

Investigation the activity of Pt Loading on Commercial Monolithic Catalyst in Automobile Exhaust Gases

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ABSTRACT

Catalytic monolith reactors have numerous applications in industrial processes and as technical devices, so the focus is set specially on automotive catalytic converters.

In present work the experimental investigation has been carried out to study the performance of monolith reactor on the oxidation and reduction of exhaust gas (NO, CO, HC, O₂ and H₂O) which emitted from gasoline generator. Commercial and modified commercial ceramic monolith catalyst was used in the present work. The modified commercial catalyst was obtained by loaded Pt metal .A laboratory unit was constructed for this purpose where a versatile stainless steel monolith reactor of 0.02 m inside diameter and 0.2 m height was used.

The catalytic performance of the catalysts was studied in the following operating conditions: gas space velocity in the range 17.69 – 44.23s⁻¹, reaction temperature in the range 373 – 673 K, bed length in the range 0.075 – 0.15 m and at atmospheric pressure and constant air / fuel ratio (14.6).

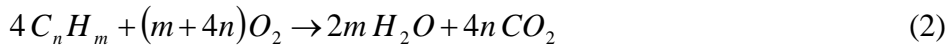
The results show that the conversion of NO, CO, HC, O₂ and H₂O are slightly changed with the bed length. The conversion of exhaust gas reactant enhances in presence of water (wet condition) rather than in the absence of water (dry condition) and increases with the increasing reaction temperature and decreases with increasing gas space velocity.

Monolithic catalyst which loaded with 0.4% Pt gives high conversion compared with an commercial catalyst (unloaded) for the CO oxidation reaction more than hydrocarbon oxidation and NO reduction.

Key Words: Three way catalyst, platinum, wash coat.

INTRODUCTION

Automobile exhaust gases are a major source of manmade emissions. Many improvements were made by engine modifications. However, 1975 U.S Federal and California limits could not met by engine modifications alone and the catalytic converter was shown to be the best forward. Considerable research into catalytic system was taken by both industry and academics. Catalysts using base metals such as nickel, copper, cobalt and iron seemed initially to be attractive on the basis of cost. However, these catalysts were adversely affected by sulfur and residual traces of lead in the fuel, and catalysts eventually chosen were based on the platinum – group metals. For a few years, the emission limit could be reached by oxidation of carbon monoxide and hydrocarbon emitted by the engine ⁽¹⁾, as in equations (1) and (2):-



The most common catalyst called conventional oxidation catalyst (two ways catalyst) was based on platinum and palladium on the alumina support ⁽²⁾. However, as legislation tightened further, it became necessary to control the NO_x emission. This brought two further requirements: closer around the stoichiometric air/fuel ratio, and the addition of a further catalytic metallic component, Rhodium, to the catalyst formulation to enable the NO_x to be selectivity reduced to nitrogen as in equations (3) and (4) ⁽³⁾ :



Monolith catalyst research since 1987 has focused on reactor design, increasing efficiency and the search for more suitable catalyst. Thus the complete oxidation of *CO* and *HC* and reduction of *NO* can be found over either noble metal (*Pt*, *Pd* and *Rh*) or other metal oxide (*Ba* and *La*)^(4, 5).

During the last years, extensive efforts were made to develop new formulations, reporting on the specific role of platinum, palladium and rhodium in single and multiple compositions on the total conversion under real conditions, and studied the aging resistance of the catalyst under oxidizing, reducing environments. Furthermore, the Platinum / Rhodium three – way catalyst converter is an essential device in controlling the three regulated pollutant emissions (*CO*, *NO_x* and *HC*)^(6, 7).

The aim of the present work is to study the effect of bed length of the catalyst at constant gas space velocity with reaction temperature and to investigate the performance of the modified commercial monolithic catalyst loaded with 0.4 % *Pt* at wet and dry conditions for various operating condition and compared the results with commercial catalyst .

EXPERIMENTAL ARRANGEMENTS

Feed Gases

Average emission gases produced from commercial gasoline generator (Single piston Astra 2.5 kv) are used as feedstock in this investigation. Table (1) shows the chromatographic analysis of this feedstock.

Commercial Monolith

Three-way monolith made from ceramic honeycomb substrate (cordierite) was used in this investigation. The chemical composition (analyzed by The State Company of Geological Survey and Mining / Ministry of Industry) and physical properties (determined by Petroleum Research and Development Center and Production and Metallurgy Department of University of Technology) are presented in Table (2).

Catalyst Preparation

The sample of monolith commercial catalysts was loaded with 0.4 wt % Pt by impregnation with aqueous solution of hexachloroplatinic acid. The platinum content of catalyst was calculated from the weight of sample of catalyst and amount of metal in impregnation solution and is presented in Table (3).

The impregnation was carried out in the apparatus shown in Figure (1) which consists of conical flask (500 ml) connected to a vacuum pump (Awelch 1402, one stage Germany) and funnel for impregnation solution addition..

The impregnation process was carried out under vacuum with shaking at room temperature for four hours. The impregnation solution was added drop by drop, and the impregnated sample was left in its solution over night followed by drying at 120 °C and calcined at 400 °C for four hours in an electrical furnace (W.C. Heraeus GMBHV) with dry air then stored in a porcelain crucible in glass desiccators over silica gel. Table (4) presents the properties of the prepared catalysts.

EXPERIMENTAL WORK

Equipments

The schematic diagram of the unit employed is shown in Figure (2). The experimental setup consisted of preheating zone, tubular reactor, electrical heater, thermocouple, calibrated rotameter, condenser and gas analyzer. The isolated stainless steel reactor (20 mm inside diameter and 200mm length), is heated by external tape heater (HERAEUS type TM56, 2000 watt).

Calibrated gas rotameter was used to measure exhaust gas flow rate. The inlet exhaust gas temperature was measured by temperature indicator. The reaction temperature was measured and controlled by electrical controller type (Yang Ming C x TA 3000) with thermocouple (Ni-CrNi) which was placed in the reaction zone.

The Procedure

The flow rate of reactants (gas emitted from generator) was adjusted to the desired value for each particular experiment. The mixture of reactants was entered the preheating zone and then passed the catalyst bed after it was heated to desired temperatures. After 20 minutes, when steady state condition was obtained the samples were taken from the outlet condenser to be analyzed by gas chromatography. The above-mentioned procedure was used for wet condition study. During the dry condition, dehumidifier was placed before the reactor entrance to remove the humidity from the emission gases, therefore samples were taken from the outlet of dehumidifier analyzed by gas chromatography to insure that the gases were dried.

ANALYSIS SYSTEM

Reactants and products were analyzed by a gas chromatography (GC) (SHIMADZU – 14A) equipped with a thermal conductivity detector (TCD). The apparatus consists of two stainless steel columns; the first column was porapak N while the second one was porapak Q. All the samples were trapped in a especial trap and sealed well, and analyzed in Abin Siena company. The operating conditions of the gas chromatography are shown in Table (5).

RESULTS AND DISCUSSION

Effect of Operating Variable on Monolith Performance

The Effect of Bed Length

Figures (3 to 6) show the effect of bed length on conversion of the exhaust gas reactants for NO reduction and C₃H₆ and CO oxidation.

From these figures, it can be noticed that the conversion slightly changes with the bed length, so there is no appreciable external mass transfer limitation for both NO reduction and CO and C₃H₆ oxidation. This observation is noted also by George et al⁽⁸⁾.

From these figures, all the experiment on 0.4 % Pt and commercial catalyst were carried out at bed length of 10 cm, this length was chosen as a right proper position in the reactor.

From Figure (3), the zero percentage of NO conversion at 373K is due to no reduction of NO at this temperature while at 473 K, 573 K and 673 K were exist in the different bed length.

Effect of Water

Figures (7 to 9) show the effect of water in the exhaust gas of about 11% on the NO, CO and C₃H₆ conversion at a given temperature and different gas space velocities of exhaust gas on commercial monolith catalyst.

It has been observed that the conversion of exhaust gas reactants is enhanced in presence of water (wet conditions) rather than in the absence of water (dry conditions). This is attributed to two main reasons; the water reduces self poisoning of the metal surface by CO (Gonzales-Velasco)⁽⁹⁾, and contributes to the oxidation of ceria, being in much higher concentration than oxygen in the reaction environment. Oxygen from ceria is thought to contribute significantly to the oxidation of CO and C₃H₆ adsorbed on the metal (Bunluesin)⁽¹⁰⁾.

Also water weakens the adsorption strength, allowing the components to react continuously to high conversion (Botas)⁽¹¹⁾. These results are in agreements with the finding of Gonzalez-Velasco⁽⁹⁾ and Chen⁽¹²⁾.

