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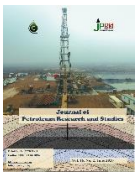

Preparation and Characterization of MoO3/TiO2 Catalysts for Hydroisomerization of n-Hexane

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Abstract

This research investigated the isomerization of n-hexane using molybdenum trioxide supported on titanium dioxide as a catalyst. The study explored the effect of liquid hourly space velocity (LHSV) and reaction temperature on n-hexane isomerization. The synthesized catalyst contained 20.46 wt.% molybdenum trioxide, with LHSV values ranging from 0.5 to 2 hr-1 and temperatures between 200oC and 275oC. The research aimed to identify the best reaction conditions, specifically temperature and LHSV, for maximizing the conversion of n-hexane to isomers. The results indicated that the highest conversion, 53.21%, occurred at an LHSV of 0.5 hr^{-1} and a temperature of 275°C. The reaction followed first-order kinetics with an activation energy of 25.753 kJ/mol.

Keywords: Isomerization, n-hexane conversion, MoO3/TiO2 catalyst, kinetic study.

تحضير وتوصيف محفز اوكسيد المولبدينيوم المحمل على اوكسيد التيتانيوم لعملية ازمرة الهكسان الطبيعي

الخلاصة:

تناول هذا البحث دراسة عملية تكوين أيزومرات الهكسان الطبيعي باستخدام ثلاثي أوكسيد الموليبدينوم المدعوم بثاني أوكسيد التيتانيوم كمحفز. واستكشفت الدراسة تأثير محتوى أوكسيد الموليبدينوم وسرعة السائل في الفراغ ودرجة حرارة التفاعل. احتوى المحفز على 20.46% من ثلاثي أكسيد الموليبدينوم، مع قيم سرعة فراغية تتراوح من 0.5 إلى 2 ساعة$^{-1}$ ودرجات حرارة تتراوح بين 200 درجة مئوية و275 درجة مئوية. وكان الهدف الرئيسي هو الاستفادة من هذا المحفز وتحليل حركية تفاعل تكوين أيزومرات الهكسان الطبيعي. وهدف البحث إلى تحديد الظروف المثلى للتفاعل، وتحديدًا درجة الحرارة، والسرعة الفراغية، لزيادة تحويل الهكسان الطبيعي إلى أيزومرات. وأشارت النتائج إلى أن

أعلى تحويل، 53.21%، حدث عند سرعة السائل في الفراغ بالساعة 0.5 ساعة$^{-1}$ ودرجة حرارة 275 درجة مئوية. وقد اتبع التفاعل حركية من الدرجة الأولى مع طاقة تنشيط مقدارها 25.753 كيلوجول/مول.

1. Introduction

Isomerization, cracking, and alkylation are acid-catalyzed reactions. Given the significant global oil consumption, acid-catalyzed hydrocarbon reactions are crucial [1],[2]. The primary process for improving the octane number of gasoline is catalytic reforming, which results in a high concentration of aromatic compounds [3]. However, the product from this process has adverse environmental effects, such as increased CO_2 emissions and a higher risk of cancer than the product from isomerization [4]. Over the years, different methods have been used to increase the octane number of fuel. One approach involved adding lead compounds, specifically tetraethyl lead. However, leaded fuel was eventually restricted and banned due to its toxicity and harmful effects on exhaust gas converters. Another method to enhance the Research Octane Number (RON) was blending the fuel with benzene or other aromatics. However, as regulations on the permissible levels of these aromatics in fuel became stricter, this approach faced challenges. Additionally, using Methyl Tertiary Butyl Ether (MTBE) to boost the octane rating has been linked to groundwater pollution, although it is less hazardous than lead [5].

Isomerization is a cost-effective and straightforward technique for improving octane number. It is a catalytic process that involves rearranging the molecular structure of a hydrocarbon without gaining or losing any components. This process increases the amount of hydrocarbon octane, such as butane, pentane, and naphtha, as well as conventional hexane fractions in gasoline [6].

A problem encountered in the isomerization of hydrocarbons is the formation and accumulation of carbon (coke) within the pores [7]; as a result, lower reaction temperatures favor the generation of isoparaffins. Hence, catalysts that operate at low temperatures are often more selective to isomers than those that operate at high temperatures [5].

