



RESULT AND DISCUSSION

The results from the experiments in chapter 5 were presented in tables and figures. The results were also discussed in the followings.

6.1 Properties of the In-house Catalysts

The properties of the in-house catalysts were presented in Table 6.1

Table 6.1 Properties of the In-house Catalysts

Catalyst No.	Type of Catalyst	Support	% Ni	Particle size (mesh)
1	Impregnated Ni	alumina	9.30	100-150
2	Impregnated Ni	alumina	9.30	325-400
3	Impregnated Ni	alumina	16.63	325-400
4	Raney Ni	non	47.09	

6.2 Screening commercial catalysts

The comparative results of hydrogenation with different catalysts presented in Table 6.2 showed the superiority of the G 53D catalyst (Expt. No.6) over other nickel catalysts. This

Table 6.2 The Comparative Results of Hydrogenation of Castor oil with Different Catalysts
 (H₂ Pressure 150 psig, Reaction Temperature 140°C, Agitation 800 rpm, Concentration
 of Catalyst 0.2% Ni/Oil, Reaction Period 5 hours)

Expt. No.	Type of Catalyst	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. (°c)
1	Ni 3712 p	2.3	-	0.81	81
2	Ni 5132 P	3.23	-	0.97	80
3	Ni 3609 F	1.63	-	0.78	80
4	Ni 0104 P	26.81	-	0.75	semisolid
5	G 95 E	4.27	-	0.74	80
6	G 53 D	1.54	137.85	0.74	84

catalyst produced a product with a minimum iodine value of 1.54 and a maximum melting point of 84°C which is comparable to commercial castor wax (Table 6.10). Therefore, it should be noted that the G53D catalyst was suitable for hydrogenation of castor oil.

6.3 Selecting an optimum operating conditions by varying the parameters

Because of its superior activity, the G53D catalyst was used in determining the optimum operating condition

6.3.1 Effect of Reaction Temperature. From the results given in Table 6.3 and Figure 6.1 it was seen that increasing the reaction temperature from 80°C to 180°C decreased both the iodine value and the hydroxyl value but increased the acid value. Increasing the reaction temperature increased the rate of hydrogenation. At temperature 80°C the rate of hydrogenation was very low and was increased rapidly with increasing temperature from 80°C to 100°C. The dehydroxylation was increased rapidly with increasing reaction temperature especially at 180°C so the reaction temperature strongly effected on dehydroxylation. The increase in the acid value showed the presence of such side reactions as hydrolysis of fats to fatty acids and glycerine. The suitable temperature was 140°C.

6.3.2 Effect of H₂ Pressure.

The results presented in Table 6.4 and Figure 6.2 indicated that increasing the H₂ pressure from 75 psig to 175 psig decreased the iodine value and the acid value but decreased the loss of hydroxyl value. Increasing H₂ pressure increased the rate of

Table 6.3 Effect of Reaction Temperature on Hydrogenation of Castor Oil (H_2 Pressure 150 psig, Agitation 800 rpm, Concentration of Catalyst 0.2% Ni/oil Reaction Period 2 hours, G 53 D Catalyst)

Expt. No.	Reaction Temperature ($^{\circ}C$)	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. ($^{\circ}C$)
7	80	49.50	159.68	0.70	semi solid
8	100	6.63	155.84	0.73	84
9	120	4.70	152.09	0.83	85
10	140	3.69	147.20	0.76	86
11	180	3.40	131.03	1.02	82