From these results all the experiments on 0.4 % Pt loaded catalyst, were carried out all in wet conditions

Effect of Temperature

The effluence of temperature in the range of (373-673 K) on the exhaust gas conversion at a given gas space velocity (17.69 s⁻¹) for commercial monolith catalyst at wet and dry conditions and 0.4% Pt loading catalyst at wet condition only, at bed length 10 cm is shown in Figures (10 to 12).

It can be seen from these figures, the conversion of exhaust gas increases with increasing temperature. This is attributed to the increasing of diffusivity of molecular gases, thinner layer of

gas film where the external resistance is neglected leading to direct access of gaseous to the catalyst.

Figures (10 to 12) also show that the conversion of direct oxidation of CO and C₃H₆ is higher than the conversion of the reduction of NO. This is due to higher value of activation energy of NO reduction compared with the activation energy of CO and C₃H₆ oxidation (see Table 6). This indicates that NO reduction requires higher temperature to reach similar CO and C₃H₆ conversions.

These results are in agreement with previous finding of Hoebink et al.⁽¹³⁾ and Granger, et al.⁽⁶⁾

Effect of Gas Space Velocity

Figures (13 to 15) illustrate the effect of gas space velocity in the range of (17.69 - 44.23 s⁻¹) on the conversion of exhaust gas at temperature (573 K) for commercial monolith catalyst at wet and dry condition and for Pt loading catalyst at wet condition only.

It can be noticed from these figures that the conversion of the exhaust gas components decreases with increasing gas velocity, owing to decrease in the residence time (decreasing contact time of the feed reactant) with the catalyst inside reactor, such trends are in agreement with the observation of previous finding of Williamsons et al.⁽¹⁴⁾, Tomasic et al.⁽¹⁵⁾ and Fabiano et al.⁽¹⁶⁾.

Effect of Pt loaded on monolithic catalyst

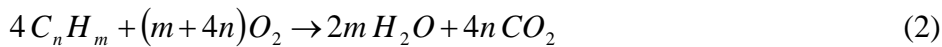
Figures (16 - 18) show the effect 0.4% wt loading of Pt on the conversion of exhaust gas at a given temperature 673 K and different space velocities.

From these figures, it can be seen that the conversion of NO reduction using Pt loading catalyst is higher than those obtained by commercial catalysts. This may be attributed to the

lower apparent activation energy of the NO reduction over 0.4% Pt loading catalyst compared with the commercial catalyst (see Table 6).

Estimation of Reaction Kinetic Parameters

The kinetic rate prediction is necessary for the reactor and monolith catalyst model simulation. The overall catalytic reactions that are important for controlling exhaust condition are given by questions 1, 2, 4 and 5 below. The kinetic reaction rate is considered to follow simple power law. Differential method analysis is used to estimate the reaction rate for small variation in the conversion or for shallow small reactor (Levenspiel)⁽¹⁷⁾.



For each run in a differential reactor the performance equation becomes (Levenspiel)⁽¹⁷⁾:-

$$\frac{w}{F_{Ao}} = \int_0^x \frac{dx}{-r_A} = \frac{1}{(-r_A)_{av}} \int_0^x dx_A = \frac{\Delta x}{(-r_A)_{av}} \quad (6)$$

From equation (6) the average rate for each run is found, thus each run gives directly a value for the rate of the reaction at the average concentration in the reactor.

From Figure (19 to 21) show the results of the rate of reaction estimated for NO reduction, CO oxidation and C₃H₆ oxidation using commercial and Pt loaded catalyst.

Non-linear regression analysis using Statistics (version 6) software is used to estimate reaction rate parameters (rate constant and reaction order) for each set of flow rate at a given temperature for each reaction component using commercial and Pt loading catalyst.

The statistical analysis gives correlation coefficients between (0.932-0.94) which is statistically significant.

The apparent activation energy was established from Arrhenius equation (7) that satisfies the relationships between rate constant and reaction temperature.

$$K = A e^{-E/Rg T} \quad (7)$$

A plot of $\ln (k)$ vs. $\left(\frac{1}{T}\right)$ is shown in Figures (22) which are used for calculation of E_a values, since $(\ln A)$ represents the intercept and the slope is represented by $\left(\frac{-E_a}{R}\right)$. Table (7) show the apparent activation and Arrhenius factor for commercial catalyst and Pt loading .

The results of the analysis of the parameters estimation are outlined in Table (6).

From the above table, the apparent activation energy values are in the range of (18-56 KJ/mol), this indicates that the internal diffusion severely limits the reaction which is in a good agreement with Farrauto⁽¹⁸⁾, Fabiano⁽¹⁹⁾ and tomasic⁽²⁰⁾.

CONCLUSIONS

1. The conversion of exhaust gas reactants (NO, CO, HC, H₂O and O₂) increases with increasing reaction temperature from 373 to 673K and decreases with increasing gas second space velocity (17.69 – 44.23 s⁻¹).
2. The reaction rate of exhaust gas reactants increases with increasing reaction temperature and gas second space velocity.
3. The conversion of exhaust gas reactants is enhanced in presence of water (wet conditions) rather than in the dry condition.
4. The apparent activation energy values in the range of 17-56 kJ/mol indicate that the internal diffusion severely limits reaction.
5. Conversion of CO oxidation on Pt loaded catalyst is higher than the hydrocarbon oxidation and NO reduction than which obtained on commercial catalyst.

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Nomenclature

Symbols	Definition	Unit
E_a	Activation energy	kJ/mole
F_A	Molar rate	mole/m ³ .s
K	Rate constant	mole ^{1-(n+m)} /kg _{cat} .s.m ^{3(n+m)}
R_g	Gas constant	m ³ .atm/mole.K
r_A	Reaction rate	mole/kg _{cat} .s
T	Temperature	K
W	Weight catalyst	Kg
X	Conversion	(-)

Table (1): Composition of the feedstock, % volume

<i>CO</i> %	<i>NO</i> %	<i>HC</i> %	<i>CO₂</i> %	<i>O₂</i> %	<i>H₂O</i> %	<i>N₂</i> %
2.00	0.03	0.50	10.00	16.40	11.00	Balance

Table (2): Chemical and physical properties of monolith catalyst.

<i>Chemical composition of monolith %</i>	
$\gamma - \text{Al}_2\text{O}_3$	69.76
SiO_2	11.3
Fe_2O_3	9.4
CeO_2	6.7
BaO	2.6
TiO_2	0.01
CuO	0.02
Pt	0.05
Rh	0.16
<i>Physicals Properties</i>	
Channel Structure	Circle
Number of channels, channel / cm^2	64
Channel spacing, mm	1.4
Wall thickness, mm	0.3
Wall porosity %	60
Bulk density, g / cm^3	1.4
Pore volume, cm^3 / gm	0.93
Surface area, m^2 / g	97

Table (3): The amount of reagents used for preparation of 0.4 % Pt catalysts

No.	wt. of catalyst sample, gm	Type of metal	wt. of metal gm	wt. of salt gm
1	14.02	Pt	0.07	0.184

Table (4): The properties of the prepared catalysts

Catalyst Specification	Pt
Surface area, m ² /gm	70
bulk density, gm/cm ³	1.48
Porosity	0.415
Equivalent radius, nm	8

Table (5): Temperature program and operating conditions of gas chromatography.

<i>Temperature Program for the Column</i>	
Initial Column temperature, K	303
Final Column temperature, K	393
Rate of temperature increasing °C / min	5
<i>Other Condition</i>	
Injection temperature, K	423
Detector temperature, K	523
Flow rate of Helium, ml / min	20

Table (6): Apparent activation energy values and reaction order for commercial catalyst and Pt loading metal.

