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Nano Pt-W/γ-Al_2O_3 Catalyst is Used for Reforming Sweet Heavy Naphtha

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Abstract

Catalytic reforming process of sweet heavy naphtha to produce high octane number gasoline (reformate) using bimetallic platinum (Pt) and tungsten (W) loaded on Nano γ-Al_2O_3 as support, prepared by wet impregnation method and characterization by atomic absorption spectrophotometer (AAS), atomic force microscope (AFM), scanning electron microscopy (SEM), surface area BET and crushing strength. Results showed high surface area 340.1m^2/mg, pore volume 0.326 cm^3/gm, pore size is 6.139 nm and average particle size 64.64 nm when loading 0.3wt%Pt and 0.3wt%W, on Nano γ-Al_2O_3. The activity of the prepared Pt-W/γ-Al_2O_3 Nano catalyst was investigated using sweet heavy naphtha at the temperature range (470 to 520°C), pressure of 15 bar and H_2/ HC ratio = 6 in a packed bed reactor for catalytic reforming pilot unit. The best result was obtained research octane number equal 88.5 for product gasoline at temperature 500°C. Last but not least, the results of adding tungsten to the Nano-catalyst gave an improvement in the chemical reactions.

Keywords: Nano γ-Al_2O_3, Bimetallic Catalyst, Sweet Heavy Naphtha, Octane Number.

استخدام المحفز النانوي (Pt-W/γ-Al_2O_3) في إعادة تشكيل النافثا الحلوة الثقيلة

الخلاصة:

عملية إعادة تشكيل تحفيزية للنفثا الثقيلة الحلوة لإنتاج بنزين عالي الرقم الأوكتاني (reformate) باستخدام البلاتين ثنائي المعدن (Pt) والتنغستن (W) المحمّل على نانو γ-Al_2O_3 كحامل, حُضرّت بطريقة التشريب الرطب، وحُدّدت خصائصها باستخدام مطياف الامتصاص الذري (AAS)، ومجهر القوة الذرية (AFM)، والمجهر الإلكتروني الماسح (SEM)، والمساحة السطحية (BET)، وقوة السحق. أظهرت النتائج ارتفاع المساحة السطحية إلى 340.1 متر مربع/جم، وحجم المسام إلى 0.326 سم مكعب/جم، وقطر المسام إلى 6.139 نانومتر، ومتوسط حجم الجسيمات إلى 64.64 نانومتر عند تحميل 0.3% وزناً من البلاتين و0.3% وزناً من W على نانو γ-Al_2O_3. تم دراسة فعالية المحفز النانوي المُحضر من (Pt-W/γ-Al_2O_3) باستخدام النافثا الحلوة الثقيلة عند نطاق درجة حرارة (470 إلى 520 درجة مئوية)، وضغط 15 بار، ونسبة $H_2/HC = 6$ في مفاعل حشوات لوحدة تجريبية للإعادة التشكيل التحفزية. وتم الحصول على أفضل نتيجة بحثية، حيث بلغ الرقم

الأوكتاني 88.5 للبنزين الناتج عند درجة حرارة 500 درجة مئوية. وأخيرا وليس آخرا، أظهرت نتائج إضافة التنغستن إلى المحفز النانوي تحسنا في التفاعلات الكيميائية.

