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APPENDIX A

Sample of Calculation

A.1 Calculation of Designed Metal Loading for Catalyst Preparation.

The sample of calculation shown below is for 28V-Mg-O catalyst.

Aqueous solution consists of NH_4VO_3 0.5 wt%
NH_3OH 1.0 wt%

Volume of solution is designed to be 100 ml., hence NH_4VO_3, NH_3OH and H_2O are weighted for 0.5, 1.0, and 98.5 gram respectively.

The amount of MgO powder for 28%(by weight) vanadium oxide in V-Mg-O catalyst is calculated as follows:

if the weight of catalyst is 100 gram,

28V-Mg-O would compose of V_2O_5 28 g. and MgO 72 g.,

therefore in this system (compose of V_2O_5 0.5 g.),

the amount of MgO = $72/28 * 0.5 \approx 1.2857$ g.

therefore there is vanadium oxide on MgO support $= 0.5/(0.5+1.2857) * 100$
$= 28$ wt%

APPENDIX B

Calculation of Flow Rate and Explosive Limit

B.1 Calculation of Flow of C_3H_8, air and argon with varying HC/O_2 Mole ratio.

The calculation shown below is for feed composition of 4 vol% propane, 8 vol% oxygen and the balance argon:

for total flow = 100 ml./min.

flow of 21% O_2 in air is assumed to be X ml./min.

flow of 20% C_3H_8 in N_2 is assumed to be Y ml./min.

if the feed contains 8 vol% oxygen,

then there is O_2 in flow of O_2 in air = 21X/100 = 8

also there is C_3H_8 in flow of C_3H_8 in N_2 = 20Y/100 = 4

therefore X = 38.09 ml./min.

Y = 20 ml./min.

argon used as balance gas, hence its flow is equal to 100 - 38.09 - 20

= 41.91 ml./min.

B.2 Calculation of explosive limit of propane in air.

The explosive limit of propane in air are shown below [82],

	lower limit in air	upper limit in air
propane	2.37 vol%	9.5 vol%

Therefore, the amount of propane in the feed must not be in the range of this explosive limit (between 2.37-9.5 % in air).

Sample of calculation for the condition of feed stream 4:8:88,

flow rate C_3H_8 used in this study is fixed to 4 ml./min.

flow rate of air is 38.09 ml./min.

hence, there is propane in air = 4/38.09 * 100

= 10.50% (this value is O.K.)

APPENDIX C

Chemisorption at Oxide Surfaces [29]

Table C.1 Classification of heterogeneous catalysts [29]

Class	Functions	Examples
Metals	hydrogenation dehydrogenation hydrogenolysis (oxidation)	Fe, Ni, Pd, Pt, Ag
Semiconducting oxides and sulphides	oxidation dehydrogenation desulphurization (hydrogenation)	NiO, ZnO, MnO_2, Cr_2O_3, Bi_2O_3-MoO_3, WS_2
Insulator oxides	dehydrogenation	Al_2O_3, SiO_2, MgO
Acids	polymerization isomerization cracking alkylation	H_3PO_4, H_2SO_4, SiO_2-Al_2O_3, zeolites

Table C.1 shows the kinds of reaction catalysed by oxides depending upon whether the oxide is a semi-conductor or an insulator. It is now time to try to explain these terms and to seek a rationalization of catalytic behavior in terms of the chemisorption of molecules on oxide surfaces. The problem is in some ways more difficult than for metals for several reasons [29]:

1) there is the simple fact that the surface contains two types of species, anions and cations, and in general their relative amounts and steric disposition will

vary from one crystal plane to another. It has to be established whether both species participate in a given chemisorption.

2) Oxides differ greatly from one another in their thermal stability. Those of the pre-transition elements, the so-called ceramic oxides, are remarkably stable and find numerous high-temperature applications, while those of the transition and post-transition elements in general lose oxygen under vacuum, especially when heated. For this reason, and because they are not electrical conductors, they are not so easy to study by the physical techniques that have proved rewarding in the case of metals. Less is therefore known about the adsorbed states of molecules on oxides.

3) Many, indeed most, of the oxides used as catalysts in practice are in fact binary or more complex mixtures of oxides. It is difficult to define their surface composition, and fundamental studies of chemisorption on them require extremely careful work. Further complications will appear in due course.