Reaction	E_a / R	E_a KJ / mol	Ao	Reaction order	
				a_i	b_i
Wet Condition					
NO + CO	6500.5	54.05	3008.8	0.5	1.25
CO + $\frac{1}{2}$ O ₂	3090.2	25.691	16.85	1	0.5
CO + H ₂ O	2194.4	18.24	0.0432	1	0.5
C ₃ H ₆ + 4.5 O ₂	3421.5	28.446	15.34	1	0.5
Dry Condition					
NO + CO	6809	56.61	47.002	0.5	1.25
CO + $\frac{1}{2}$ O ₂	3427.6	28.49	10.580	1	0.5
C ₃ H ₆ + 4.5 O ₂	3708.4	30.82	3.265	1	0.5
Pt					
NO + CO	5500.4	45.7	5.6757	0.4	1.3
CO + $\frac{1}{2}$ O ₂	2808.1	23.4	15.818	1	0.3
CO + H ₂ O	2227.6	18.5	0.4438	1	0.6
C ₃ H ₆ + 4.5 O ₂	3080.5	25.6	2.3059	1	0.4

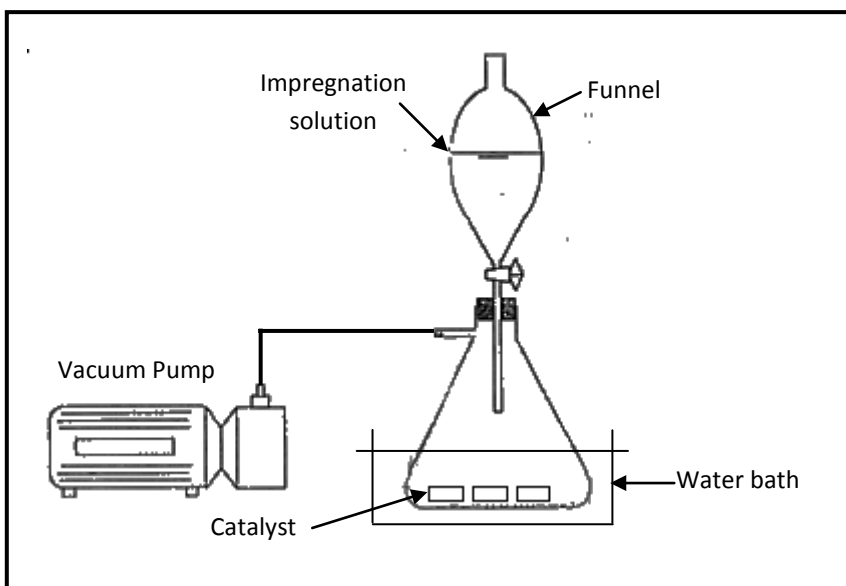


Figure (1): Impregnation apparatus

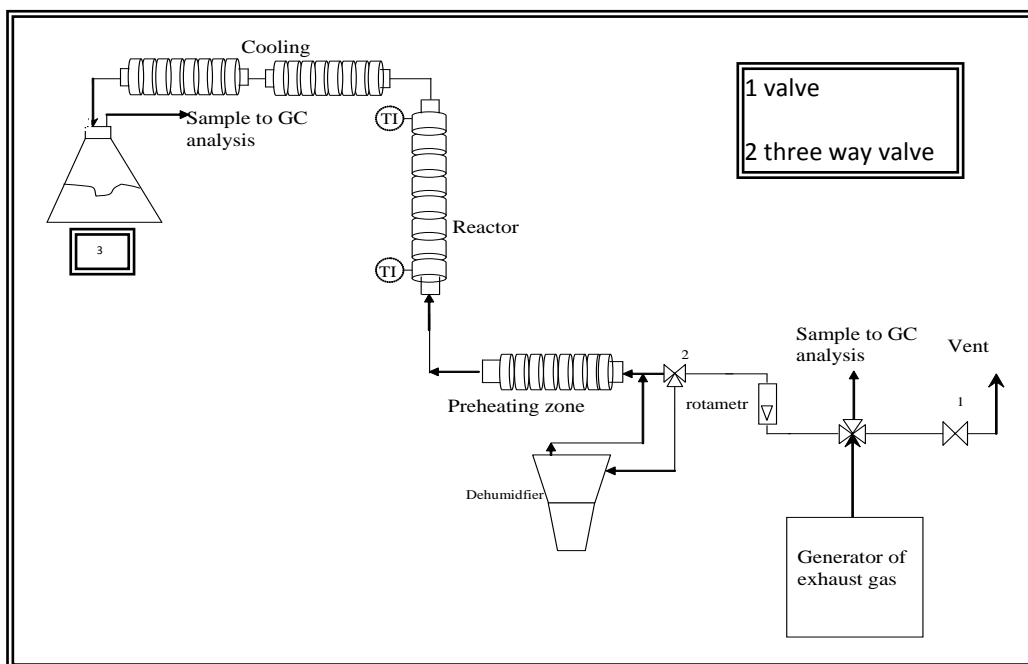


Figure (2): Schematic diagram of experimental setup

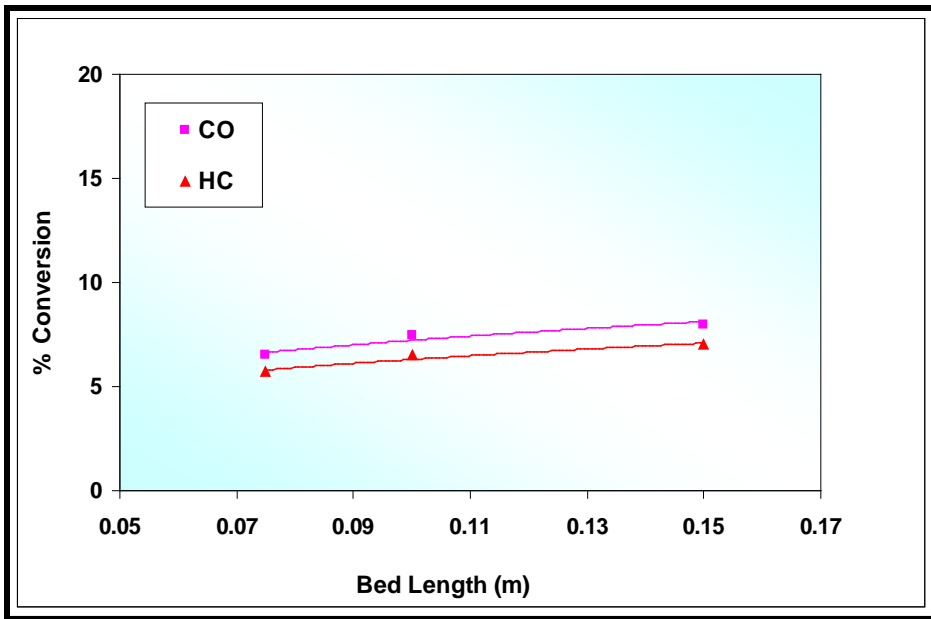


Figure (3):- Effect of bed length of catalyst on conversion at 373K.

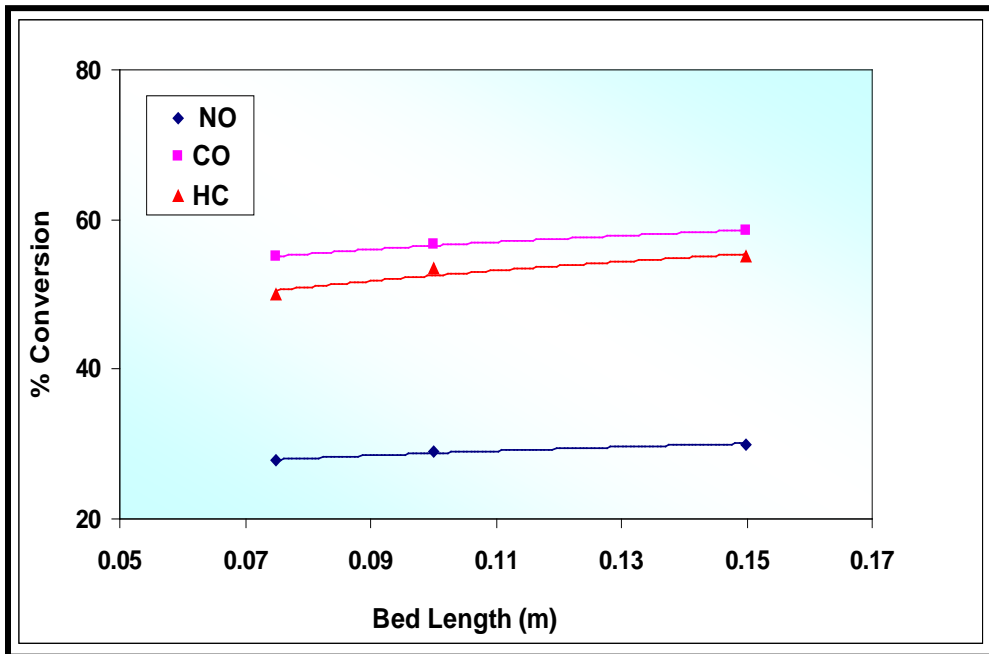


Figure (4):- Effect of bed length of catalyst on conversion at 473 K.