C5/C6 (pentane/Hexane) isomerization is a catalytic process in which n-pentane and n-hexane are transformed into their isomeric forms. These isomeric forms are then added to the gasoline pool to increase the octane number through isomerization reactions of n-hexane. When n-hexane undergoes isomerization, four forms of iso-hexane are created: 2-methyl pentane (2-MP), 3-methyl pentane (3-MP), 2,3-dimethylbutane (2,3-DMB), and 2,2-dimethylbutane (2,2-DMB) [8]. Bifunctional catalysts are crucial in converting hydrocarbons as they contain metal and acid sites that catalyze various chemical reactions. To accomplish high isomerization selectivity, a balance between the active component (metal) and acid functions is required [9]; the impregnation method

is a suitable mechanism that is used to load the active metal on the acidic function [10]. The metal sites facilitate hydrogenation and dehydrogenation reactions to convert normal alkanes, while the acid sites are responsible for isomerization and cracking reactions. These reactions produce iso-alkanes of the same molecular weight or below [11]. The hydro-dehydrogenation (HD/DHD) component can consist of a noble metal (Platinum and Palladium; Pt, Pd) or a combination of non-noble metal-sulfides (Molybdenum and Tungsten; Mo, W) and at least (Cobalt and Nickel; Co, Ni). The acidic component can be an amorphous oxide like silica-alumina, a zeolite, or a mesoporous material [12].

In industrial isomerization units, platinum-loaded chlorinated alumina catalysts are utilized because of their high acidity and low reaction temperature of approximately 170°C [13]. However, there are several drawbacks to using this type of catalyst. The use of chlorine in catalyst regeneration is associated with various corrosion and environmental issues [14]. Several issues are associated with Pt-based catalysts, including their relatively high cost, formation of toxic benzene as a by-product, corrosion problems due to the introduction of chlorine, and sensitivity to sulfur poisoning [15], [16].

This issue highlights the need for a more efficient and less harmful catalyst for n-alkane isomerization, which can enhance the production of isomerate. The MoO_3 is a newly developed material that has caught the attention of researchers. It has excellent potential to replace platinum as a catalyst. The reduced form of MoO_3 (MoO_2) is a metallic function for dehydrogenation and hydrogenation reactions [17]. In contrast, TiO_2 has an acidic function for carbocation and n-alkanes isomerization reactions [18].

The current work used the sol-gel technique to prepare anatase TiO_2 nanoparticles with a high surface area as a photocatalyst. The catalyst was characterized by detecting x-ray diffraction and measuring surface area and pore volume. Molybdenum trioxide was then deposited on the TiO_2 to produce MoO_3/TiO_2 catalysts. The study involved the isomerization of (n-Hexane) as a simulated model to detect the generation of relevant isomers in a fixed-bed reactor unit by sending the product to GC- chromatography test with kinetic data analysis dependent on the reaction rate equation.

2. Experimental work

2.1. Chemical materials

Many chemicals used in catalyst preparation and loading methods. Table (1) illustrates the chemical compounds used with their specification. n-Hexane is used as a feed investigated. It is provided by CDH company. Table (2) illustrates this hydrocarbon's physical properties.

Table (1): Chemical Compounds Specifications

Chemical	Phase	Density g/cm^3	M. wt. g/gmol	Purity %	Supplier
Titanium tetra Isopropoxide ($C_{12}H_{28}O_4Ti$)	Liquid	0.96	284.36	99.9	Himedia
Hydrochloric acid (HCl 36%)	Liquid	1.16	36.46	99	CDH
Ethanol (C_2H_6O)	Liquid	0.789	46.08	99.9	DiamonD
Ammonium hepta molybdate-tetrahydrate ($(NH_4)_6Mo_7O_{24}.4H_2O$)	Solid	2.498	1235.86	99	Sinopharm

Table (2): n-Hexane physical properties

Property	The value
Octane number	60
Sulfur content	1.6 ppm
Density	0.664 g/cm^3
Initial boiling point	36^{o}C
End boiling point	84^{o}C