Figure 6.1 Effect of reaction temperature

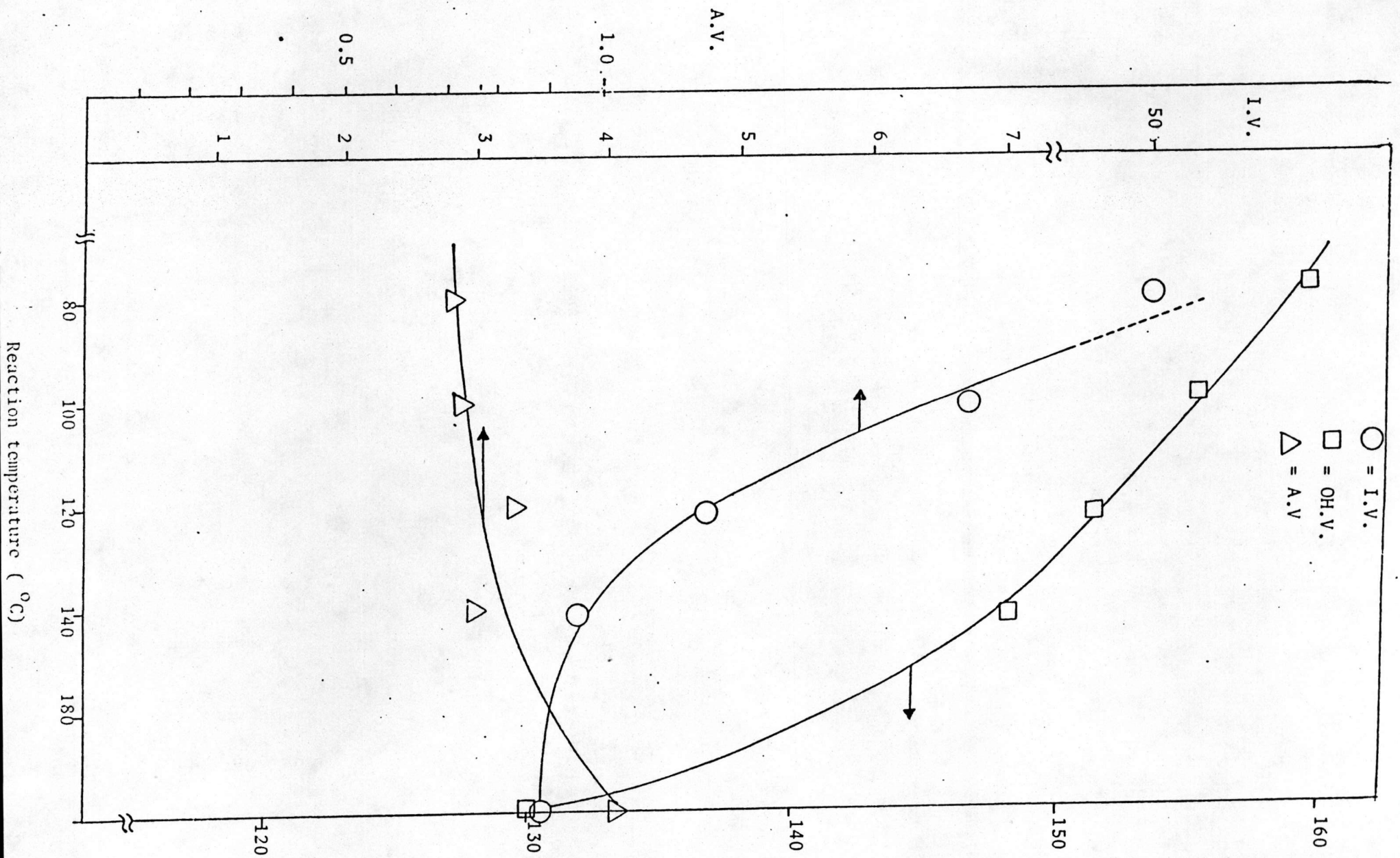
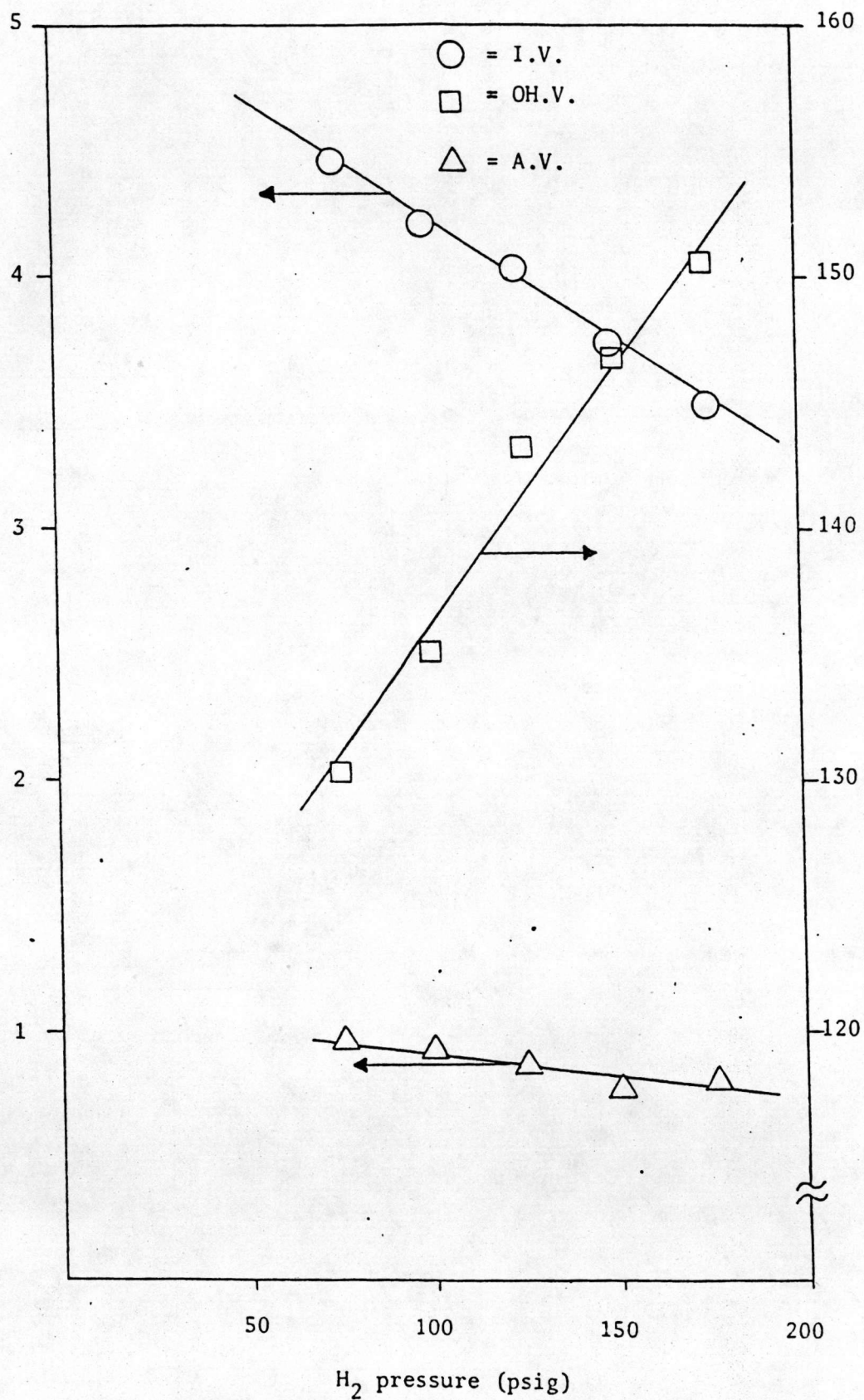


