorption spectra of the catalysts were recorded by the KBr pellet method over the range 4000-600cm^{-1} using a Jasco FTIR-410 instrument. SEM micrographs of few representative samples were recorded, to check the morphology of these catalysts. MAS solid state ^{51}V-NMR has been recorded to obtain information about the local coordination of vanadium oxide species, in supported catalysts.

2.3. Catalytic activity studies.

Catalytic activity measurements have been carried out in liquid phase benzyl alcohol reaction as follows. 5 ml of benzyl alcohol was refluxed for 6hrs with 0.5 g of catalysts in a round bottomed flask, fitted with a water condenser. The catalyst was separated by filtration after the reaction and the number of products formed were analysed by TLC and further analysed by GC using 10% SE 30 chromosorb column. The nature of the products formed was confirmed by ^{1}H-NMR, ^{13}C-NMR and GC-MS.

3. Results and Discussion

3.1. Percentage composition of vanadium.

The percentage of vanadium present in the various samples prepared was found to be matching with the theoretical values. The experimentally determined % composition of vanadium is tabulated in table1. This method was found to be very accurate for the estimation of vanadium in solid catalyst as the results were reproducible and values matching with the theoretical.

3.2. BET surface area.

The BET surface area of uncalcined and 550 ºC calcined commercial aluminum hydroxide was found to be 191 and 245 m^2/g respectively. The increase in the surface area of support on calcination is due to the conversion of crystalline aluminum hydroxide to amorphous nature as indicated by the powder diffraction pattern of these samples. The BET surface areas of various supported catalysts calcined at 550 ºC are shown in table 1. There is a consistent decrease in surface area with increase in vanadium content. This is in agreement with values appeared in the literature for activated alumina supported vanadium catalysts reported by Reddy et.al. [26]. Margarita et. al also report deep decrease in specific surface area in the presence of vanadia on alumina [27]. However it is noteworthy that samples containing 20 wt% vanadium stands out with high surface area against the trend in the series, probably due to the change in the nature of chemical environment of vanadium species present in the sample, which is indicated by its X-ray diffraction and solid state VNMR patterns which is discussed in the later part of this paper in detail. The decrease in surface area of supported catalysts compared to pure support is mainly due to the penetration of vanadium species into the pores of the support, resulting in the dispersion of vanadium species on the support

3.3. Powder X-ray diffraction

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The powder X- ray diffraction patterns of pure support, 5 - 20 wt% V containing support as well as pure V_2O_5 are presented in figure 1. The broad diffraction peaks of calcined pure support were matching with γ phase alumina as reported earlier [28]. It is noticed that crystalline V_2O_5 is not formed up to15 wt % vanadium loading. Hence it can be inferred from diffraction studies that vanadium oxide is completely dispersed on the surface of the support. The decrease in surface area due to the inclusion of vanadium species in to the pores further confirms this aspect. Absence of crystalline V_2O_5 indicates strong interaction of support with the metal oxide. Hence by using uncalcined $Al(OH)_3$ as support the extent of interaction between the support and precursor can be enhanced. X-ray diffraction pattern of AlV-20 and AlV-25 samples exhibited peaks corresponds to 2θ values 15.2, 20.09, 21.5, 26.09, 30.89, 32.33 and 34.25. Pure V_2O_5 obtained by the calcination of ammonium metavanadate also exhibited peaks at the same 2θ values. Hence it is very clear that crystalline V_2O_5 has been formed at this vanadium content. The very low surface area ($86 m^2/g$) of AlV-25 sample can be attributed to the formation of large number of V_2O_5 crystallites. Hence it can be inferred from X-ray diffraction studies that at low vanadium loadings dispersion is higher and at higher vanadium loadings the dispersion decreases. Bars et.al discussed earlier that vanadium-oxo species exist as tetrahedral VO_4^{3-} units on alumina support up to monolayer coverage [18]. When uncalcined $Al(OH)_3$ is used as the support, monolayer coverage persist till a loading level of 15 wt % of vanadium. X- ray diffraction patterns, solid state V NMR and surface area values of these samples confirm this aspect . The higher capacity of $Al(OH)_3$ support to disperse vanadium species may be attributed to higher number of hydroxyl groups available on the surface of uncalcined $Al(OH)_3$.