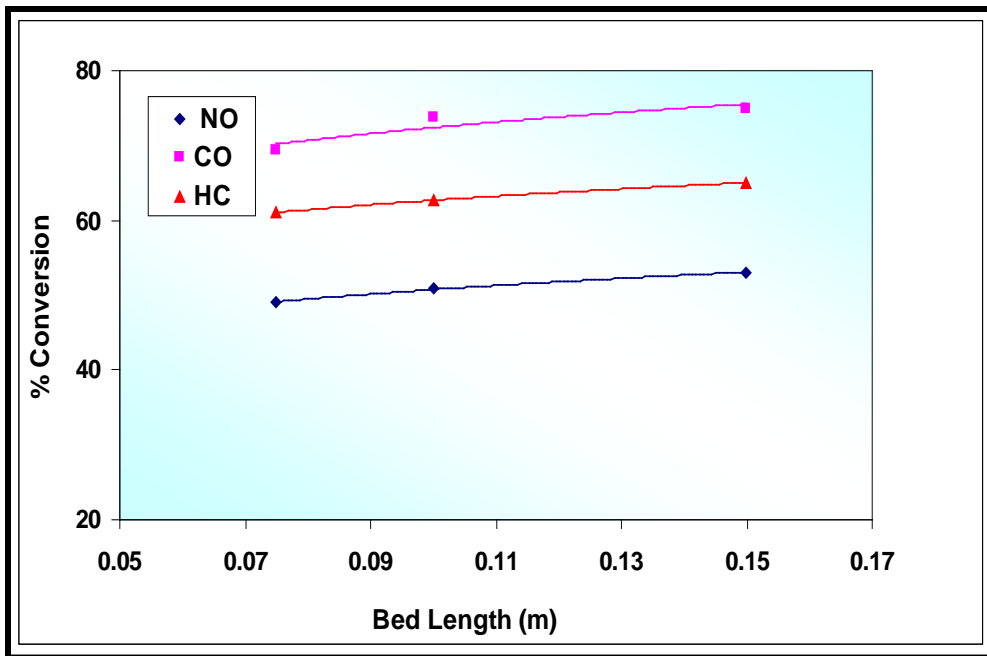


Figure (5):Effect of bed length of catalyst on conversion at 573K.

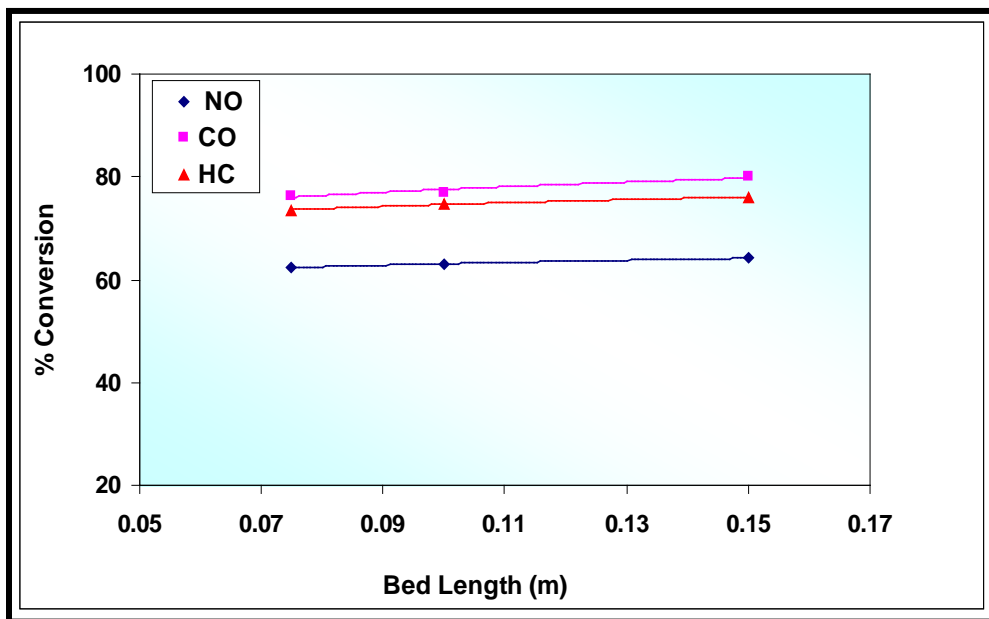


Figure (6):- Effect of bed length of catalyst on conversion at 673 K.

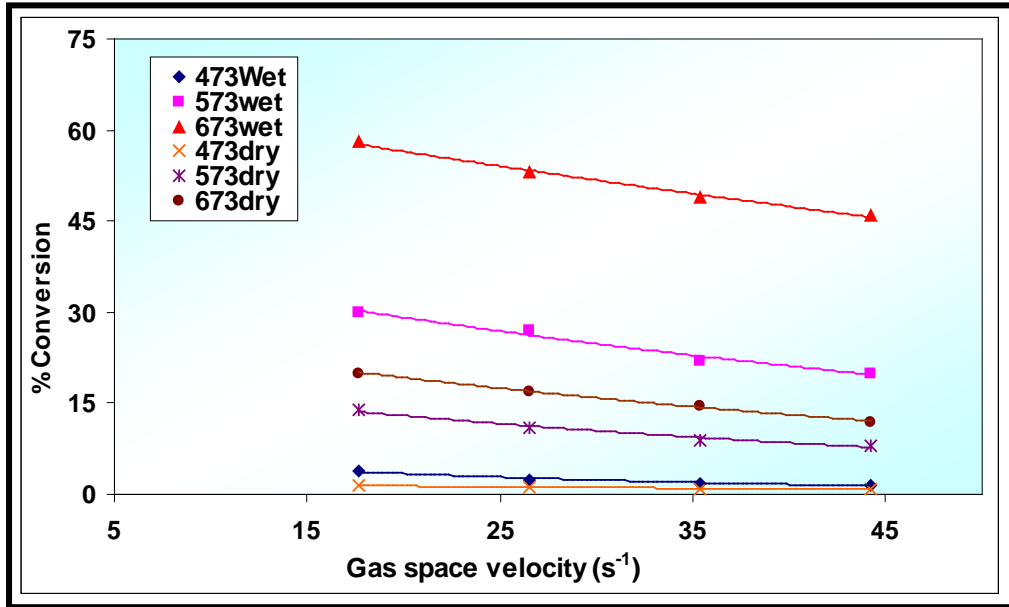


Figure (7):- Effect of water on NO conversion.

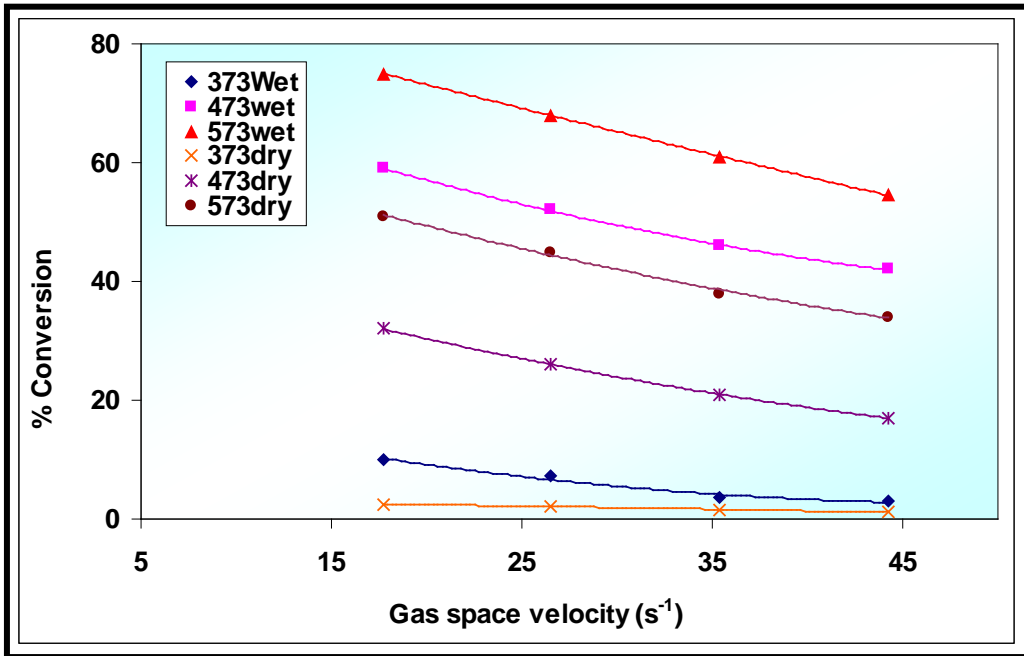


Figure (8):- Effect of water on CO conversion.