Table 6.4 Effect of H₂ Pressure on Hydrogenation of Castor Oil (Reaction Temperature 140°C, Agitation 800 rpm, Concentration of Catalyst 0.2 % Ni/oil, Reaction Period 2 hours, G 53 D Catalyst)

Expt. No.	H ₂ Pressure (psig)	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. (°C)
12	75	4.43	130.63	0.97	81
13	100	4.21	134.97	0.97	82
14	125	4.03	143.25	0.89	84
10	150	3.69	147.20	0.76	86
15	175	2.48	150.59	0.82	87

Figure 6.2 Effect of H₂ pressure

dissolution of hydrogen in oil and thus increased the rate of hydrogenation but decreased the rate of dehydroxylation. At higher H_2 pressure the side reaction which increased the acid value was reduced and the hydrogenation of the double bond was the predominant reaction. Therefore, higher H_2 pressure was the suitable condition for hydrogenation of castor oil. The suitable H_2 pressure was 175 psig.

6.3.3 Effect of Reaction Period.

As shown in Table 6.5 and Figure 6.3, the main reduction of the double bond in castor oil took place in 1 hour, after which the process was slow and in 2 hours a product with desired properties was obtained. Increasing the reaction period from 1 hour to 5 hours, the loss of the hydroxyl value was increased but the acid value was unaffected.

6.3.4 Effect of Concentration of Catalyst.

The results in Table 6.6 and Figure 6.4 showed that increasing the concentration of catalyst from 0.05% to 0.5% decreased the iodine value but increased the loss of the hydroxyl value and the acid value. Increasing the concentration of catalyst from 0.05% to 0.2% increased the rate of hydrogenation but on increasing concentration of catalyst from 0.2% to 0.5%, the rate of hydrogenation was increased only slightly. The increase in the loss of hydroxyl value was probably due to hot spots on the catalyst surface. The use of 0.2% nickel catalyst resulted in a suitable product. (Expt. No. 10)

6.3.5 Effect of Agitation.

The results given in Table 6.7 and Figure 6.5 showed that increasing agitation from 200 rpm. to 800 rpm decreased the

Table 6.5 Effect of Reaction Period on Hydrogenation of Castor Oil (H_2 Pressure 150 psig, Reaction Temperature 140°C, Agitation 800 rpm, Concentration of Catalyst 0.2% Ni/oil, G 53 D Catalyst)

Expt. No.	Reaction Period (hr.)	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. (°C)
16	1	5.34	152.16	0.76	85
10	2	3.69	147.20	0.76	86
17	3	2.45	143.47	0.77	85
6	5	1.54	137.85	0.74	84

