

**Investigation of the Heterogeneous  
Catalytic Conversion of Cycloalkanes Over  
Nickel-Alumina /  $\alpha$ -Alumina Catalyst  
Part I – The Catalytic Dehydrogenation  
of Cyclohexane**

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**ABSTRACT.** The catalytic conversion of cyclohexane on a Ni-Al<sub>2</sub>O<sub>3</sub>  $\alpha$ -Al<sub>2</sub>O<sub>3</sub> catalyst (31.1% of Ni by wt.) was studied using a flow technique. The catalytic activity was measured in the temperature range 400 to 480°C at a space velocity of 9.4-17.8 X10<sup>-2</sup> min<sup>-1</sup> under normal pressure (1 atm). The gaseous and liquid products of the conversion reaction were analysed chromatographically. The gaseous products were hydrogen and methane, and the liquid products were benzene and cyclohexene together with unreacted cyclohexane. The kinetics of the gaseous products and of cyclohexane revealed a zero-order reaction, but that of cyclohexene and benzene did not. The apparent activation energy was found to be 23.76  $\pm$  0.2 kcal mol<sup>-1</sup> independent of the space velocity.

The activity and selectivity were discussed referring to the Ni-Al<sub>2</sub>O<sub>3</sub> solid catalyst prepared before (Ni-A).

It is known that the activity and selectivity of a certain solid catalyst are influenced by different factors among which are the method of preparation and the nature of the support materials (Anderson 1975 and Krylov 1967).

In the previous work (Ezzo *et al.* 1981) the kinetics of heterogeneous catalytic conversion of cyclohexane was studied over nickel-alumina catalyst prepared by coprecipitation in flow system under normal pressure, and a polymolecular mechanism was proposed. According to this mechanism, an intermediate

polymolecular catalytic complex is formed which may decompose in different ways, according to the kinetic conditions, to give the reaction products. It is of interest to study the influence of the method of preparation of nickel alumina catalyst on the nature of the condensating products of heterogeneous catalytic decomposition of cyclohexane giving rise to one of the different metal support interactions under the same experimental conditions (Rudenko 1971).

In this investigation the kinetics of heterogeneous catalytic process of dehydrogenation of cyclohexane was studied over a catalyst of nickel-alumina supported on  $\alpha$ -alumina.

Some surface and textural characteristics of the prepared solid were reported.

## 1. Experimental Details

### 1.1. Materials

The nickel-alumina supported on alumina solid catalyst (Ni-A/A) was prepared from aluminium oxide (Merck; highly pure for polishing). The granules of 0.5 mm diameter were calcined at 1100°C in an electric oven for 4 h. The adsorptive water capacity of alumina was examined at 21.5°C (ml of H<sub>2</sub>O/g of alumina). A solution of 0.17 g of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O (Laboratory BDH reagent) and 0.23 g of Al(NO<sub>3</sub>)<sub>3</sub>·9H<sub>2</sub>O (ALEC Arabic laboratory reagent Egyptian Company) dissolved in 20 ml of distilled water, according to the adsorptive capacity of alumina, was added to 15 g of the alumina granules, mentioned previously, with constant stirring for 30 m. The resulting paste was dried in an electric oven at 140°C for 2 h. The solid was crushed in a pyrex mortar to the desired grain size (0.25-0.5 mm), which was chosen for both catalytic and surface measurements.

Gravimetric analysis of the solid catalyst using dimethylglyoxime, showed that the nickel content of the catalyst is 31.1% by weight.

The cyclohexane was purified by keeping the product obtained from Rectapure Prolabo Laboratory Chemicals, over a Linde Va molecular sieve for 72 h and then distilling it at a constant boiling point of 80.3°C. The distillate was chromatographically pure.

### 1.2. Apparatus and technique

The catalytic activity was measured using a flow technique. The reactant was introduced by a microdose pump (Unipan 335 A) and the speed of feeding was

varied in the range  $9.4$  to  $17.8 \times 10^{-2} \text{ min}^{-1}$ . The reaction temperature of the different experiments was in the range  $400$ - $480^\circ\text{C}$ . The catalyst sample (of volume  $1 \text{ ml}$ ) was introduced into a silica tube reactor with an internal diameter of  $12 \text{ mm}$ . The reactor was heated by a tubular electric furnace (Rock A), the temperature of which was controlled to  $\pm 0.1^\circ\text{C}$ . The reactor and the furnace were mounted in an inclined position.