3.4. FTIR

Figure 2 shows infrared spectra of pure support, V_2O_5 and $Al(OH)_3$ support containing 25 wt % vanadium. A comparative IR spectral analysis of pure support, V_2O_5 and vanadium containing supports, indicated the formation of V_2O_5 phase on the support only when the wt % of V exceeds 20 %. In case of samples below 20 wt % of vanadium, characteristic peaks due to V_2O_5 were not observed. This is in good agreement with XRD results. However as is clearly seen in the IR spectra presented, the crystalline V_2O_5 formation in 25wt % V containing sample is characterized by a small shoulder peak at 1020 cm^{-1} corresponds to V=O stretching [29]. Coincidently, X-ray diffraction pattern of this sample also indicate appearance of crystalline V_2O_5. Though XRD pattern of 20 wt % vanadium content also indicate V_2O_5 formation the FTIR does not exhibit corresponding absorption bands. This can be attributed mainly to two reasons, firstly the amount of V_2O_5 phase formed is less in case of 20 wt % sample and secondly there is an overlap of absorption bands of Al-O (540-900 cm^{-1}) and V-O band which makes the later undetectable.

3.5. SEM

Figures 3 shows SEM back scattered images of 5 and 20 wt% V containing catalysts. Very clear distinction can be made between these two pictures with respect to the nature of the particle size. In the case of AlV-5 sample, there are no clear crystallites seen as observed in the case of AlV-20 sample. The clear particles observed in the case of AlV-20 sample may

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be attributed to small crystallites of V_2O_5 as indicated by the powder X-ray pattern of this sample.

3.6. MAS solid state ^{51}V-NMR.

The MAS ^{51}V solid-state NMR experiments provide detailed information about the local coordination of the two-dimensional surface vanadium oxide species on support. The structure of these surface phases depends on the surface coverage [30], the nature of the oxide support, and the extent of hydration. V NMR studies on 5-25 wt% vanadium content samples in the present studies indicate that at different percentage levels of vanadium, different phases are formed. For example, at 5wt % vanadium loading, the signals located at -774, -584, -477, -422 ppm indicates tetrahedral vanadium sites of
Q^0 and Q^2 type. The proximity in the band positions of 5-15 wt % vanadium containing sample indicating that vanadium is located in similar environment over this range. Upon increasing the vanadium content above 15 wt % a central band located at 613-614 ppm is observed. According to the reported literature, this pattern indicates a V_2O_5 like structure i.e square pyramidal. There is a striking resemblance in the spinning side band intensity patterns between the 20-25 wt % V catalyst and pure V_2O_5 obtained by the calcination of ammonium metavanadate. Hence it can be noticed that when vanadium-oxo species is supported on aluminum hydroxide support, up to 15 wt %, vanadium species exist in an environment of tetrahedral oxygen (Figure 4). When the percentage is 20 - 25, it exists in square pyramidal geometry. Even pure V_2O_5 obtained by the calcination of precursor at 550 ºC also exhibit square pyramidal structure [31, 32]. When Hellmut Eckert suggests square pyramidal structure for V_2O_5, Lapina et.al report distorted octahedral or trigonal bipyramidal of 3=2 type structure. The environment could be described as 6- coordinate, by considering the very long second axial V-O distance as reported by Hellmut Eckert. It is noticed that with increase in the percentage of vanadium, the environment of vanadium species in the catalyst changes from tetrahedral to square pyramidal. The 5 wt % V containing sample also shows four discernible peak patterns as reported earlier for model compound $Na_6V_{10}O_{28}.18\ H_2O$, corresponding to crystallographic in equivalent sites. [33 and references sited there in].

3.7. Catalytic activity.

First of all, the % conversion of benzyl alcohol over supported vanadium samples was much higher than pure support. When toluene, benzaldehyde and dibenzyl ether (DBE) were the products formed in case of pure support, 1, 2 –diphenyl ethane (DPE) was also formed as an additional product in case of supported vanadium catalyst (Table 2). Absence of DPE formation in the case of support indicates that this product is formed due to the presence of vanadium species. However it was noticed that there was no increase in the % formation of DPE with respect to vanadium content. Earlier Tuel et. al reported DPE over [V]ZSM -48 catalyst on toluene oxidation reaction following a homolytic mechanism involving the formation of benzyl radicals [34,35]. Hence it can be assumed here also that DPE is formed from toluene and not from benzyl alcohol directly. The reduction in the % of toluene when DPE is formed strongly support this point. Thus it can be inferred that vanadium species enhances benzyl free radical formation.

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Further, catalytic activity studies on benzyl alcohol reaction over $Al(OH)_3$ supported vanadia catalysts indicated a good correlation between structure and catalytic activity. At the outset it was observed that percentage conversion of benzyl alcohol increased with increase in the percentage of vanadium in the samples (figure5). Interestingly the % selectivity towards various products was relied on the environment of vanadium on the support which in turn depends on the % of vanadium in the sample. For instance yield of oxidation-reduction products (benzaldehyde and toluene) increased significantly when the coordination of vanadium changed from tetrahedral to square pyramidal. ie when the wt % of V was above 15. This can be accounted as follows.