Figure 6.3 Effect of reaction period

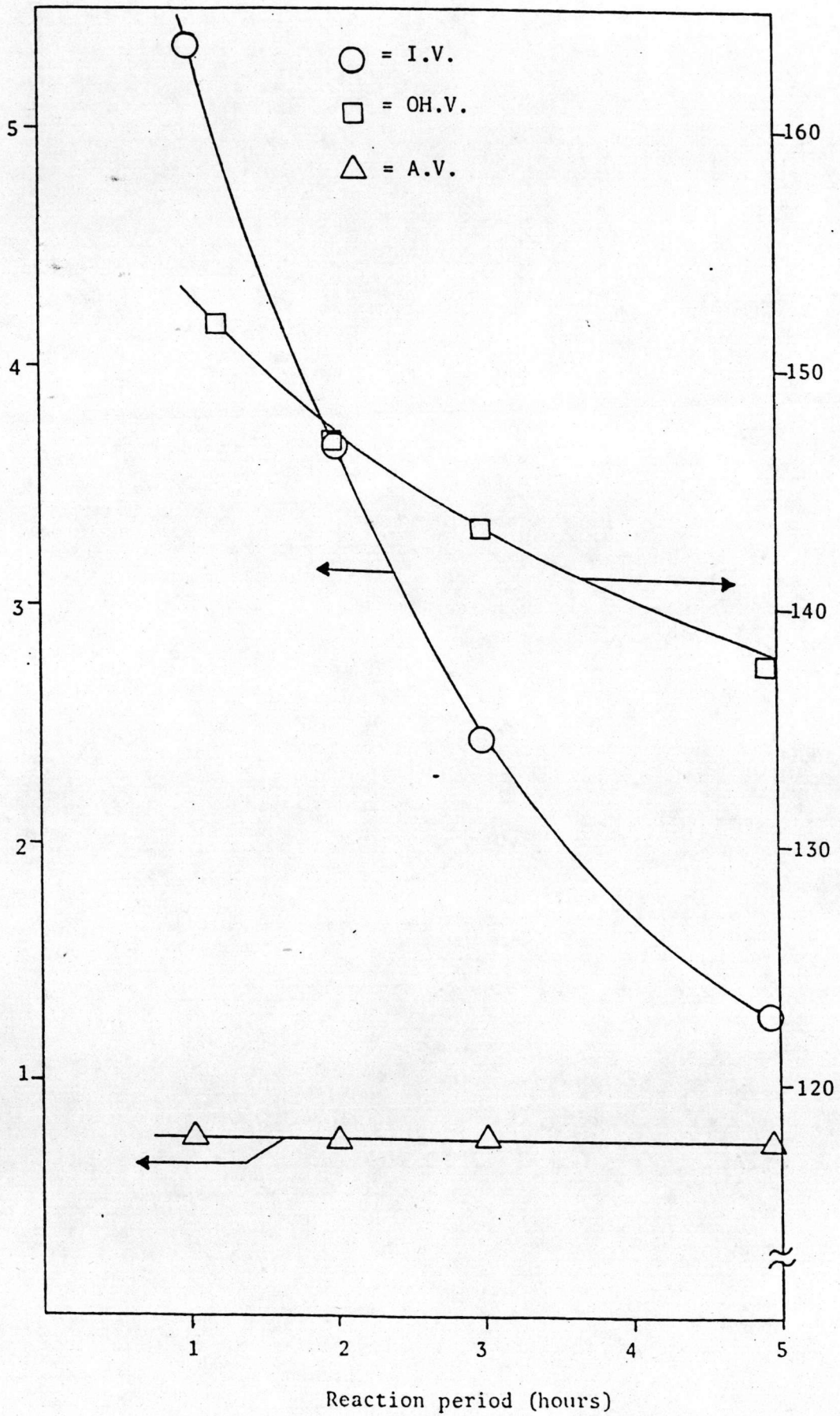


Table 6.6 Effect of Concentration of Catalyst on Hydrogenation of Castor Oil (H_2 Pressure 150 psig, Reaction Temperature $140^\circ C$, Agitation 800 rpm, Reaction Period 2 hours, G 53 D Catalyst)

Expt. No.	Concentration of Catalyst (% Ni/oil, wt/wt.)	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. ($^\circ C$)
18	0.05	7.10	150.51	0.75	83
10	0.2	3.69	147.20	0.76	86
19	0.5	3.44	138.55	0.90	83