The gaseous and liquid products were analysed in a programmed gas-liquid chromatograph with a heated dual flame ionization detector (Pye series 104) on 2/10 PEGA using nitrogen as the carrier gas.

### ***1.3. Activation of the catalyst sample***

Each solid sample was activated in situ by calcination at  $500^\circ\text{C}$  for  $4 \text{ h}$  in a current of dry air free from  $\text{CO}_2$  (Ni-A/AO), or in a current of dry hydrogen free from  $\text{H}_2\text{O}$  (Ni-A/AH). The activated catalyst was then cooled from the activation temperature to the catalytic reaction temperature. The activation temperature was chosen to be  $500^\circ\text{C}$  since thermogravimetric analysis showed that the catalyst Ni-A/A lost its water of crystallization and water of hydroxylation at  $452^\circ\text{C}$ .

### ***1.4. Adsorption measurements and X-ray diffraction analysis***

#### ***1.4.i. Adsorption measurements***

The adsorption-desorption isotherm of nitrogen at  $-195^\circ\text{C}$  was determined using a conventional apparatus. The adsorption was monitored until near saturation and then desorption was monitored down to the relative pressures at which the desorption and adsorption branches coincided. The sample was degassed at  $300^\circ\text{C}$  for  $3 \text{ h}$  under a reduced pressure of  $10^{-5} \text{ Torr}$  before any adsorption measurements were made.

#### ***1.4.ii. X-ray diffraction analysis***

The X-ray diffraction patterns of the solid sample and its products after calcination for  $4 \text{ h}$  at  $400^\circ$ ,  $500^\circ$  and  $750^\circ\text{C}$  in dry air were obtained using a Philips model PW 1050 X-ray diffractometer at  $\lambda = 1.54 \text{ \AA}$ . A  $\text{CuK}_\alpha$  target with a nickel filter was used. All the diffraction patterns were measured at room temperature; the peak was recorded in a single run.

## 2. Results and Discussion

### 2.1. Textural characteristics of Ni-A/A catalysts

The surface area  $S_{\text{BET}}$  was calculated using the conventional Brunauer-Emmett-Teller (BET) equation (Brunauer *et al.* 1938). This surface area value was compared with the area obtained by the  $V_1-t$  method (Lippens *et al.* 1964, Lippens and De Boer 1965).

The adsorption-desorption isotherm of nitrogen at  $-195^\circ\text{C}$  on Ni-A/A catalyst is shown in Fig. 1(a). It appears to be type II isotherm for nonporous solids of the Brunauer, Deming, Deming and Teller classification (Brunauer *et al.* 1940 and Gregg and Sing 1982) with a knee at low values of  $p/p^\circ$  and a sloping plateau that extends to high values of adsorption according to the BET equation. Fig. 1(b) shows the linear plot for nitrogen adsorption according to the BET equation. The specific surface area estimated from the BET plot was found to be equal to  $152 \text{ m}^2\text{g}^{-1}$ .

### 2.2. X-ray study of the solid samples

The d spacing was estimated at each temperature ( $400^\circ$ ,  $500^\circ$  and  $750^\circ\text{C}$ ) and compared with the values given in the ASTM cards Powder Diffraction File (1978). The results obtained are given in Table 1 for Ni-A/A solid.

It follows from Table 1, Fig. (2) and the ASTM cards that five main peaks appeared at all the calcination temperatures; these peaks are characteristic for NiO, with rhombic crystal structure, the other peaks obtained were characteristic for  $\alpha\text{-Al}_2\text{O}_3$ .

The crystallite size (t value) was calculated for each sample at 100% intensity by applying the following expression (Fitton and Griffith 1968).

$$\beta = K \lambda / t \cos \theta$$

where  $\beta$  is the peak width at half-intensity, t is a linear dimension of the crystallite and K is a numerical constant of the order of unity.