2.2. Catalyst preparation

TiO_2 was synthesized using the sol-gel method. The chemicals used in the synthesis method were titanium tetra isopropoxide, hydrochloric acid, ethanol, and ammonium hepta molybdate-tetrahydrate. Initially, 7 ml of titanium isopropoxide was added to 30 ml of ethanol at an ambient temperature; at the same time, 5 ml of concentrated hydrochloric acid was added to 20 ml of ethanol and 2 ml of water. These two mixtures will mix for 30 minutes, and then the first solution will add dropwise to the second one with mixing. After stirring for 2 hours, a homogenous mixture was obtained, and then the solution was left for 24 hours until a gel was obtained. Then, the sample will dry for 24 hours and calcinated for 4 hours at 400°C, this preparation method was achieved by ref. [19].

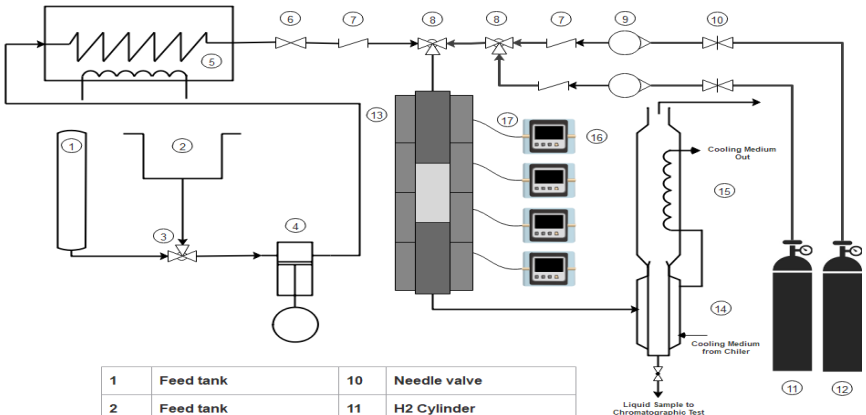
The alkane isomerization process can be improved by increasing the stability and selectivity of branched isomers while also preventing the poisoning of the active metal sites. The impregnation method was adapted to add molybdenum as a promoter. The necessary quantity of Mo (20.46 wt.%) was attained by adding the appropriate amount of ammonium heptamolybdate tetrahydrate (AHM) ($(NH_4)_6Mo_7O_{24}.4H_2O$) solution onto TiO_2. The necessary stoichiometric amount of AHM salt was dissolved in distilled water at room temperature. The impregnated solid was then dried overnight in an oven at 110^{o}C and calcined for 5 hrs at 550^{o}C. Table (3) displays the physical properties of the MoO_3/TiO_2 that were obtained.

Table (3): MoO_3/TiO_2 physical properties

Specifications	Value
Surface area (m^2/g)	63.925
Pore volume (cm^3/g)	0.1139
Bulk density (g/cm^3)	1.2865

2.3. Isomerization process

After preparing the catalyst MoO_3/TiO_2, the catalyst was charged to the reactor in the middle part. At the same time, a glass ball with a diameter of 0.2 cm was used in the upper and lower parts of the reactor to give an effective catalyst surface area with uniform flow, as shown in Figure (1). In the beginning, nitrogen gas enters the reactor from the top of the reactor to remove the air; then, the reactor is heated to the desired temperature. After a period of time, when the reactor reached the reaction temperature, the nitrogen valve was closed. After that, n-hexane with a known flow rate evaporates in the evaporator before entering the reactor and mixed with hydrogen gas at the top with good distribution to reach the catalyst. After isomerization reaction the product gas enters to throttling valve followed by the condenser. A refrigerant system cools the reactor product with ethyl alcohol, chills it in a chiller, and passes it between the two sections to condense products. This process helped cool the reaction products to -15°C, the hydrogen was separated by the separator. Then, the condensable vapors were collected as a liquid in the collector and sent to the GC chromatograph analyzer to detect and classify the products. The isomerization reaction experiment conditions were listed in Table (4).