Figure 6.4 Effect of concentration of catalyst in oil

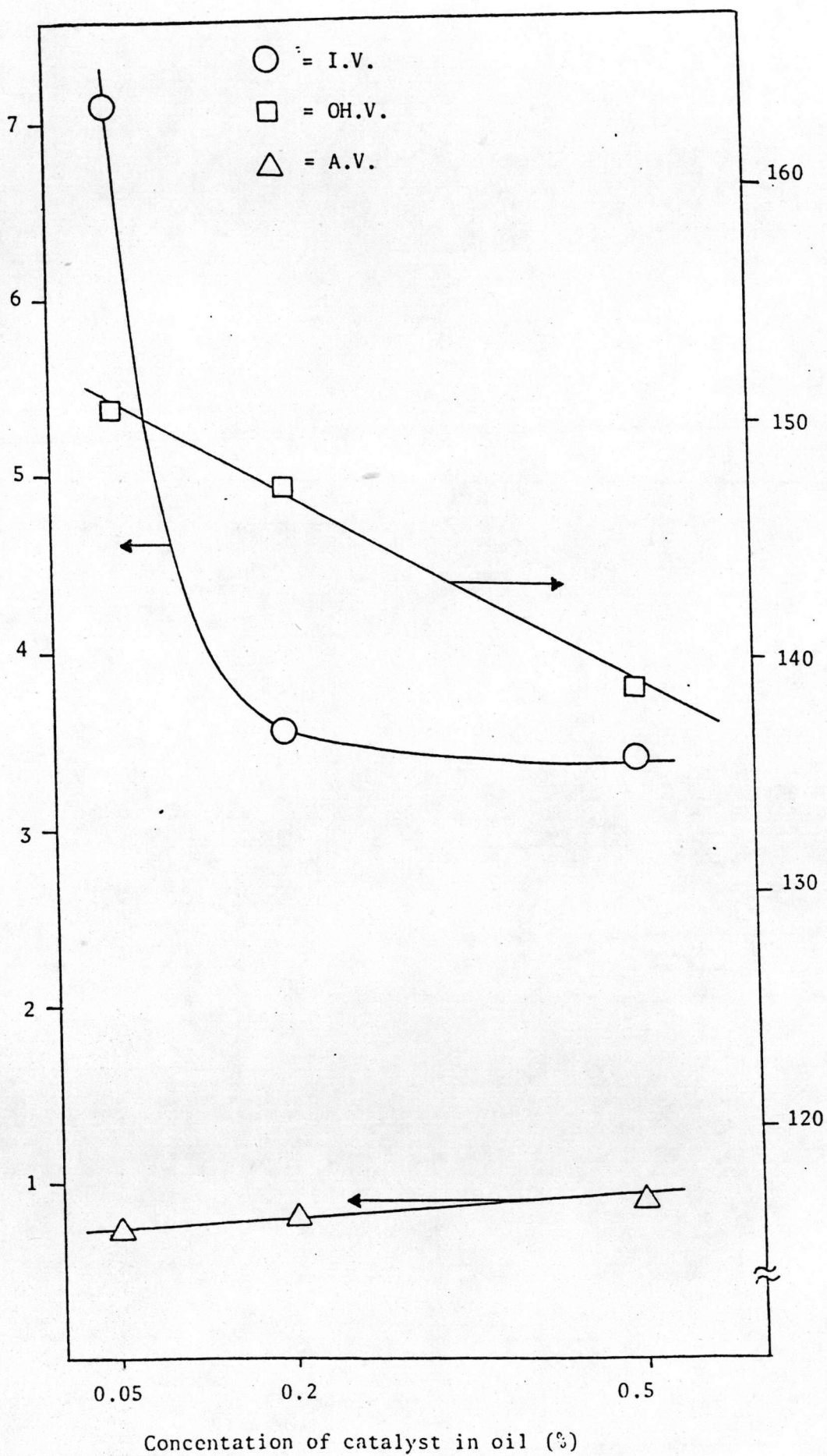
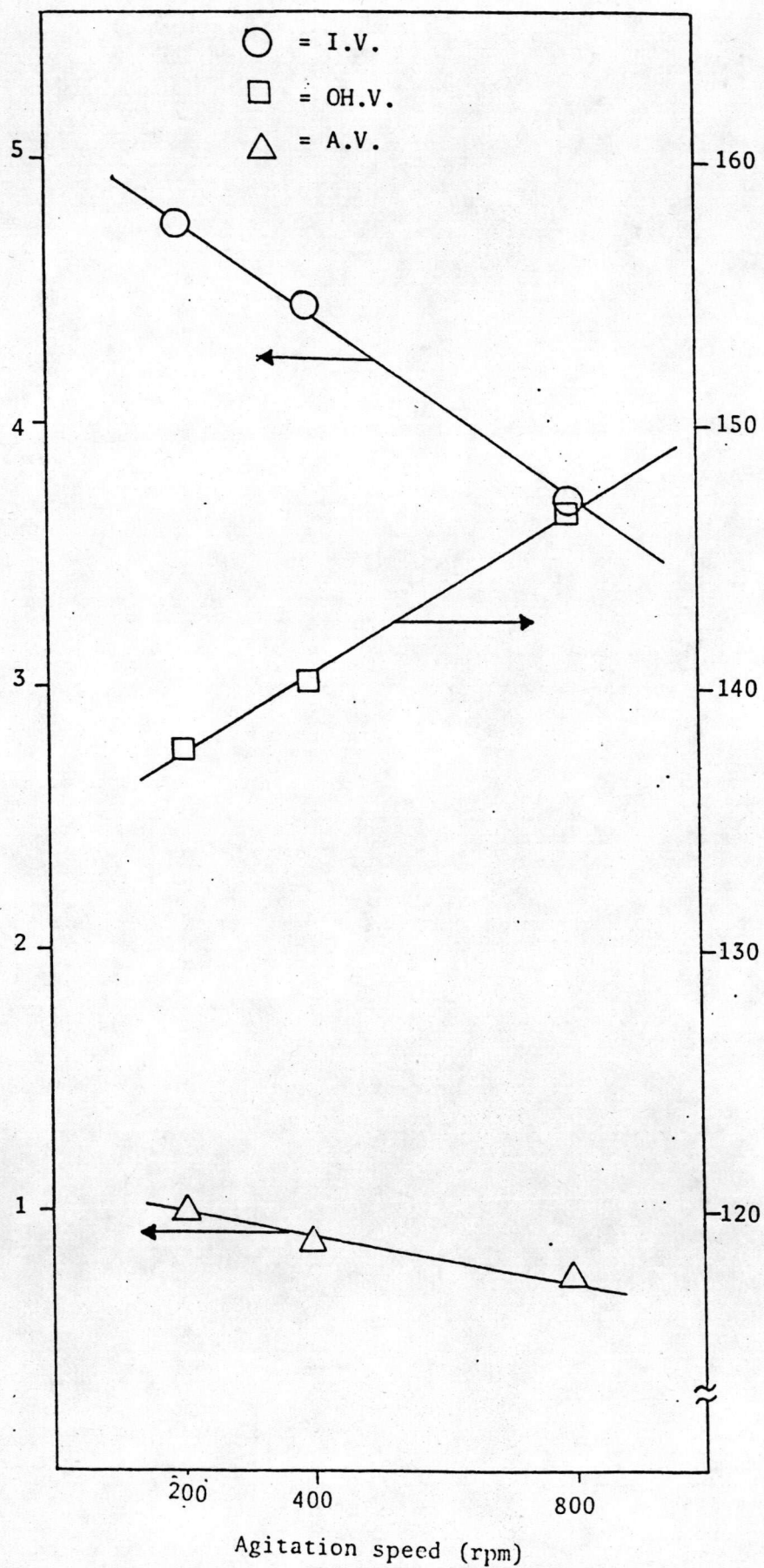