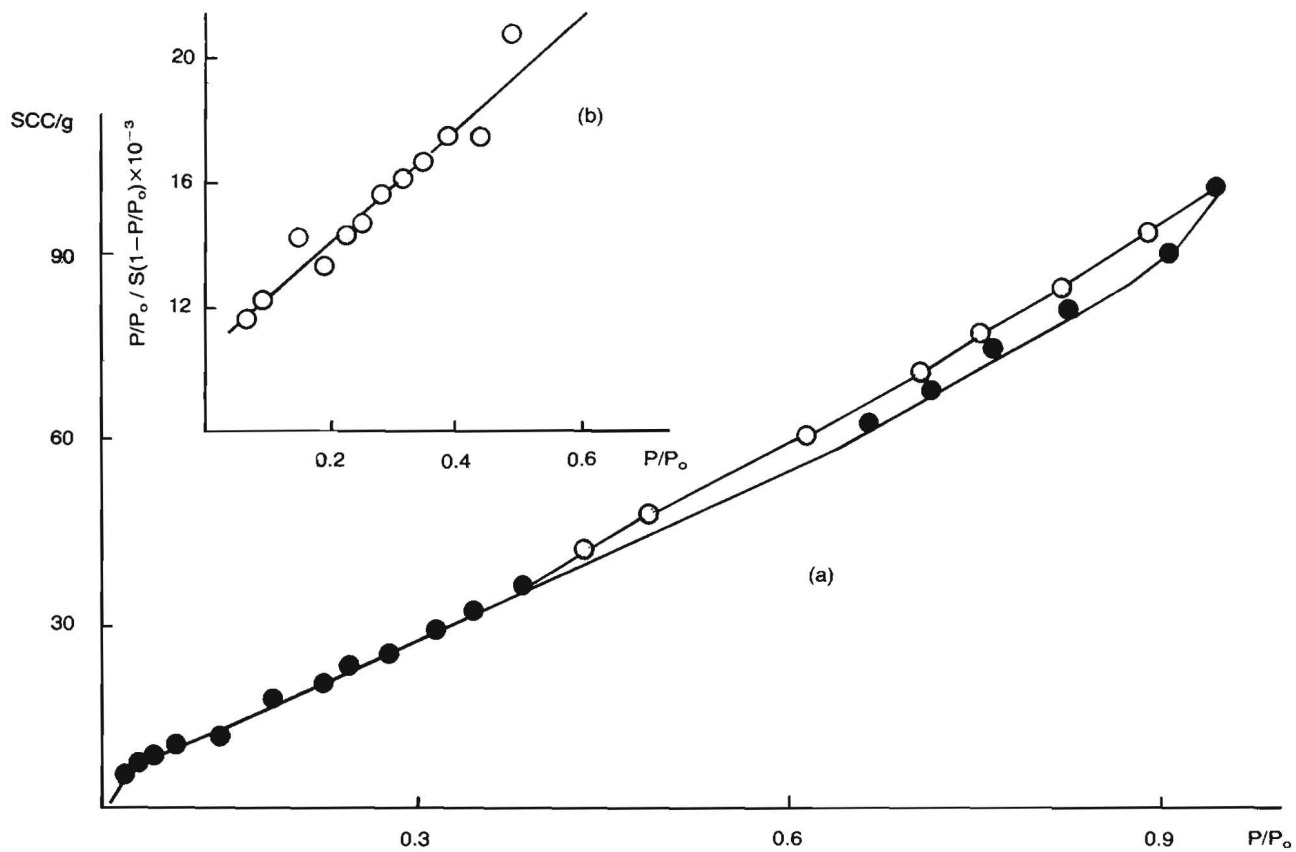


Fig. 1. Surface characteristics for Ni-A/A catalyst heated at 500°C.  
 (a) adsorption - desorption isotherm. (b) BET plot.