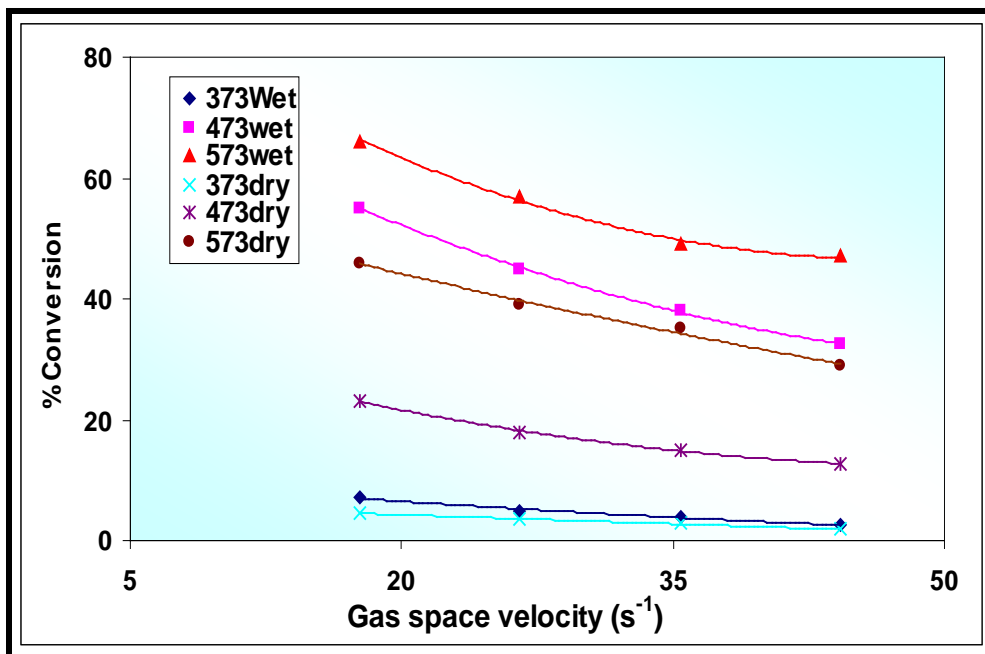


Figure (9):- Effect of water on C_3H_6 conversion.

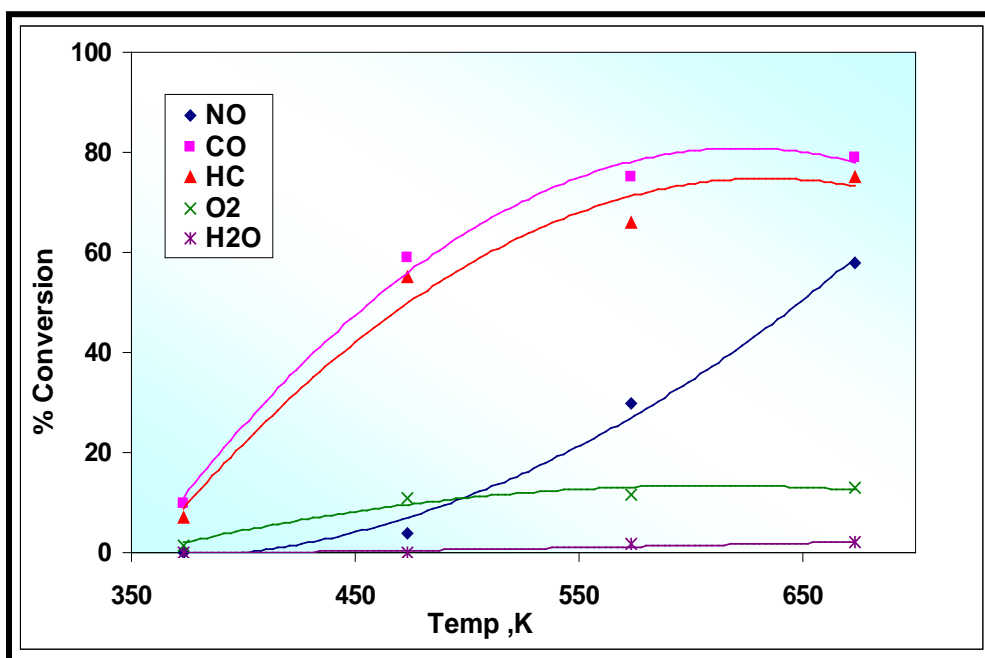


Figure (10):- Effect of temperature on conversion percentage using commercial catalyst for wet condition.

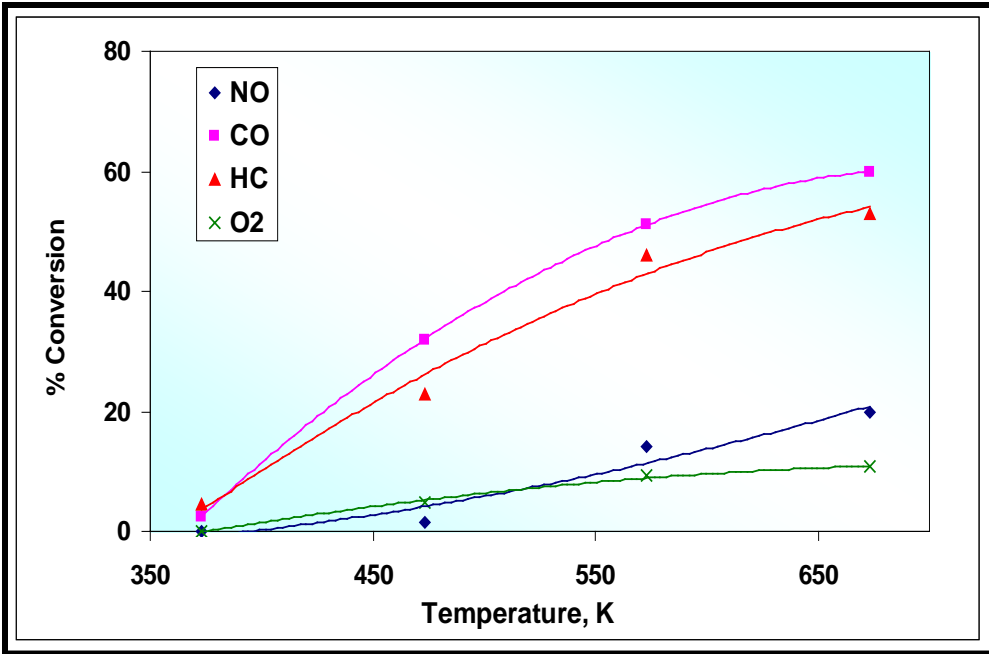


Figure (11):- Effect of temperature on conversion using commercial catalyst for dry condition.

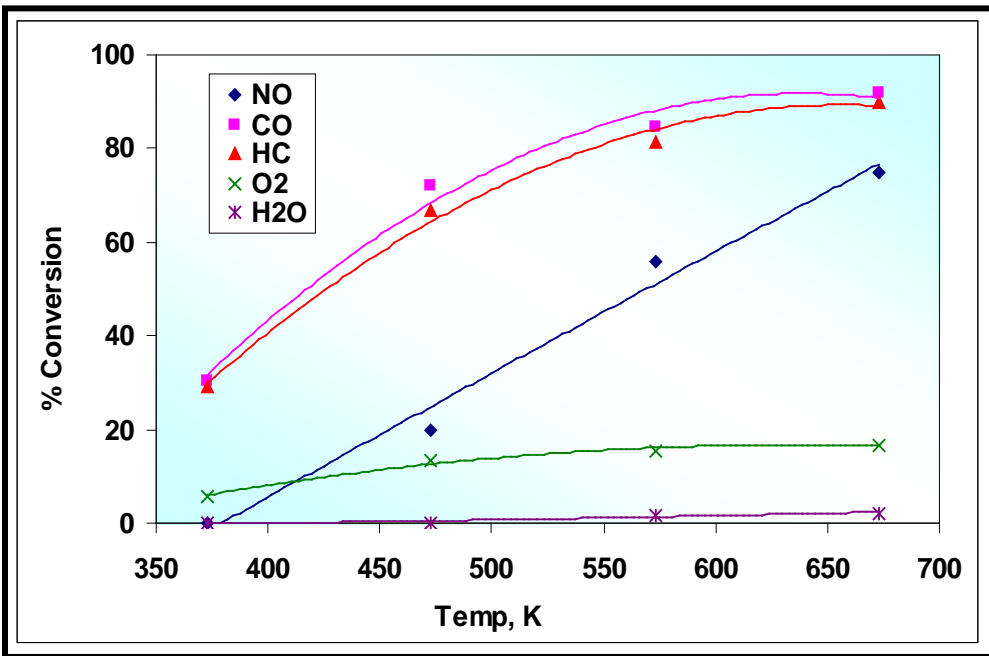


Figure (12):- Effect of temperature on conversion percentage for Pt loading.