1. Introduction

Catalytic reforming is a very significant process in the crude oil refinery It is defined as the restructuring of the hydrocarbon molecules in sweet heavy naphtha (SHN) for producing high octane gasoline(reformate) as the main product with hydrogen and liquefied petroleum gas (LPG) as occasional products. Generally, in three or four fixed bed reactors the naphtha reforming process is accomplished[1] [2]. The study appeared that gasoline is essentially aromatic, iso-paraffinic and naphthenic nature. The aromatic hydrocarbons compounds in gasoline contribute significantly to the octane number. N-paraffins have a low octane number and their octane number lowering with increasing molecular weight, isoparaffins have a higher octane number when compared to the identical molecular weight as n-paraffins. Naphthenic hydrocarbons compounds have a bit high octane number[3]. The heart of the reformer unit is the catalyst, subsequently, most of the enhancement in the reformer unit efficiency has been due to the development of catalysts. Ago 1940, was molybdenum oxide has carried the alumina catalyst in reformer unit reactors with a short period of time among regenerations for catalysts activity [4] [5] [6]. In 1950 the catalyst fluidized bed type was used with continuous regeneration in the reformer unit to reduce the cycle times and increases catalyst activity. And also in 1948 introduced platinum-alumina catalyst platforming process was first by Universal Oil Products (UOP), when it was discovered that platinum(Pt) is more stable and active than catalysts formerly used. This catalyst made it possible to give a higher reformer intensity to meet the requirements of high-quality gasoline engines[4] [5] [6]. In 1969 chevron research advertised its rhenium (Re) as promoted a second metal to platinum-alumina catalyst and the effect of rhenium is to reduce amount of carbon deposition above the surface of catalyst[7] [4] [5] [6]. The deactivation of platinum is due to the formation of coke, as this requires the use of high pressure, and this severity in the operating conditions of the catalyst leads to the use of a dual-function catalyst by addition a second metal such as tin, rhenium or tungsten [8]. The addition of tungsten (W) to platinum (PT) is a successful promotion of a catalytic reforming as it has improved performance significantly and this has been shown by the low precipitation of coke on the catalyst surface, high thermal stability for catalyst and resistance to high operating temperatures. Perhaps this high catalyst stability is due to the formation of mixed surface oxides of W6 robustly connected with Al_3 stabilization the alumina surface, this works against the rearrangement of the crystal structure at high operating temperatures, as well as the positive effect of tungsten against platinum sintering and increase the selectivity for total aromatics product, especially for small amounts from (0.1wt % to

0.3wt%) of tungsten because the larger amounts of (W) lead to decrease in selectivity[9] [10]. The support material is often the amorphous pored refractory oxide, and generally without crystalline molecular sieves, was calcined at a temperature above 800°F. such as (Al_2O_3, silica-Al_2O_3, silica, γ-Al_2O_3, theta-Al_2O_3, zirconia, titania, and magnesia). but, γ- Al_2O_3 is widely utilized because it has a high surface area [11] [12]. γ- Al_2O_3 is used in the synthesis of the catalyst as its support because of its high thermal stability and ability to form into parts small (spherical or cylindrical) mechanically stable, accordingly γ-Al_2O_3 is used as catalytic support in different types of chemical reactions[13]. Generally reforming catalyst is a bi-metal and functional catalyst include a metal function basically platinum, should be well dispersed on the acidic carrier for the purpose of maintaining a high activity of the catalyst because a higher metallic dispersion can reduce the effect coke deposition on the surface of the catalyst active sites, acid function commonly is chloride and Al_2O_3. The metal function stimulates dehydrogenation and hydrogenation reactions and the acid function enhances the isomerization, dehydrocyclization and hydrocracking reactions, for a purpose to obtain the best effectiveness of the reformer catalyst, it is necessary to achieve an acceptable balance in the performance of metallic and acidic functions[14] [15] [16]. Naphtha reformer units are commonly classified into three types depending on the catalyst regeneration process type as: (A) Semi-Regenerative Catalyst Reformer (B) Cyclic Regenerative Catalytic Reformer (C) Continuous Catalyst Regeneration Reformer[17] [14] [18] [19]. The purpose of this research is to study the extent of the effect of adding tungsten to the catalyst on the nature of reforming reactions under different operating conditions.

2. Materials and Method

1.2. Feed

Sweet heavy naphtha (SHN) is provided by the midland refineries company/Al-dura refinery as feed for catalyst activity test in reforming pilot unit. Sweet heavy naphtha properties are list in Table (1).

Table (1): Properties of (SHN).