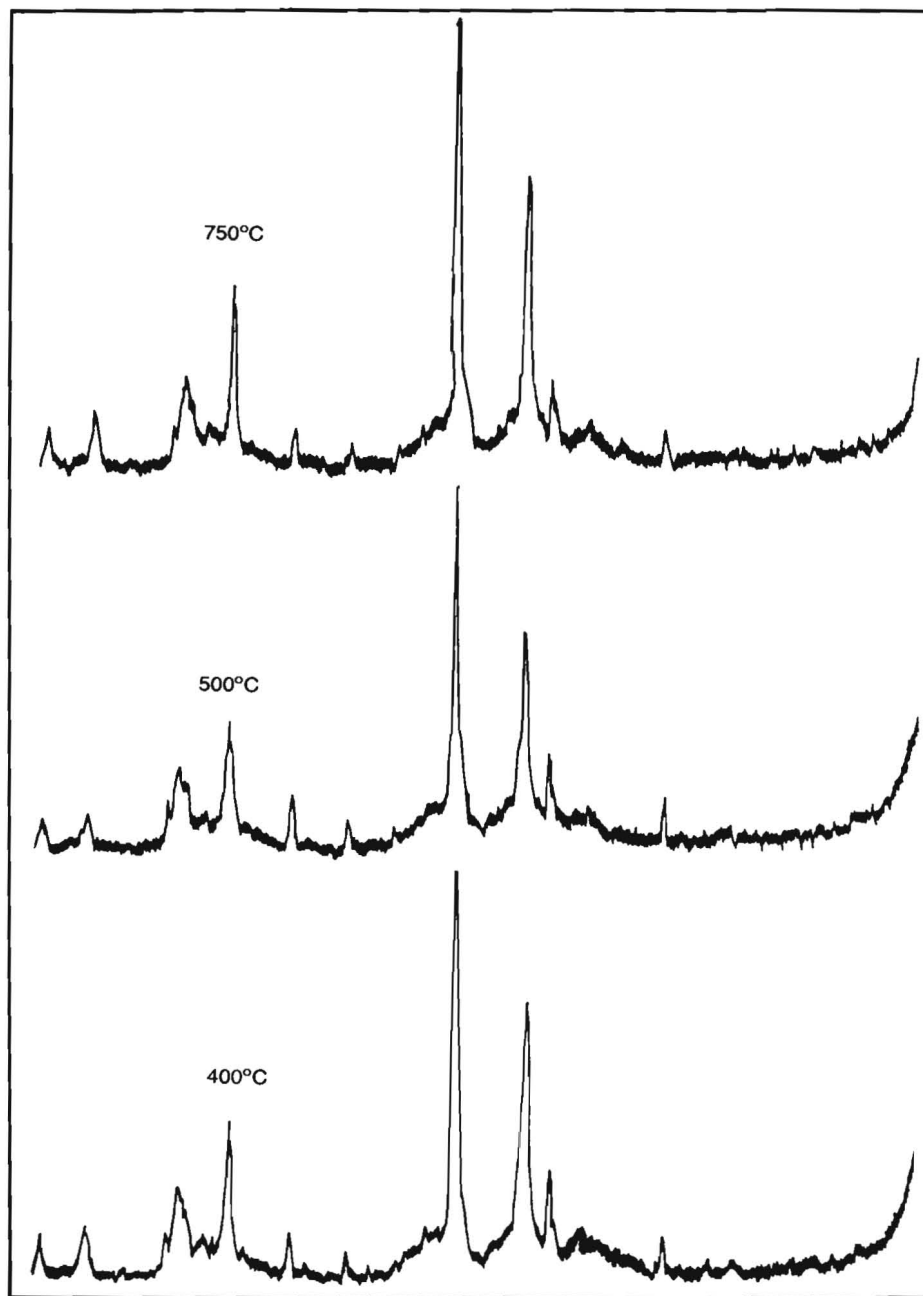


Fig. 2. X-ray diffraction patterns for Ni-A/A catalyst, thermally treated at 400, 500 and 750°C.

### 2.3. Effect of space velocity ( $f$ )

The reaction temperature was monitored at 400°C, and a constant flow rate of cyclohexane vapour was allowed to pass over 1 ml of the Ni-A/A solid at space velocities from 9.4 to  $17.8 \times 10^{-2} \text{ min}^{-1}$ . This experiment was repeated at 430°, 460° and 480°C over fresh portions of Ni-A/AO or Ni-A/AH catalysts.

The gaseous products were found to be hydrogen and methane, and the liquid products were cyclohexene and benzene at 400°, 430°, 460° and 480°C on Ni-A/AO and Ni-A/AH catalysts, the amount of each product was affected by the kinetic conditions, Table 2. The volume of the gaseous products was determined at various intervals. When this volume was plotted against time, a straight line was obtained, the slope of which gave the rate of formation of the gaseous products, which was then corrected to standard temperature and pressure ( $V^\circ$ , in millilitres per minute).

Fig. (3) shows the effect of variation in the time of contact  $\tau$  on the rate of conversion of cyclohexane at 460°C [(a) over Ni-A/AO and (b) over Ni-A/AH catalysts].

It follows from this figure and the results given in Table 2 that the rate of formation of gaseous products was independent on the rate of flow of the cyclohexane *i.e.* the process of conversion of cyclohexane is a zero-order reaction. This indicates that the catalyst surface, or at least the active part of it, is saturated with chemisorbed molecules. Moreover, the rate determining step is neither the diffusion of the reactants or products nor their adsorption and desorption, but it is one of those of the surface reactions. However, the percent formation of cyclohexene and benzene was decreased with the rate of flow of cyclohexane at 400°, 430°, 460° and 480°C over Ni-A/AO and Ni-A/AH catalysts.