1	Feed tank	10	Needle valve
2	Feed tank	11	H2 Cylinder
3	3 way valve	12	N2 Cylinder
4	Feed pump	13	Reactor
5	Evaporator	14	Collector
6	Gate valve	15	Condensor
7	One way valve	16	Temperature Controller
8	Mixer	17	Thermocouples
9	Rotameter	18	

Fig. (1): Isomerization unit scheme

Table (4): Experiment conditions

Conditions	The value
Temperature (°C)	200 to 275
Liquid hourly space velocity (LHSV) (hr^{-1})	0.5 to 2
H_2/hexane mole ratio	4:1
Pressure (atm)	1

3. Result and Discussion

3.1. XRD (X-Ray Diffraction)

The powder of MoO_3/TiO_2 was characterized using X-ray diffraction (XRD), and 2θ range of (20°-80°) was used to verify the structure of the synthesized catalyst. Figure (2) shows the XRD of the prepared catalyst. It a single contained agree with ref. [19]. Also, it can be shown that molybdenum was impregnated successfully and its peak can be demonstrated at 2θ = 12.585°, 23.222 and 25.393°, these; results agree with ref. [20]. Also, it can be observed that the synthesized catalysts did not peak at 2θ values of 27 and 31, which indicates that the catalyst is free from rutile or brookite impurities, which in agreement with ref. [21].

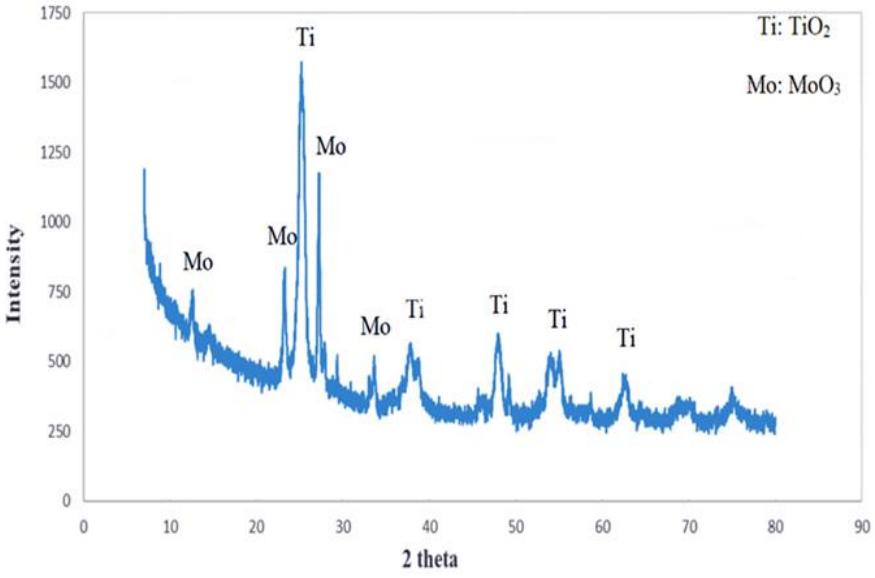


Fig. (2): XRD of MoO_3/TiO_2 Catalyst

3.2. Effect of Temperature and LHSV on n-Hexane Isomerization

It can be shown from Figure (3) that the isomerization of n-hexane increased as temperature increased from 200 to 275°C at the same LHSV, and this is due to the nature of the isomerization

reaction, which is exothermic, so increasing temperature leads to increased isomers until the temperature reaches 300°C. At this temperature cracking reaction becomes higher than the isomerization reaction rate [22], so the temperature used were ranged between 200ºC-275ºC and this is agreed with ref. [23]. At LHSV of about 0.5 hr^{-1}, the conversion of n-hexane on MoO_3/TiO_2 increases from 33.04% at 200ºC to 53.21% at 275ºC.