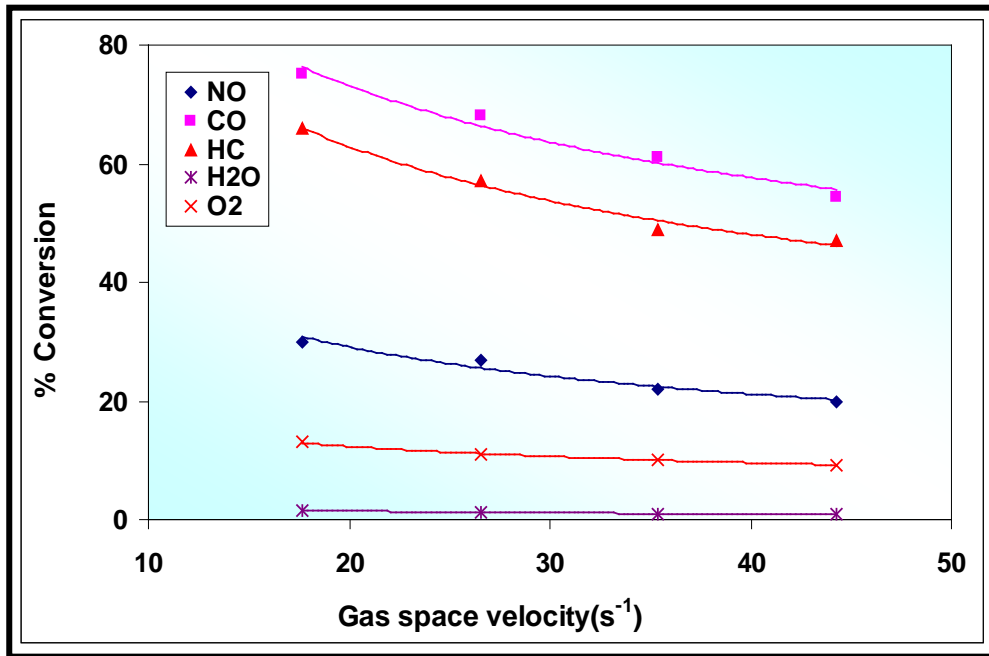


Figure (13):- Effect of gas space velocity on conversion percentage of components at wet condition at 573 K.

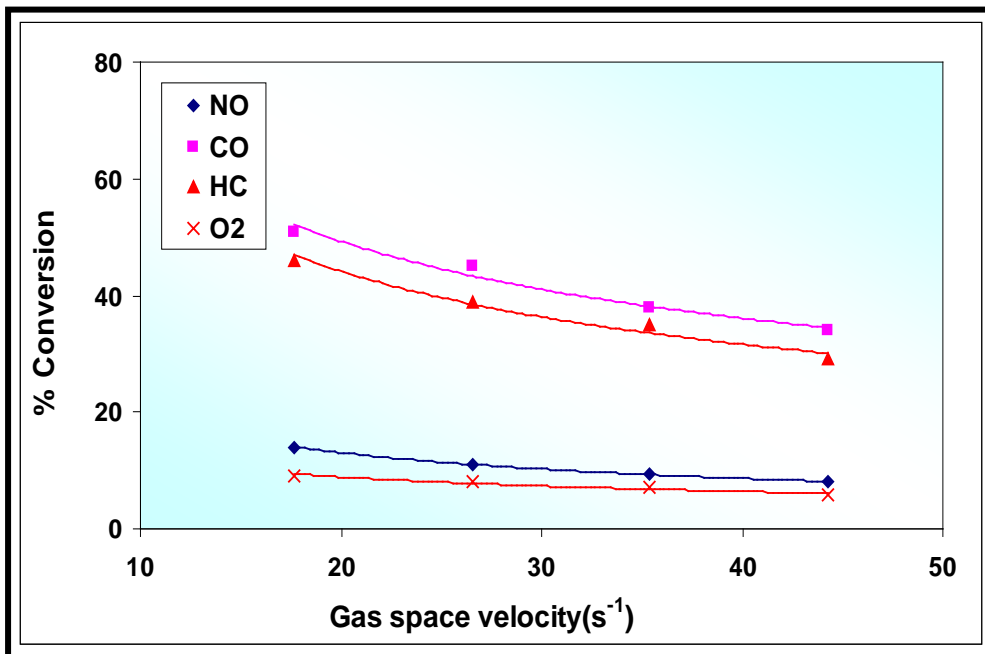


Figure (14):- Effect of gas space velocity on conversion percentage of component for dry condition at 573 K.

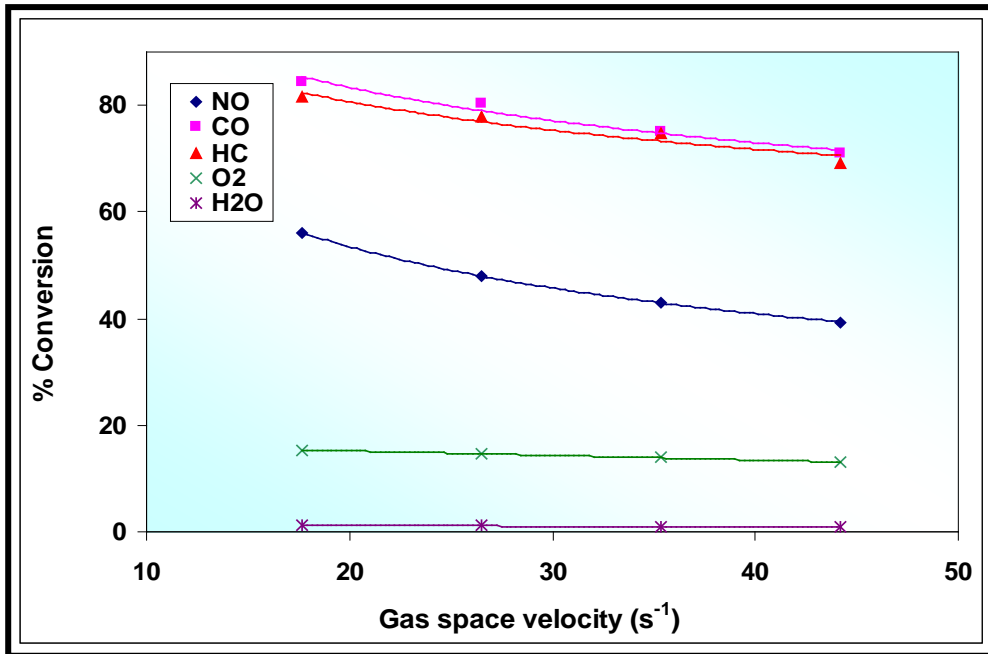


Figure (15):- Effect of gas space velocity on conversion percentage of component on Pt loading at 573 K.

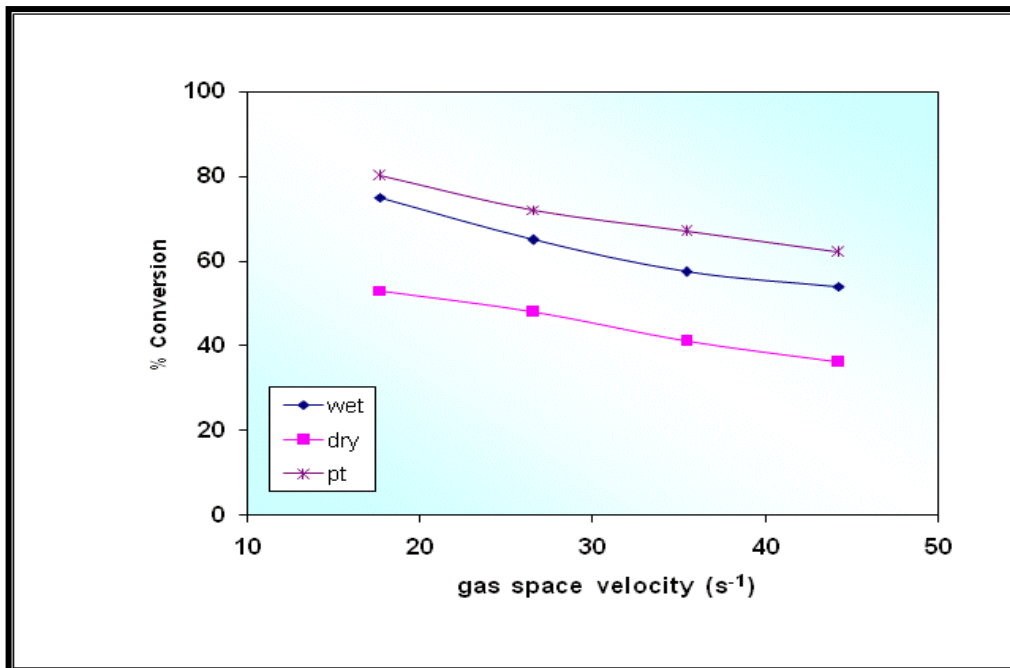


Figure (16):- Effect 0.4% loaded of Pt metal on the conversion percentage of NO component at temperature of 673 K.