According to the results obtained and given in Table 2 (column 3) the amount of gaseous products ( $V^\circ$ ) on Ni-A/AH catalyst at 400°, 430° and 460°C exceeded that on Ni-A/AO by 25, 28.6 and 21.2% respectively. However, at 480°C the results obtained were deviated.

This means that the degradation dehydrogenation of cyclohexane to cyclohexene and benzene was favoured on Ni-A/AO at 400°, 430° and 460°C. The formation of liquid products at 480°C had different character. Besides, in all cases the stoichiometry of the investigated process was not fulfilled at 400°, 430°, 460° and 480°C.

The weight of the catalyst was found to increase after the experiment, the apparent gain in the mass of the catalyst is given in Table 2. The apparent gain in

weight had its highest value at 430°C for Ni-A/AO, and it was found to increase with temperature from 400 to 480°C for Ni-A/AH. Moreover, the increase in the mass of Ni-A/AO exceeded that of Ni-A/AH at 400° and 430°C and it was lower at 460° and 480°C.

**Table 1.** The values of the d spacing and the ASTM values for the Ni-Al<sub>2</sub>O<sub>3</sub>/α-Al<sub>2</sub>O<sub>3</sub> catalyst

Sample at t°C	N°	2θ	d	ASTM value	β	t (Å)
A(400)	1	25.3	3.52	3.48	—	4.14
	2	35.1	2.55	2.55	—	
	3	37.2	2.41	2.41	—	
	4	43.2	2.09	2.09	0.4	
	5	52.4	1.74	1.74	—	
	6	57.3	1.60	1.60	—	
	7	62.6	1.48	1.48	—	
	8	64.8	1.43	1.40	—	
	9	67.0	1.39	1.37	—	
	10	75.3	1.26	1.26	—	
	11	79.4	1.20	1.20	—	
B(500)	1	25.6	3.47	3.48	—	3.32
	2	35.2	2.54	2.55	—	
	3	37.2	2.41	2.41	—	
	4	43.2	2.09	2.09	0.5	
	5	52.6	1.73	1.74	—	
	6	57.6	1.59	1.60	—	
	7	63.0	1.47	1.48	—	
	8	67.2	1.39	1.40	—	
	9	75.5	1.25	1.26	—	
	10	79.5	1.20	1.20	—	
C(750)	1	25.6	3.47	3.48	—	5.52
	2	35.1	2.55	2.55	—	
	3	37.5	2.39	2.41	—	
	4	43.3	2.08	2.09	0.3	
	5	52.3	1.74	1.74	—	
	6	57.4	1.60	1.60	—	
	7	63.0	1.47	1.48	—	
	8	67.2	1.39	1.40	—	
	9	75.4	1.25	1.26	—	
	10	79.4	1.20	1.20	—	

β = peak width at half-intensity.

t = linear dimension of the crystallite.






#### 2.4. Effect of the reaction temperature (determination of the apparent activation energy)

The data on  $\log V^{\circ}$  at flow rates of 9.4, 12.7, 15.4 and 17.8  $\times 10^{-2}$ /min. determined at various temperatures between 400° and 480°C allowed us to calculate the apparent activation energy  $\Delta E$  of conversion of cyclohexane Fig. (4), and the entropy change  $\Delta S^*$  of activation was calculated according to

$$K = e \frac{kt}{h} e^{\Delta S^*/R} e^{-E \exp/RT}$$

where K is the rate constant, k is Boltzmann's constant, h is Plank's constant, R is the gas constant and T is the absolute temperature.