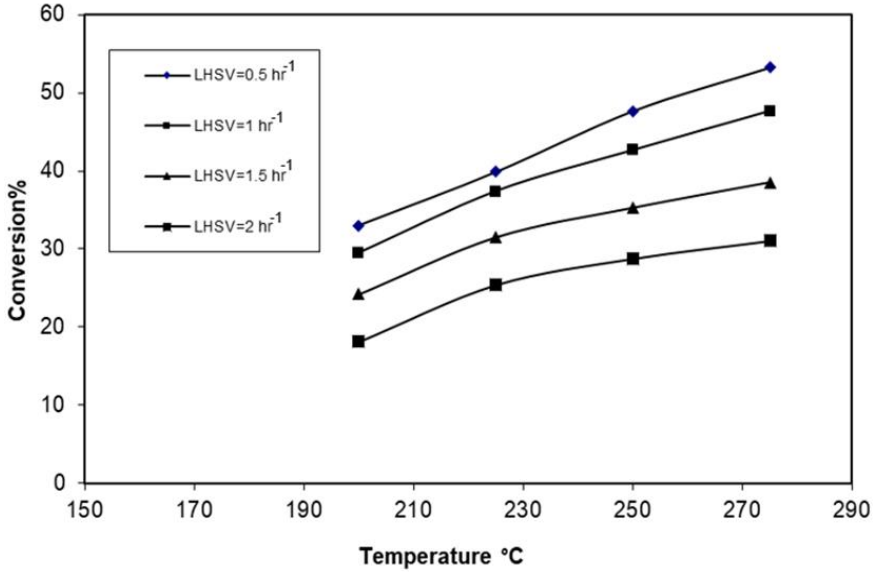


Fig. (3): n-Hexane Conversion at different temperatures

Figure (4) illustrates the impact of LHSV on n-hexane isomerization, revealing that a decrease in LHSV leads to an increase in isomers due to longer contact time between the feed and the catalyst [24], [25]. These finding indicate that low LHSV is favorable for the isomerization process, resulting in higher conversion to isomers. At an LHSV of $0.5hr^{-1}$ and a temperature of 275˚C, the n-hexane conversion for MoO_3/TiO_2 reached 53.21%. However, when the LHSV was raised to $2hr^{-1}$ at the same temperature, conversion dropped to 31.08%. The conversion results are summarized in (4).

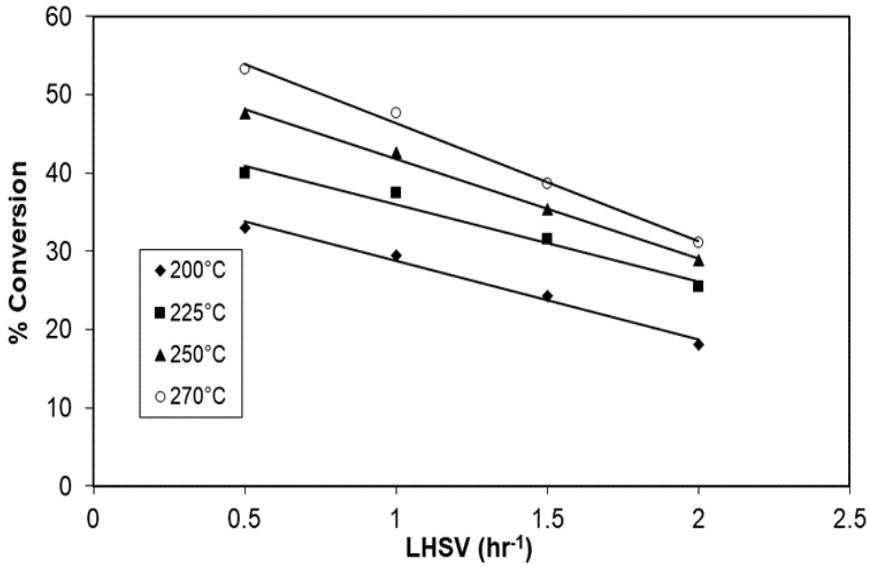


Fig. (4): n-Hexane Conversion with different LHSV

Table (4): Conversion resulted in different temperatures and LHSV

	Temperature (°C)			
LHSV (hr^{-1})	200	225	250	275
0.5	33.04	39.87	47.65	53.21
1	29.5	37.39	42.64	47.64
1.5	24.2	31.49	35.28	38.54
2	18.1	25.41	28.79	31.08

4. Kinetic Study

4.1. The rate of the reaction

Differential method is employed to estimate the isomerization reaction rate for n-hexane conversion [26].