Table 6.7 Effect of Agitation on Hydrogenation of Castor Oil (H_2 Pressure 150 psig, Reaction Temperature $140^\circ C$, Concentration of Catalyst 0.2% Ni/oil, Reaction Period 2 hours, G 53 D Catalyst)

Expt. No.	Agitation (rpm)	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. ($^\circ C$)
20	200	4.74	137.67	1.01	83
21	400	4.47	140.29	0.90	83
10	800	3.69	147.20	0.76	86

Figure 6.5 Effect of agitation



iodine value, the acid value and the loss of the hydroxyl value. Increasing agitation increased the dissolution rate of hydrogen in oil and adsorption rate of hydrogen in oil on the catalyst surface because it more quickly whipped the hydrogen from gas phase into the reaction mixture and decreased the film resistance of the mass and heat transfer around the catalyst particle so the rate of hydrogenation was increased. Increasing agitation not only increases hydrogenation but also decreases both dehydroxylation caused by hot spots or over heat on catalyst surface, and hydrolysis.

6.4 Comparing the in-house catalyst to the best commercial catalyst.

The comparative results of hydrogenation using different catalysts given in Table 6.8, it was seen that the best catalyst from the in-house catalysts was Impregnated Ni No.2 (Exp. No.23). It produced a product with an acid value of 0.99 and melting point 84°C. The G53D catalyst (Expt. No.10) gave a product with an iodine value of 3.69, a hydroxyl value of 147.20, an acid value of 0.76 and melting point 86°C. The properties of the product obtained by the Impregnated Ni No.2 was almost the same as the product from the G 53D. It was expected that the activity of the G53D was better than Impregnated Ni No.2 because of the smaller particle size of the G 53D ($< 3 \mu\text{m}$) than the Impregnated Ni No.2 (325-400 mesh, 43-38 μm)