After calcination the vanadium oxide is mainly in oxidation state V^{5+}. On alumina support it can be reduced to an average oxidation state between V^{4+} and V^{3+} .The reduction behaviour of the calcined vanadium oxide is mainly influenced by the vanadium content of the catalyst and by the support [36]. Vanadium in tetrahedral environment of oxygen at lower loadings has fewer tendencies to get reduced compared to vanadium at square pyramidal environment due to the difference in electronegative oxygen environment. This accounts for the higher amount of oxidation products at higher % loading of vanadium.

Even in the absence of catalyst, 6-10 % of toluene and benzaldehyde are formed. Absence of dibenzyl ether (DBE) in blank reaction clearly indicates that catalytically active sites are required for the formation of dibenzyl ether. Dehydration product DBE was formed in presence of support as well as supported catalyst. Formation of DBE over support indicates that required acid sites are available with the support .

The increase in the % of DBE at higher loadings of vanadium is an indication of higher concentration of acid sites. Addition of excess vanadium creates Lewis acid sites which influences the formation of DBE. This is in agreement with earlier report by Blasco et.al who observed increase in Lewis acid sites on addition of vanadium [30,37, 38]. However % yield of DBE was negligible at lower loadings of vanadium. It indicates the need of critical amount of vanadium content on the support to exhibit dehydration activity by vanadium species on $Al(OH)_3$ support.

It is noteworthy, that all the characterization techniques used such as FTIR, XRD, NMR support the fact that there is a monolayer dispersion of vanadium up to 15 wt% V on the $Al(OH)_3$ support with out the formation of crystallites of V_2O_5 . The higher % dispersion of vanadium has showed its effect on increased catalytic activity of supported V catalysts when compared to pure support.

It is also interesting to note that a change in structure of V species on supports from tetrahedral to square pyramidal environment, influenced a noticeable change in catalytic activity towards the formation of oxidation reduction products. It is not only the nature of the support but also the amount and its chemical environment of the active species on the support influence the % conversion as well as the selectivity in benzyl alcohol reaction; thus showing a good correlation between structure and catalytic activity of supported vanadium catalyst.

Recycling of all the catalysts found to be almost equally good to the first trial. Benzyl alcohol reaction is also conducted in the presence of pure V_2O_5. However seperation of V_2O_5 from products were rather difficult. After the reaction, though V_2O_5 turned black in colour

(probably due to the decomposition of products on the surface of catalyst), on further calcination it regained its original yellow color. Reusability of the catalyst indicated that V_2O_5 does not undergo decomposition during the reaction. From the catalytic activity difference of supported catalyst and pure V_2O_5 it can be inferred that aluminum hydroxide supported vanadium oxide catalysts is better than pure V_2O_5 in benzyl alcohol conversion. More over the former works out to be much cheaper than the latter and the usage of aluminum hydroxide in the place of Al_2O_3 as support saves energy as it is not calcined to higher temperature before using as the support.

When 25 wt% vanadium containing sample is used in the conversion of benzyl alcohol, the activity reached maximum i.e 88 %. Thus this heterogeneous catalyst provided not only high yield but also a simple and practical protocol for benzyl alcohol conversion.

Our further work in this area is aimed at reducing the number of products formed in the conversion of benzyl alcohol, by selectively poisoning/activating the catalytic centres.

4. Conclusions

Thus the present study shows that $Al(OH)_3$ is a good support for vanadium species, as it allows a higher percentage vanadium dispersion on it, resulting in higher catalytic activity. Further a change in the structure of vanadium species from tetrahedral to square pyramidal on this support when the % of vanadium is in between 15- 20 % also exhibit a noticeable increase in the % conversion of benzyl alcohol. These two observations, supported by IR, XRD and NMR spectral analysis of the catalyst, clearly indicate a good structure and catalytic activity correlation of $Al(OH)_3$ supported vanadium catalyst in benzyl alcohol conversion.

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Table1: Physico-chemical properties of aluminum hydroxide support and aluminum hydroxide supported vanadium catalyst

Catalyst	Specific surface area m^2/g	% composition of vanadium
Al	245	-
AlV -5	229	5.0
AlV -9	193	9.0
AlV -15	172	15.1
AlV -20	200	20.0
AlV -25	86	25.1

Al: Aluminum hydroxide support;

AlV -5: Aluminum hydroxide support containing 5 wt% vanadium.

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Table 2: Percentage formation of various products in benzyl alcohol reaction.