To understand more about the chemisorption properties of oxides, and the reactivity of their surfaces, it is necessary first to know more about what happens below the surface. It has long been recognized that the mobility of electrons within a solid gives useful information about the nature of the chemical bonds that it contains. The following theory is the examination about the electrical conductivities of solids. This in turn will assist the understanding of chemisorptive and catalytic properties of oxide surfaces.

C.1 Electrical Conductivity of Solids

On the basis of their electrical conductivities, solids are traditionally divided into four classes as shown in Table C.2. Superconduction is only shown by metals at very low temperatures, and they are included simply to emphasize the astonishing range of 55 orders of magnitude over which electrical conductivity can be measured. There are two classes of semiconductors. Group III-GroupV compounds like gallium arsenide, are termed *intrinsic* semiconductors because their conductivity is an inherent feature of their chemical structure. However, important as these substances are in solid state devices, they are not catalytically active and will not be considered further. The greater interest are the oxides and sulphides whose conduction is due to their departure from precise stoichiometry: these substances are termed *extrinsic* or defect semiconductors. The more non-stoichiometric they are, the greater their conductivities increase with temperature according to a relation similar to the Arrhenius equation, so that a straight line is obtained on plotting log (conductivity) against reciprocal absolute temperature.

Table C.2 Classification of solids by electrical conductivity [29]

Class	Conductivity range ($\Omega\ cm^{-1}$)	Chemical class	Examples
Superconductors	up to 10^{35}	metals at low temperatures	-
Conductors	10^{4}-10^{6}	metals and alloys	Na, Ni, Cu, Pt etc.
Semiconductors	10^{3}-10^{-8}	(a) intrinsic:	
		semi-metals	Ge, Si, GaAs, etc.
		(b) extrinsic:	
		oxides and sulphides of transition and post-transition elements	ZnO, Cu_2O, NiO, ZnS, MoS_2, NiS etc.
Insulators	10^{-9}-10^{-20}	Stoichiometric oxides	MgO, SiO_2, Al_2O_3 etc.

Now some oxides when heated in air becomes oxygen deficienct: zinc oxide is an example. Others like nickel oxide acquire oxygen, and become non-stoichiometric by having an excess of oxygen in the lattice.

C.2 Chemisorption on Semiconducting Oxides

Table C.3 Classification of semiconducting metal oxides [29]

Effect of heating in air	Classification	Examples
Oxygen lost	Negative (n-type)	ZnO, Fe_2O_3, TiO_2, CdO, V_2O_5, CrO_3, CuO,
Oxygen gained	Positive (p-type)	NiO, CoO, Cu_2O, SnO, PbO, Cr_2O_3

A qualitative understanding of the chemisorption of simple gases on semiconducting oxides follows simply from their chemistry as the band theory of solid [5]. Reducing gases such as hydrogen and carbon monoxide are adsorbed strongly, but irreversibly; on heating, only water and carbon dioxide respectively can be recovered. Hydrogen probably dissociates heterolytically on adsorption, viz.

$$H_2 + M^{2+} + O^{2-} \rightarrow HM^{+} + OH^{-}$$

The hydroxyl ions will decompose on heating to form water and anion vacancies, and an equal number of cations will be reduced to atoms. Carbon monoxide usually chemisorbs first on the cation, whence it reacts with an oxide ion:

$$(CO)...M^{2+} + O^{2-} \rightarrow M + CO_2$$

What is observed here is the first stage of a process that can lead ultimately to the complete reduction of the oxide to metal. These steps are also similar to those involved in the catalysed oxidation of these molecules.

The chemisorption of oxygen on p-type oxides which gain oxygen on heating in air occurs by a mechanism involving the oxidation of Ni^{2+} ions at the surface to Ni^{3+} :

$$2Ni^{2+} + O_2 \rightarrow 2(O^{-}...Ni^{3+}).$$

High coverages by the O^{-} ion can result, and it is easy to see that this is the first step in the incorporation of excess oxygen, referred to above. When the n-type oxides which lose oxygen on heating in air (exemplified by zinc oxide) are exactly stoichiometric, they cannot chemisorb oxygen; when however they are oxygen-deficient, they can chemisorb just as much as is needed to restore their stoichiometry by refilling the anion vacancies and reoxidizing the zinc atoms. For the reasons mentioned at the beginning of this appendix, there is not the equivalent body of quantitative information concerning chemisorption on oxides that there is for metals. The principles just described will however be helpful towards rationalizing their catalytic behaviour.