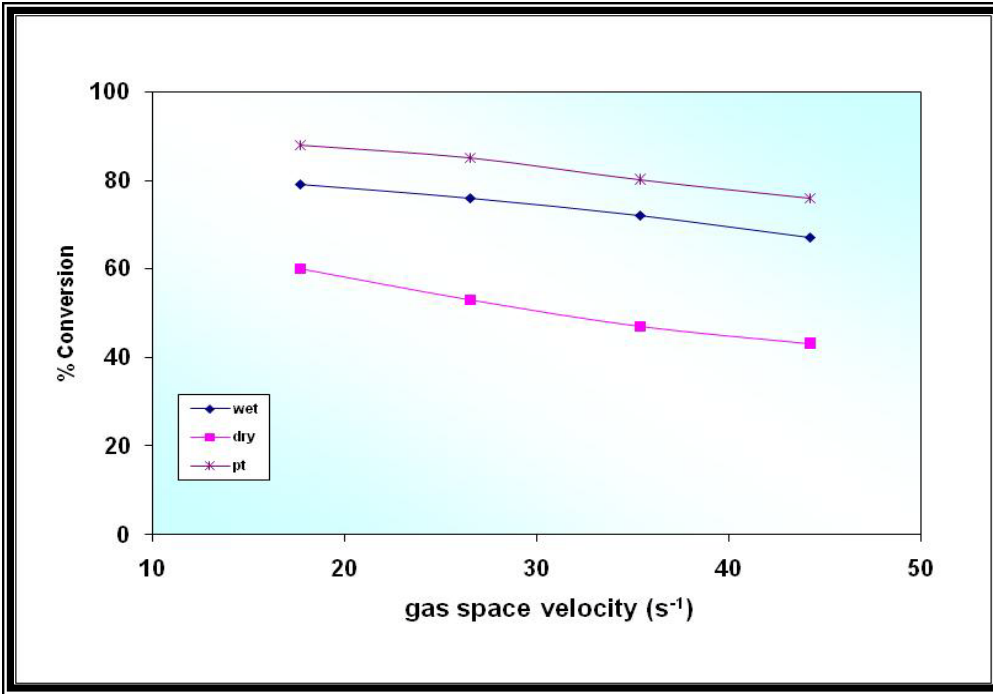


Figure (17):- Effect of 0.4 % loaded of Pt on the conversion percentage of CO at temperature of 673 K.

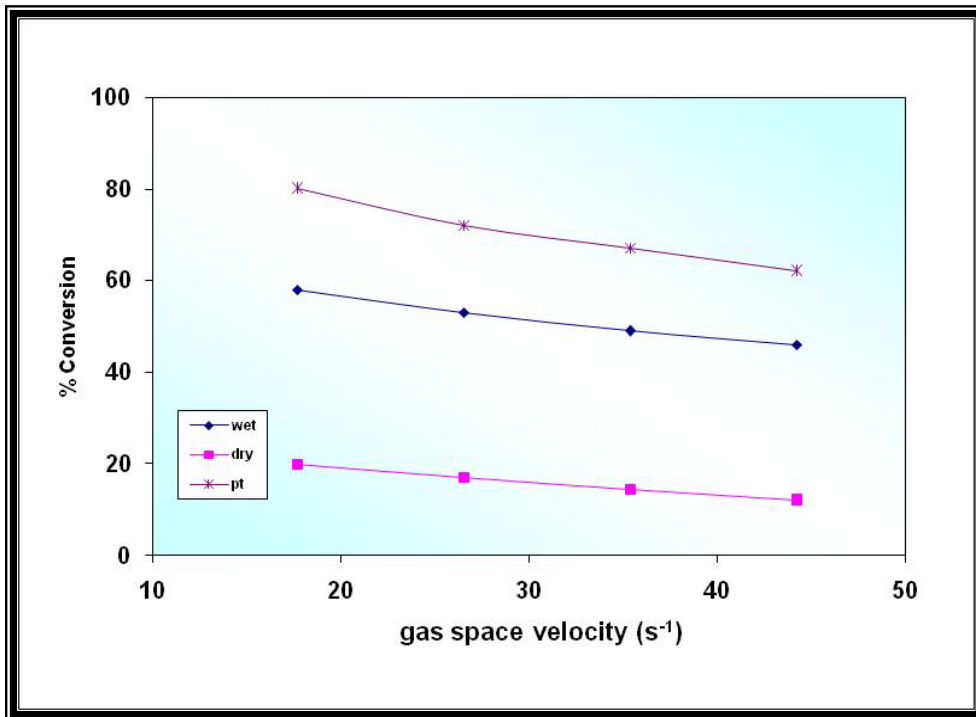


Figure (18):- Effect 0.4% loaded of Pt on the conversion percentage of C₃H₆ at temperature of 673 K.

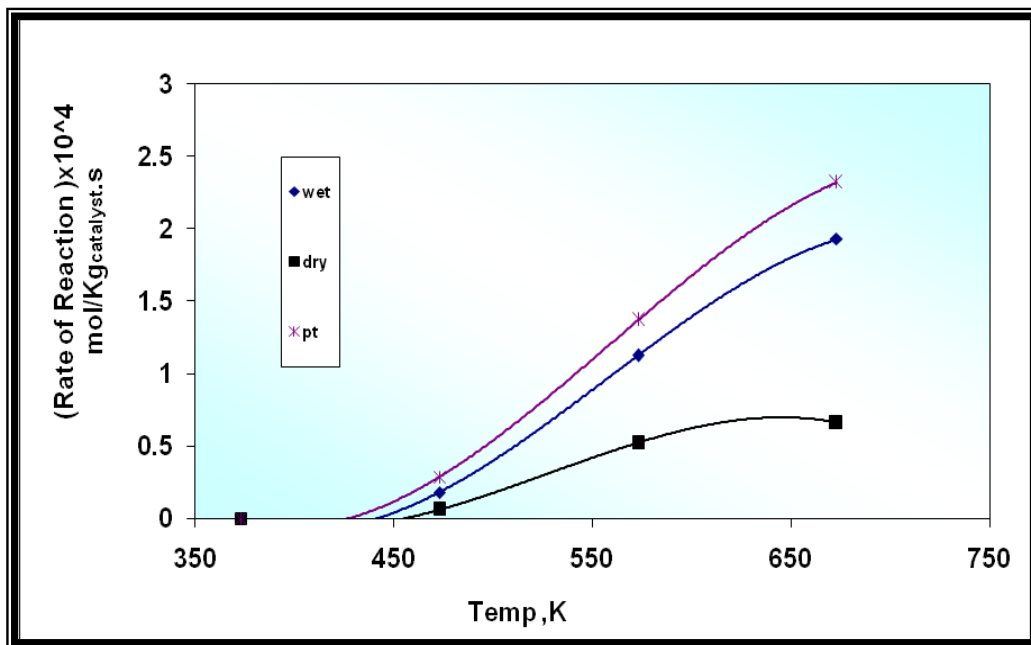


Figure (19):- Effect of temperature on the rate of reaction for NO component at gas space velocity (17.69 s^{-1})