6.5 The Effect of particle size and Ni loading of the Impregnated Nickel Catalysts

The results given in the Table 6.9 showed that the product (I.V. 4.68, OH.V. 139.11, A.V.0.99, m.p. 84°C) from the Impregnated

Table 6.8 Comparing the In-house Catalysts to the best commercial catalyst (H_2 Pressure 150 psig, Reaction Temperature $140^\circ C$, Agitation 800 rpm, Concentration of Catalyst 0.2% Ni/oil, Reaction Period 2 hours)

Expt. No.	Type of Catalyst	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. ($^\circ C$)
10	G 53 D	3.69	147.20	0.76	86
22	Impregnated Ni No.1	18.14	131.19	2.02	78
23	Impregnated Ni No.2	4.68	139.11	0.99	84
24	Impregnated Ni No.3	4.97	136.09	1.05	83
25	Raney Ni No.4	7.89	138.51	0.72	82

Table 6.9 Effect of Particle size and Nickel Loading of the Impregnated Ni Catalyst on Hydrogenation of Castor Oil (H_2 Pressure 150 psig, Reaction Temperature $140^\circ C$, Agitation 800 rpm, Concentration of Catalyst 0.2% Ni/oil, Reaction Period 2 hours)

Expt. No.	Type of Catalyst	Properties of Hydrogenated Product			
		I.V.	OH.V.	A.V.	m.p. ($^\circ C$)
22	Impregnated Ni 9.30% 100-150 mesh size	18.14	131.19	2.02	78
23	Impregnated Ni 9.30 % 325-400 mesh size	4.68	139.11	0.99	84
24	Impregnated Ni 16.63 % 325-400 mesh size	4.99	136.09	1.05	83

Table 6.10 Properties of Commercial Castor Waxes and Some Resulting Castor Waxes

Product Name	I.V.(Wij)	OH.V.	A.V.	m.p.(°C)
Castorwax *	3.00	158.00	2.00	87
Castorwax F-1 *	9.00	158.00	2.00	84
Castorwax MP 80 *	29.00	158.00	2.00	80
Castorwax MP 70 *	38.00	158.00	2.00	70
Expt. No. 6 **	1.54	137.85	0.74	84
Expt. No. 23 ***	4.68	139.11	0.99	84

* from Baker Castor Oil Company

** from the G53D catalyst,

*** from Impregnated 9.3% Ni, 325-400 mesh



Ni catalyst with particle size 325-400 mesh (Expt. No.23) had better properties than the product (I.V. 18.14, OH.V. 131.19, A.V. 2.02, m.p. 78°C) from the Impregnated Ni catalyst with particle size 100-150 mesh (Expt. No.22). Decreasing the particle size of catalyst decreased both iodine value and the loss of hydroxyl value. The change of the hydrogenation rate with particle size showed the presence of mass (and heat) transfer effect on the reaction rate. Increasing the catalyst particle size will shift the reaction from the reaction-controlled regime toward transition regime and finally intra-particle mass transfer controlled regime as the longer path way for the diffusion of reactants and product into and from active surface in the catalyst pores is required. Therefore, the particle size of catalyst should be small as possible (not difficult for filtering) to eliminate this effect. The product with an iodine value of 4.68, a hydroxyl value 139.11, an acid value of 0.99 and melting point 84°C obtained by the Impregnated Ni 9.3% (Expt. No.23) was compared to the product with an iodine value of 4.99, a hydroxyl value 136.09, an acid value of 1.05 and melting point 83°C obtained by the Impregnated Ni 16.63% (Expt. No.24). It was seen that the activity of the Impregnated Ni 9.3% was little better than the activity of the Impregnated Ni 16.63%. It was probably because increase in nickel loading would not proportional increase in the active surface area and some pores of the catalyst could be filled when this alumina support was re-impregnated. The best of the in-house catalysts was impregnated 9.3 % nickel catalyst in particle size range 325-400 mesh.