**Table 2.** Data of the heterogeneous catalytic conversion of cyclohexane over Ni-A/A catalyst in flow system under normal pressure

Temp. °C	f.10 <sup>2</sup> min <sup>-1</sup>	V <sup>o</sup> ml.min <sup>-1</sup>		wt. gain %		Analysis of liquid products %					
											
						AO	AH	AO	AH	AO	AH
400	9.4	2.0	3.0	36.15	19.43	1.88	0.92	2.26	Tr	95.86	99.08
	12.7	1.9	2.5			0.35	0.77	2.26	Tr	99.47	99.23
	15.4	1.8	2.0			0.54	0.28	2.26	Tr	99.47	99.70
	17.8	1.4	2.1			0.62	0.12	Tr	Tr	99.40	99.88
430	9.4	6.7	9.2	54.60	50.00	5.19	1.91	0.52	Tr	94.30	98.09
	12.7	6.5	9.2			3.59	1.71	0.60	Tr	95.80	98.29
	15.4	6.0	9.2			2.30	1.36	0.30	Tr	97.40	98.64
	17.8	6.5	8.8			2.40	1.36	0.24	Tr	97.30	98.64
460	9.4	12.0	14.9	30.80	52.43	9.50	5.31	0.67	1.83	89.83	92.86
	12.7	11.2	14.6			7.90	4.11	0.60	0.48	91.50	95.41
	15.4	10.8	14.5			5.60	3.13	0.62	0.52	94.14	96.34
	17.8	11.8	14.5			4.00	1.78	0.12	0.28	94.90	97.94
480	9.4	24.1	18.7	35.38	64.95	12.20	16.00	0.78	1.10	87.04	82.87
	12.7	23.2	19.0			8.50	12.17	0.74	0.87	90.75	86.96
	15.4	23.4	19.3			6.00	6.21	0.70	0.68	93.33	93.10
	17.8	23.6	16.8			4.30	1.95	0.71	0.36	95.01	97.69

f = space velocity

Tr = traces.

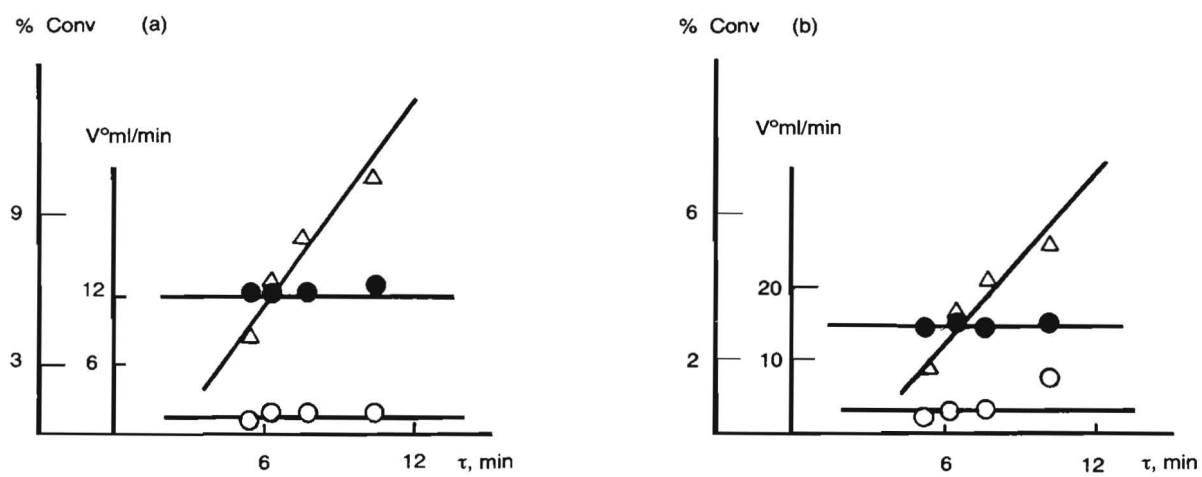


Fig. 3. The effect of variation of time of contact on the rate of conversion of cyclohexane at 460°C, (a) over Ni-A/AO and (b) over Ni-A/AH catalysts.