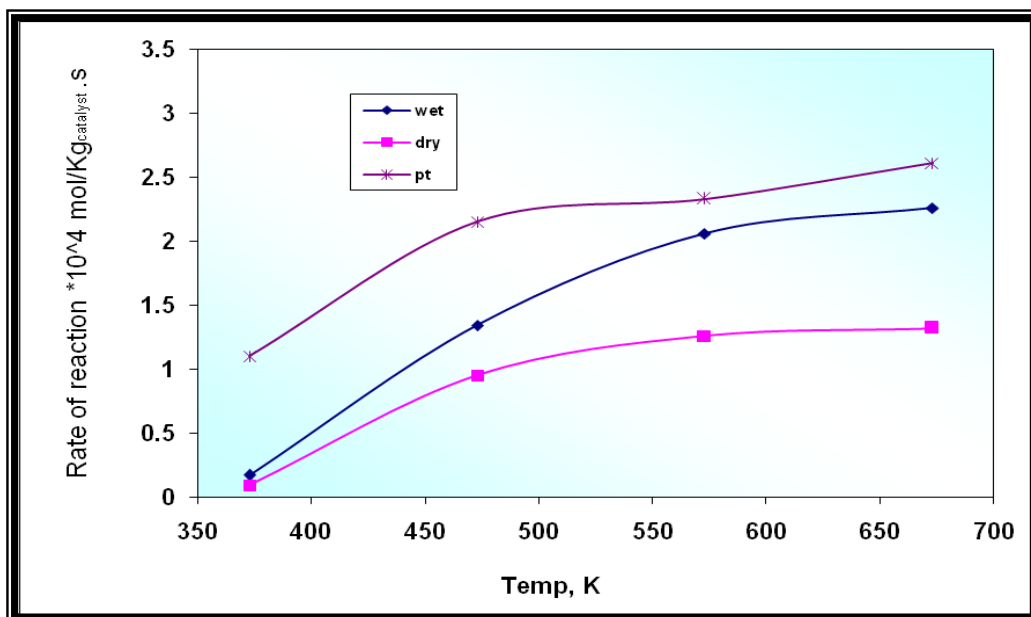


Figure (20):- Effect of temperature on the rate of reaction for CO component at gas space velocity (17.69 s^{-1})

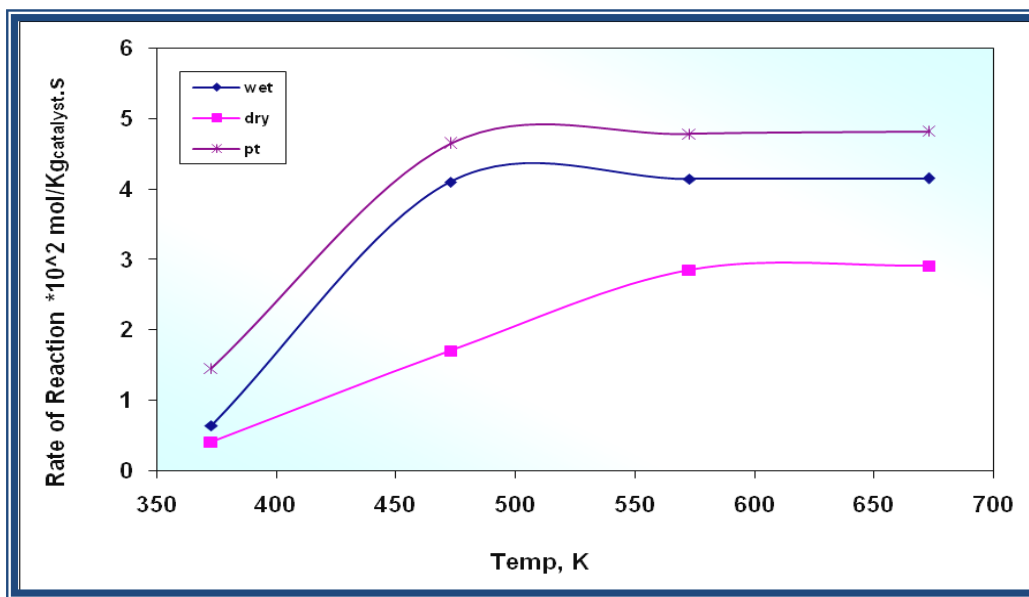


Figure (21):- Effect of temperature on the rate of reaction for HC component at gas space velocity (17.69 s^{-1})

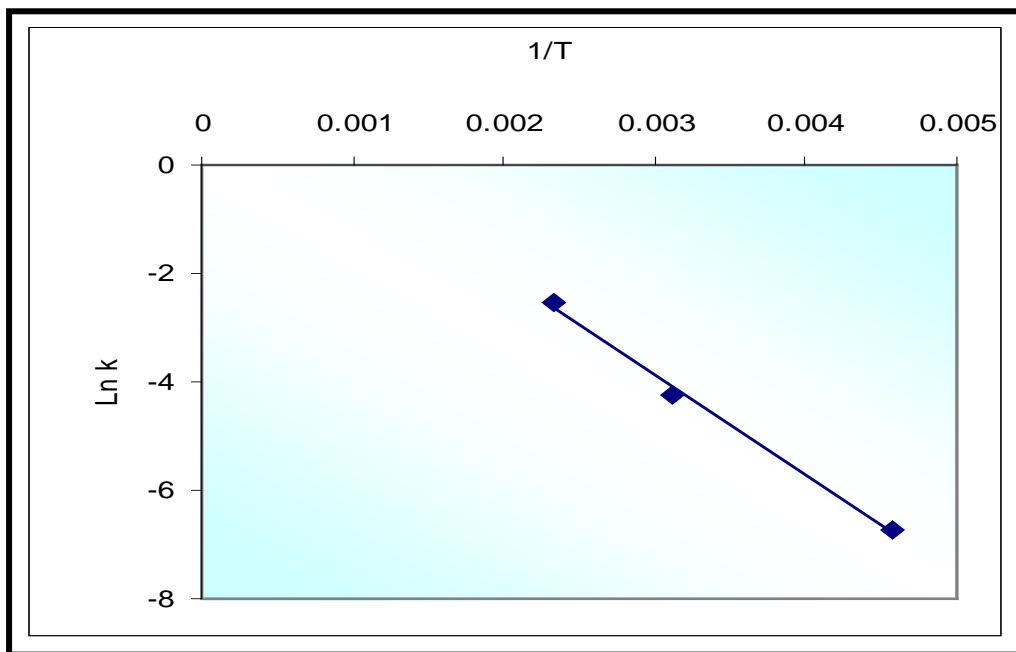


Figure (22):- Arrhenius plot for CO + NO for dry condition of commercial catalyst.

دراسة تأثير تحميل البلاينيوم على أداء العامل المساعد التجاري المونوليثي في إزالة الغازات الملوثة من الغاز العادم

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الخلاصة

أن مفاعلات العوامل المساعدة المونوليثية لها استخدامات متعددة في العمليات الصناعية وخصوصاً العمليات الحاوية على أجهزة تكنولوجية ولذلك فإنه بدأ التركيز على مفاعلات الحاوية على العوامل المساعدة في مكان الاحتراق الداخلي .

تضمن البحث دراسة عملية ونظرية لكفاءة أداء المفاعل المونوليثي (Monolithic Reactor) لمعالجة عمليات الأكسدة والاختزال لغازات الاحتراق (NO, CO, HC, O₂ and H₂O) الناتجة من مائع احتراق الكازولين .

ان عملية المعالجة تمت باستخدام العامل المساعد السيراميكي التجاري والعامل المساعد المحمل على العامل المساعد السيراميكي (0.4 % بلاينيوم) .

تم تصميم ونصب وحدة مختبرية ذات مفاعل من الفولاذ بقطر 0.02 متر وارتفاع 0.2 متر . تم دراسة أداء العامل المساعد التجاري المونوليثي والعامل المساعد المحمل بحدود السرعة الفراغية الغازية 17.69 – 44.32 /ثانية ، درجة الحرارة بحدود 373 – 673 كلفن وطول فرشاة العامل المساعد من 0.075 – 0.15 متر تحت ضغط جوي اعتيادي ونسبة ثابتة للهواء إلى الوقود قدرها 14.6 .

أثبتت النتائج العملية إن نسبة التحول لغازات الاحتراق (NO, CO, HC, O₂ and H₂O) لا تتأثر بشكل ملحوظ بتغير طول فرشاة العامل المساعد المستخدم . عند دراسة تأثير السرعة الفراغية وجد بأن نسبة التحول تنخفض بزيادة السرعة الفراغية لكن نسب التحول تزداد بزيادة درجة حرارة المفاعل ، كما وجد إن نسبة التحول لغازات الاحتراق تتحسن بوجود الماء (Wet condition) مقارنة بغياب الماء (Dry condition) . أعطى العامل المساعد المحضر 0.4 % بلاينيوم أعلى نسبة تحول لأكسدة غاز CO مقارنة بأكسدة HC واختزال NO.

الكلمات الدالة:العامل المساعد الثلاثي،البلاينيوم ،الطبقة المطلية.