C.3 Adsorption on Insulator Oxides

Since the cations of insulator oxides can be neither oxidized nor reduced, they cannot chemisorb oxygen to any significant extent; they cannot chemisorb hydrogen or carbon monoxide for the same reason. They can, and do, react with water and other polar molecules as:

$$M^{x+} + O^{2-} \rightarrow (OH^{-}...M^{x+}) + OH^{-}$$

$$M^{x+} + O^{2-} \rightarrow (CH_3O^{-}...M^{x+}) + OH^{-}$$

Indeed under normal circumstances the surfaces of oxides such as alumina and silica are covered by a layer of chemisorbed water; the surface is then said to be fully hydroxylated, and indeed these hydroxyl groups are very firmly bound. Their complete removal by heating is almost impossible. When the oxides are suspended in water the M-OH groups can dissociate either as acids or as bases, depending on the electronegativity of the cation, e.g.

$$\text{Mg-OH} \rightarrow \text{Mg}^{2+} + \text{OH}^{-}$$

$$\text{Si-OH} \rightarrow \text{SiO-O}^{-} + \text{H}^{+}$$

The latter process will be seen later to be of value in the preparation of supported metal catalysts. Silica and alumina when well dried are useful desiccants, and alumina as well as other oxides such as titania are active catalysts for the dehydration of alcohols to alkenes because of their abilities to remove the elements of water from the reactant.

APPENDIX D

Position of the vibrational bands for the magnesium vanadate powders

Table D.1 Position of the observed vibrational bands for the Mg vanadate powders [83]

$Mg_3(VO_4)_2$			$Mg_2V_2O_7$			MgV_2O_6		
IR	Raman	assignment	IR	Raman	assignment	IR	Raman	assignment
fundamentals			975 sh		VO_3 str.			VO str.
			968	948				
915	897 sh	VO_4 str.	917	902			923	
	881 sh		880 sh	873		888		
861	862		840	845		840 sh	836	asVOV str.[a]
833	827		818					
730 sh	724 w		770 sh				731	V_3O str.
687	690 vw		690	630	VOV str.	695 sh		
610 br sh			668	625		655		
			575	570		620		
						575 sh		
						552	523	
485		VO_4 def. + lattice	462		VO_3 def. + lattice			
473	473		439	440		430	440	
	448			410				
415	411		402	403				
394	391		379	377		383		
370	351		362	354		350 br	332	
336	344		325 sh	335				
320	330		314	316				
	308 vw		302 sh	305		302	309	
291	290		285	282		286		
	275			268			268	
248	245 br		242	243				
	235 vw			228				
205	200		220	216		212	204	
			198	198		198	174	
			190	181				
171				165				
156	145			155		150	149	
				145				
136	137		130	131				
	122			113				
IR combination			1933	975 + 948				
			1910	968 + 948		1867	655 + 655 + 552	
							732 + 695 + 440	
1790	915 + 881		1790	917 + 873		1780	923 + 888	
							923 + 840	
1720	862 + 861		1694	873 + 818				
1672	861 + 827			845 + 840				
			1615	845 + 770				
1347	862 + 485		1430	818 + 620		1408	731 + 695	
			1210	630 + 575		1208	695 + 523	
			1116	569 + 575		1117	731 + 383	

str., stretch; def., deformation; sh, sharp; br, broad; as, asymmetric; vw, very weak. [a] Very asymmetric VOV bridges.

APPENDIX E

Data of Experiments

E.1 Data of Catalytic Reaction Test.

Table E-1 Data of Figure 5.1.

at (°C)	**% propane conversion**	**% selectivity to C1**	**% selectivity to C2**	**% selectivity to C3**
300	0.00	0.00	0.00	0.00
350	1.20	0.00	0.00	0.00
400	3.51	0.00	0.00	2.68
450	4.68	0.00	0.00	4.07
500	4.88	0.00	0.02	8.28
550	6.35	0.49	0.32	15.58
600	9.60	3.70	2.91	25.38

Table E-2 Data of Figure 5.2.

at (°C)	**% propane conversion**	**% selectivity to C1**	**% selectivity to C2**	**% selectivity to C3**
300	0.00	0.00	0.00	0.00
350	0.00	0.00	0.00	0.00
400	1.78	2.08	0.00	0.00
450	1.80	1.11	0.17	3.80
500	3.33	0.79	0.45	5.59
550	4.97	4.62	2.05	12.73
600	33.14	16.25	11.19	36.73