Catalyst	Toluene %	Benzaldehyde %	Dibenzyl %	DBE %	Benzyl alcohol %
Al	4	13	-	12	81
AlV-5	13.5	19.5	21.5	-	44.2
AlV-9	10.4	23.3	26.7	-	38.3
AlV-15	8.6	23.3	18.4	14.6	35.1
AlV-20	20.9	34	14.3	12.8	18
AlV-25	36	32.8	11	8.7	11.5
V_2O_5	1.1	17.8	5.9	15	60.2

DBE: Dibenzyl ether, weight of the catalyst: 0.5 g
Benzyl alcohol : 5 ml, Time of reflux: 6 hrs

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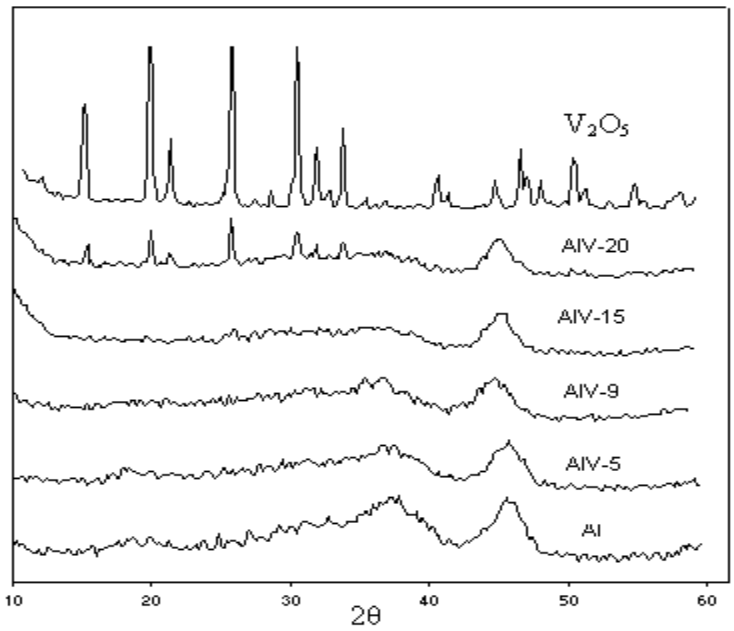

Figure 1

Figure1: X- ray diffraction patterns of support and supported V catalysts

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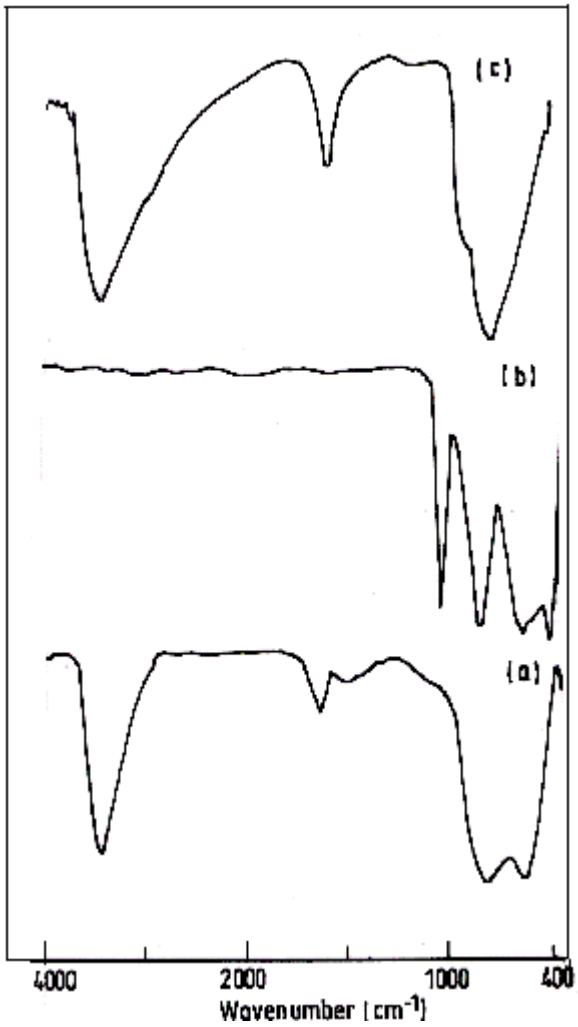
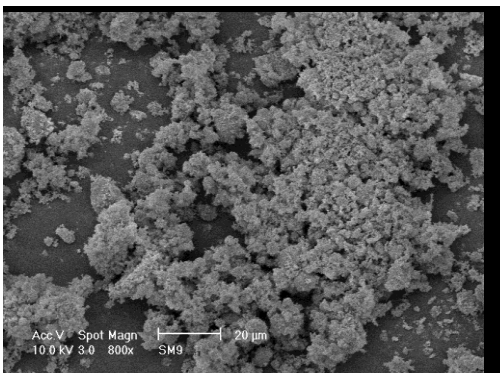
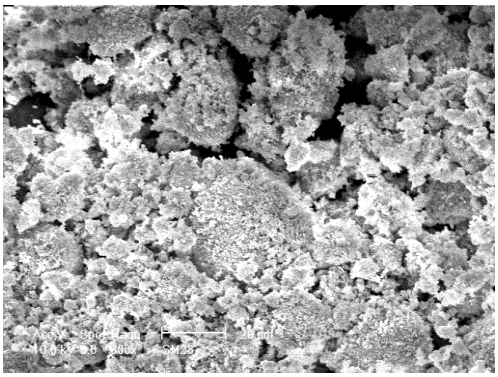