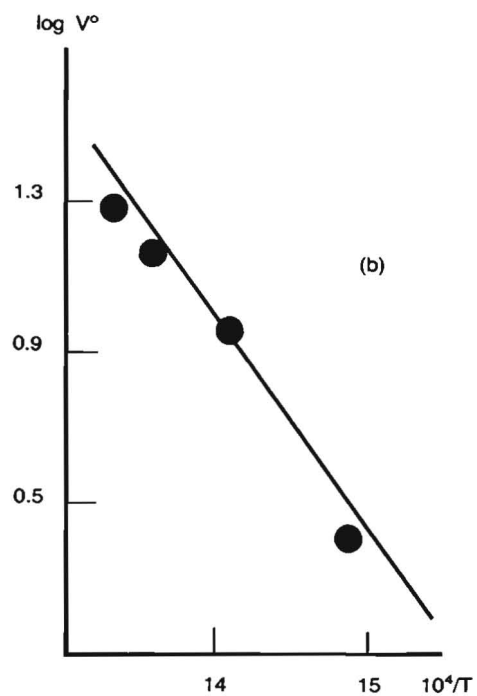
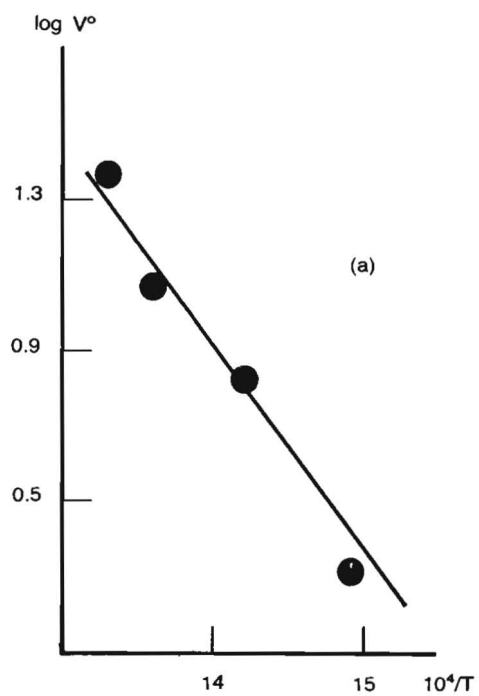





Fig. 4. Effect of temperature on the catalytic conversion of cyclohexane on (a) Ni-A/AO and (b) Ni-A/AH catalyst.

The calculated apparant activation energy is almost independent of the contact time, the working time of the catalyst and on the pretreatment conditions. An average value of  $23.76 \pm 0.2 \text{ kcal mol}^{-1}$  and  $23.75 \text{ cal mol}^{-1}$  for  $\Delta E$  and  $\Delta S^*$  respectively could be calculated for cyclohexane conversion over Ni-A/AO and Ni-A/AH catalysts. It is of interest, therefore, to compare between the catalytic activity of nickel-alumina Ni-A, 17.99% by wt Ni, prepared by coprecipitation given in (Ezzo *et al.* 1981) and nickel-alumina supported on  $\alpha$ -alumina Ni-A/A prepared by impregnation under the same experimental conditions, Table 3. The specific activity was calculated per  $S_{\text{BET}}$  of the catalyst pretreated with hydrogen for gaseous and liquid products at  $460^\circ\text{C}$ . It is obvious from these results that the average specific activity of Ni-A/AH for gaseous products exceeded that of Ni-AH by 1.11% at  $460^\circ\text{C}$ . However, Ni-AH had more activity for stepwise dehydrogenation to yield cyclohexene and benzene at  $460^\circ\text{C}$ . This fact may be due to the presence of  $\alpha\text{-Al}_2\text{O}_3$  as support giving rise to metal support interaction giving a decrease in  $S_{\text{BET}}$  and catalytic activity. Although the apparent activation energy, as calculated and given in Table 3, is larger in case of using Ni-A/AH catalyst, but the specific activity for dehydrogenation of cyclohexane is smaller. Accordingly, the decrease in catalytic activity of Ni-A/AH may be due to the nature of the condensating products formed on the active centres of the solid catalyst (Rudenko 1971). These condensating products were strongly adsorbed on the surface of the catalyst giving rise to apparent gain in weight (Table 2). This will be considered in the future work of our plan of investigation over the same catalyst.

According to a possible polymolecular mechanism (Ezzo *et al.* 1979, 1983 and 1984) we think that a polymolecular intermediate catalytic complex of the type  $\{(\text{C}_6\text{H}_{12})_n\}^{\text{K}_{\text{ads}}}$  is formed as  $n \text{ C}_6\text{H}_{12} + \text{K} \rightleftharpoons [(\text{C}_6\text{H}_{12})_n]^{\text{K}_{\text{ads}}}$ , (where K represents the catalyst) which decomposes in different ways giving the reaction products.