Table E-3 Data of Figure 5.3.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	3.56	0.00	0.00	1.94
350	4.95	0.00	0.00	3.47
400	4.78	0.00	0.00	7.48
450	5.12	0.00	0.00	14.64
500	7.39	0.00	0.00	20.19
550	11.09	0.26	0.14	22.67
600	17.57	1.11	0.91	23.89

Table E-4 Data of Figure 5.4.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	1.05	1.28	0.00	0.00
350	1.25	3.23	0.00	0.00
400	2.46	0.49	0.00	0.00
450	5.35	0.27	0.00	1.84
500	7.87	0.22	0.20	4.18
550	16.64	0.30	0.95	6.92
600	35.43	1.11	3.60	9.01

Table E-5 Data of Figure 5.5.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	6.05	0.00	0.00	3.29
350	9.72	0.00	0.00	14.68
400	20.64	0.10	0.04	32.23
450	51.95	0.11	0.06	21.17
500	65.19	0.51	0.17	16.53
550	68.19	1.26	0.52	17.64
600	77.90	6.06	3.15	17.41

Table E-6 Data of Figure 5.6.

at(^{0}C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	0.00	0.00	0.00	
350	1.49	0.00	0.00	77.98
400	8.89	0.00	0.00	57.84
450	30.30	0.00	0.11	40.21
500	55.24	0.68	0.39	29.03
540	59.26	1.04	1.03	29.03
560	60.34	1.08	1.24	27.82
600	67.30	5.22	4.31	25.56

Table E-7 Data of Figure 5.7.

at (^{o}C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	0.00	0.00	0.00	
350	0.90	1.15	0.00	73.85
400	5.86	0.35	0.04	56.21
450	21.98	0.22	0.70	41.81
500	49.40	0.31	0.09	23.74
550	63.56	0.79	0.19	18.76
600	66.98	1.43	0.79	20.09

Table E-8 Data of Figure 5.8.

hold time (min.)	% propane conversion	% selectivity to propene
10	36.61	35.16
20	35.92	35.30
30	37.02	34.55
40	38.81	33.41
50	38.14	33.76
60	36.14	35.16
80	36.52	34.95
100	35.17	34.94
120	37.85	33.75
140	36.73	33.62
160	34.28	35.85
180	38.15	34.55
200	36.11	34.88
220	36.34	33.91
240	36.77	34.21

Table E-9 Data of Figure 5.9.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	0.00	0.00	0.00	
350	0.34	4.75	0.00	31.48
400	2.00	1.33	0.00	28.34
450	7.21	0.21	0.10	25.65
500	18.13	0.33	0.44	30.91
550	39.19	3.27	3.08	21.55
600	93.03	24.07	15.72	7.88

Table E-10 Data of Figure 5.10.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	0.00	0.00	0.00	
350	0.44	2.10	0.00	44.75
400	2.61	0.73	0.00	39.37
450	7.70	0.20	0.11	41.04
500	19.43	0.23	0.40	37.41
550	37.57	1.97	2.31	26.96
600	97.17	22.46	15.17	5.22

Table E-11 Data of Figure 5.11.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	0.57	5.08	0.00	8.18
350	2.80	1.35	0.00	16.23
400	7.30	0.43	0.06	24.96
450	17.34	0.18	0.11	23.40
500	40.69	0.49	0.51	19.51
550	97.37	16.74	13.18	3.70
600	98.97	19.97	11.36	2.26

Table E-12 Data of Figure 5.12.

at (°C)	% propane conversion	% selectivity to C1	% selectivity to C2	% selectivity to C3
300	0.00	0.00	0.00	
350	0.84	0.00	0.00	64.22
400	5.80	0.15	0.07	35.65
450	17.13	0.06	0.14	31.84
500	36.81	0.07	0.29	20.14
550	64.43	0.81	1.32	20.22
600	79.37	9.56	7.40	14.68

E.2 Data of Thermal Analysis.

Table E-13 Data of Figure 5.18.

$T(^{0}C)$	area of O_2	area of H_2O
100	90	3116
125	74	3370
157	77	3614
182	66	2688
207	75	2316
230	67	2391
258	69	2871
280	135	3771
305	100	6026
331	144	11473
359	141	14629
381	137	13620
405	143	3697
431	134	2580
458	159	2274

Table E-14 Data of Figure 5.19

$T(^0C)$	area of O_2	area of H_2O
86	75	14712
95	77	5729
120	63	6590
177	33	5590
202	79	3860
227	67	3518
253	65	3616
275	83	3218
301	142	3363
325	142	3654
356	155	4169
377	145	3141
401	128	2686
427	108	2396

VITA

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