In catalytic systems, the rate equation can be formulated based on the weight of the catalyst particles, as shown in Equation (1).

$$\frac{W}{F_{A0}} = \int_0^{X_A} \frac{dX_A}{-r_A} \quad (1)$$

Differential Equation (1), obtained from Equation (2)

$$-r_A = \frac{dX_A}{d\left(\frac{W}{F_{A0}}\right)} \quad (2)$$

According to the differential method, the reaction rate at any given moment can be determined by calculating the slope of the tangent line to the curve representing the relationship between the conversion of n-hexane and Weight of catalyst/ Molar flow rate (W/FA0). Figure (5) shows the conversion vs. the (W/FA_0) plot for the MoO_3/TiO_2 catalyst, while Table (5) illustrates the rate of reaction values at various temperatures and LHSV conditions.

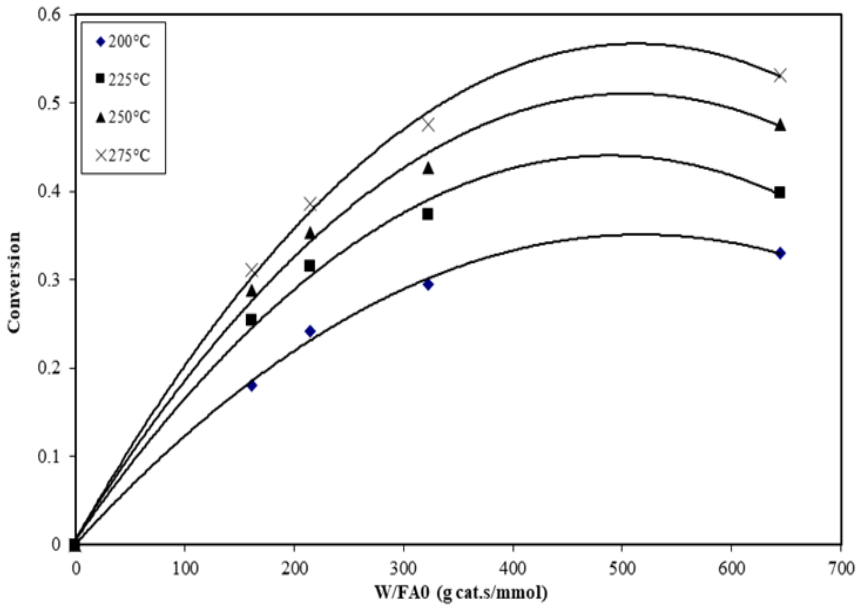


Fig. (5): Conversion and W/FA_0 Relationship

Table (5): Rate value in (mole/g_{cat}. sec)

	Temperature (°C)			
LHSV (hr^{-1})	200	225	250	275
0.5	0.05214	0.07123	0.06214	0.05214
1	0.05925	0.09284	0.09521	0.08524
1.5	0.07552	0.11254	0.13214	0.12350
2	0.09785	0.13248	0.16325	0.16547

4.2. The order of the reaction for n-hexane

Figure (6) shows that the $-r_A$ and C_A relationship is linear; therefore, the reaction is first-order. As the temperature increased from 200°C to 275°C, no significant change in the reaction order was observed. The reaction rate constants (k) at different temperatures were calculated depending on the linear relationship of $-r_A$ and C_A. Table (6) displays the values of reaction rate constants obtained at various temperatures.