Figure 2. FTIR spectra of a) Al; b) V_2O_5; c) AlV- 20





3(a) **3(b)**

Figure 3. SEM pictures of a) AlV-5; b) AlV-20

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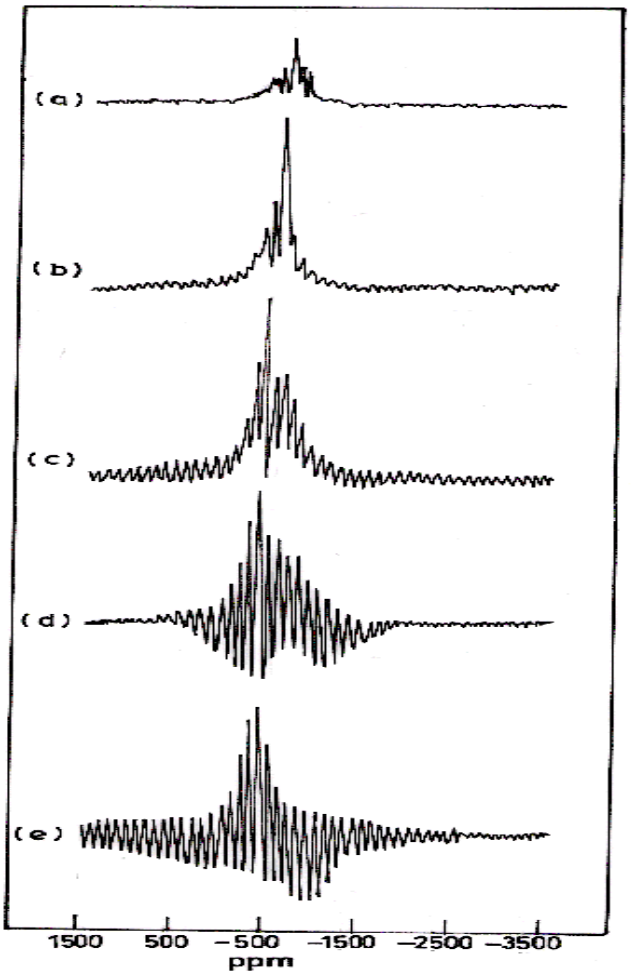

Figure 4. ^{51}V MAS-NMR spectra of a) AlV-5; b) AlV-9; c) AlV-15; d) AlV-20; e) V_2O_5

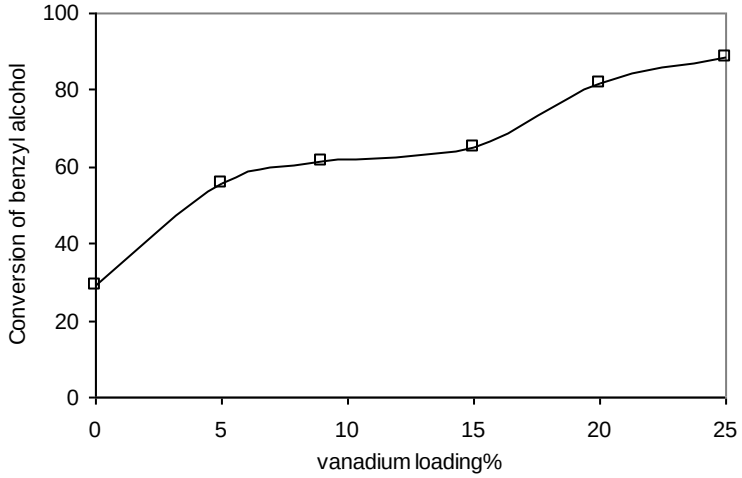

Figure 5. % conversion of benzyl alcohol as a function of vanadium content