**Table 3.** The specific activity of Ni-A/AH catalyst in comparison with that of the previously studied Ni-AH catalyst, in cyclohexane conversion in flow system under normal pressure at 460°C

f.10 <sup>2</sup> min <sup>-1</sup>	Catalyst	S <sub>BET</sub>	V <sup>o</sup> /g S <sub>BET</sub>	% prod/g S <sub>BET</sub>			wt /g S <sub>BET</sub>	ΔE, kcal mol <sup>-1</sup>
								
9.4	Ni-AH	322	0.84	0.006	0.054	0.41	0.37	21.08 ± 0.5
12.7			0.84	0.007	0.036	0.43		
15.4			0.84	0.002	0.036	0.43		
17.8			0.84	0.002	0.011	0.46		
9.4	Ni-A/AH	152	0.95	0.03	0.01	0.59	0.33	23.76 ± 0.2
12.7			0.93	0.03	0.00	0.61		
15.4			0.93	0.02	0.00	0.61		
17.8			0.93	0.01	0.00	0.63		

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## دراسة على التحول الحفزي غير المتجانس للألكانات الحلقية على حفاز النيكل الومينا المحمل على الفا - الومينا (١) العملية الحفزية لنزع الهيدروجين من الهكسان الحلقي

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يشغل حفاز النيكل المحمل على الالومينا حيز كبير من الناحية العلمية في معامل البحوث أو الناحية الصناعية في مجال البتروكيماويات. كما أن لطرق تحضير النيكل أهمية كبيرة في دراسات الحفز غير المتجانس لما لها من تأثير على خواص ونشاطية الحفاز. فمن المعلوم أن طرق تحضير المادة الصلبة تؤثر على خواصها السطوحية مما يؤدي إلى تغيير في نشاطها الحفزي.

وفي دراستنا السابقة أمكن تحضير حفاز النيكل المحمل على الالومينا وتم التعرف على خواصه الفيزيائية ونشاطيته في تفاعل نزع الهيدروجين من الهكسان الحلقي.

وتعتمد الدراسة الحالية على تحضير حفاز النيكل بهدف تحسين نشاطيته وخاصيته في تفاعل نزع الهيدروجين من الهكسان الحلقي كتفاعل نمطي.

تم تحضير حفاز النيكل الومينا المحمل على الفا - الومينا (١, ٣١٪ من النيكل بالوزن) بواسطة التشبع لحبيبات الألفا - الومينا بمحلول يحتوى على نترات النيكل والالومنيوم حسب السعة الإمتزازية للالومينا.

وقد أعطت نتائج تحليل الإمتزاز بالنروجين قيمة ١٥٢ م<sup>٢</sup> جرام لمساحة سطوح الحفاز.

وبمقارنة القيم التي تم الحصول عليها من التحليل بالأشعة السينية مع قيم ASTM أمكن تحديد حجم البلورة عند درجات حرارة ٤٠٠، ٥٠٠ و ٧٥٠°م، ووجد أن حجم البلورة يزداد بارتفاع درجات الحرارة.

وأظهر التحليل الحراري الوزني للحفاز أنه يفقد ماء التبخر والهيدروكسيل عند ٤٥٢°م، ولذلك أختيرت درجة حرارة التنشيط للحفاز قبل إجراء التجارب عند ٥٠٠°م.

وقمت دراسة التحول الحفزي للهكسان الحلقي على حفاز النيكل الومينا المحمل على الفا - الومينا باستخدام النظام المستمر تحت الضغط العادي عند درجات حرارة من ٤٠٠ إلى ٤٨٠°م وعند سرعة فراغية تتراوح بين ٤، ٩ × ١٠<sup>-٢</sup> إلى ٨، ١٧ × ١٠<sup>-٢</sup> مل / دقيقة.

وقد أجري تحليل كروماتوجرافي للنواتج الغازية والسائلة. ووجد أن النواتج الغازية هي هيدروجين وميثان، والنواتج السائلة هي بنزين وهكسين حلقي مع هكسان حلقي غير متفاعل.

وأظهرت الدراسة الكينيتيكية الرتبة الصفرية للتفاعل بالنسبة للنواتج الغازية للهكسان الحلقي.

وأمكن حساب طاقة التنشيط الظاهرية وكانت قيمتها ٢٣,٧٦ ± ٢,٠ كيلو سعر/ مول لا تعتمد على السرعة الفراغية.

ونوقشت نشاطية واختيارية الحفاز على ضوء الدراسة السابقة على حفاز النيكل المحمل على الومينا.

ويتم التفاعل بواسطة ميكانيكية التفاعل متعدد الجزئيات السابق اقتراحه عن طريق تكون وسيط من مركب متعدد الجزئيات يتفكك بطرق مختلفة تبعاً للظروف الكينيتيكية معطياً نواتج التفاعل.