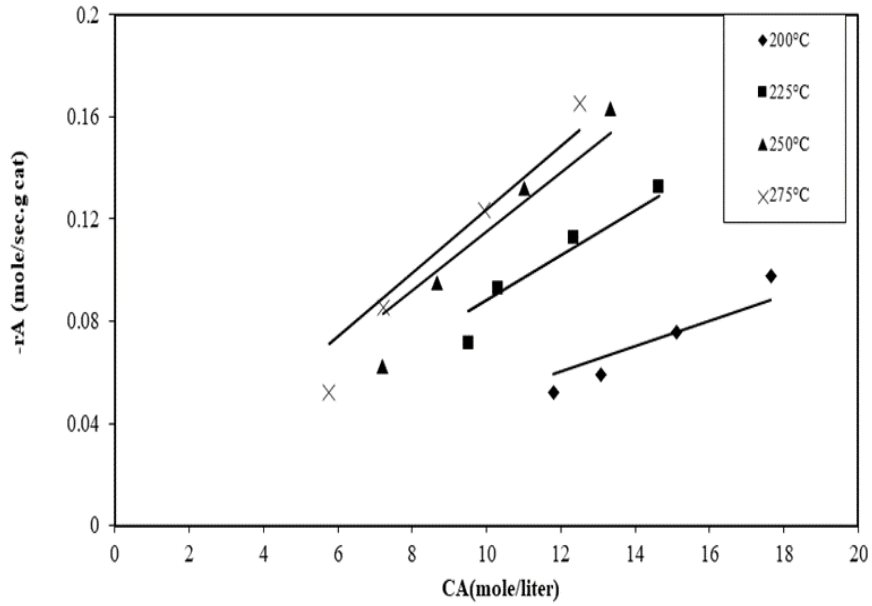


Fig. (6): $-r_A$ vs. C_A Relationship

Table (6): Reaction rate constant

Temperature °C	k (1/sec)
200	0.005
225	0.0088
250	0.011
275	0.0124

4.3. The Measurements of Apparent Activation Energy

The activation energy of the isomerization process was calculated using the Arrhenius equation. As shown in Equation (3).

$$k = Ae^{\frac{-Ea}{RT}} \quad (3)$$

Where;

k = Rate Constant

A = Frequency Constant Factor (Pre-exponential Factor)

Ea = Activation Energy

R = Gas Constant = 8.314 J/mol. K

T = Temperature

Figure (7) displays the plot of (Ln k) vs. (1/T) to calculate the activation energy for isomerization reactions. The activation energy for the MoO3/TiO2 catalyst was 25.753 kJ/mol.

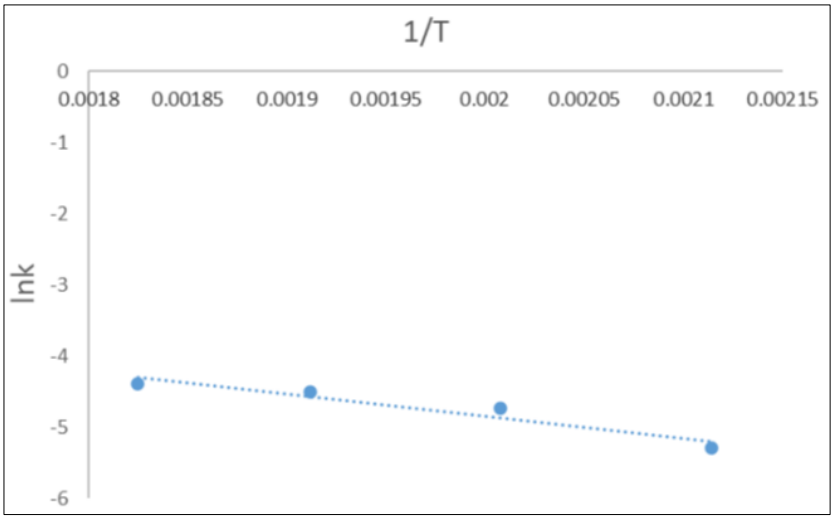


Fig. (7): ln k vs. 1/T plot

5. Conclusions

TiO_2 was successfully prepared using the sol-gel method and impregnated with MoO_3 to generate an MoO_3/TiO_2 catalyst. The catalyst was then characterized with XRD to show the TiO_2 phase and the presence of MoO_3. The surface area and pore volume were measured to ensure good physical properties that affected the isomerization reaction. The catalyst has promising activity in isomerization reactions with various ranges of temperature and LHSV. The highest conversion of n-hexane was 53.21% at 275^oC and 0.5hr^{-1}. The kinetic study of this reaction showed that the reaction order was first order, and the MoO_3/TiO_2 catalyst needs only 25.753 kJ/mol activation energy.

Author Contributions Statement: Sara A. Al-Naib contributed to the Conception; Modeling and Simulation; Writing – Original Draft. Haider A. Al-Jendeel contributed to Methodology; Investigation and Experiments. Sattar J. Hussein contributed to the Data Curation and Analysis; Writing – Review & Editing. All authors have read and approved the final version of the manuscript.

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