Specification		Value
Sulfur content, ppm		0.5>
Water content ppm		(10-15)
RON		55
Density, g /cm^3		0.74
I.B.P,°C		85
E.B.P,°C		188
(Chemical Composition	Paraffins	72.59
wt % of sweet Heavy Naphtha)	Aromatics	13.56
	Naphthenes	13.55
	Olefin	0.3

2.2. Materials

Chloroplatinic acid (H_2PtCl_6. $6H_2O$ salt- British Drug House (BDH) Company, ≥ 99.995 % purity) was used as sources of platinum metal, ammonium tungstate ($(NH_4)_{10}W_{12}O_{41}.5H_2O$ salt- HOPKIN & WILLIAMS LTD. Company, ≥ 99.99 % purity) was used as sources of tungsten metal, Nano gamma alumina powder previously prepared [20] and finally poly vinyl alcohol (PVA) as a binder.

2.3. Catalyst preparation

Nano Pt -W/γ-Al_2O_3 catalyst were prepared by wet impregnation technique. A 60 g of Nano γ-Al_2O_3 powder as support previously prepared was loaded with 0.3wt% platinum metal using H_2PtCl_6. $6H_2O$ salt as the metal precursor, the platinum salt was dissolved easily in an amount of deionized water depending on pore volume of support Nano γ-Al_2O_3, producing a yellow to orange solution, then adding the solution Pt to powder Nano γ-Al_2O_3 with stirring at room temperature for 24 h. It was observed that approximately six hours after the mixing process, the color of the solution almost disappeared, indicating that the largest amount of the platinum salt compound had been impregnated, subsequently filtered by a vacuum system, then dried at 110 °C for 24 h and calcination at 450 °C for 4 h a heating rate of 10˚C /min. The resulting powder was loaded with 0.3wt% tungsten metal using $(NH_4)_{10}W_{12}O_{41}.5H_2O$ salt as the metal precursor with the same steps as platinum metal loading, also dried at 110°C for 24 h and calcination at 450°C for 4 h at a heating rate of 10˚C /min[21] [22] [6]. The prepared 60 g of Pt-W/gamma alumina catalyst was formation using poly vinyl alcohol (PVA) as a binder with 5% in the aqueous solution. The resulting in pastes were molded to extradite shape as cylindrical pellets via using mini screw extruder 2 mm diameter and 10 mm length of extradite Pt-W/γ-Al_2O_3 catalyst was dried at 110 ºC for 24 h and calcination at 500 ºC for 5 h at a rate of 10ºC/min [23] [24].

2.4. Activity of Catalyst

Catalyst activity was tested for the synthesized Pt-W/γ-Al_2O_3 catalyst via used a catalytic reformer pilot unit which was supplied by VINCI technology / France at Petroleum Research and Development Center (PDRC), Ministry of Oil. The unit composed of a fixed bed carbon steel reactor (1.5m length, 2.5cm outside diameter) equipped with control and cooling systems as displayed in Figure (1) and Figure (2). Experiences were done filling the reactor with 60 g of the prepared catalyst while the upper and lower parts of the reactor were filled with ceramic balls (5-6 mm). Six reaction temperatures of (470, 480, 490, 500, 510 and 520 ºC) with operating constant pressure 15 bar at H_2/ HC = 6 ratio, LHSV = 1 h^{-1} were investigated. Before doing experiments, the catalyst was purged from oxygen of catalytic reforming pilot unit and drying the catalyst via nitrogen gas at 350ºC, 10 bar for 5 h. After that, it was activated the catalyst (Pt-W/γ-Al_2O_3) by hydrogen gas at 450ºC, 15 bar for 6 h. (SHN) was pumped and mixed with H_2 gas then passed through the catalyst bed, and the reaction happen .The product was cooled by the cooling system and separated in separator to product gasoline which was gathered and analyzed using gas chromatograph (G.C) (type/Agilent 6890N-Network) to paraffin, aromatic and naphthene, as well (RON) was measured by the cooperative fuel research (CFR) engine (ASTM D 2699).

Fig. (1): Catalytic reforming pilot unit.

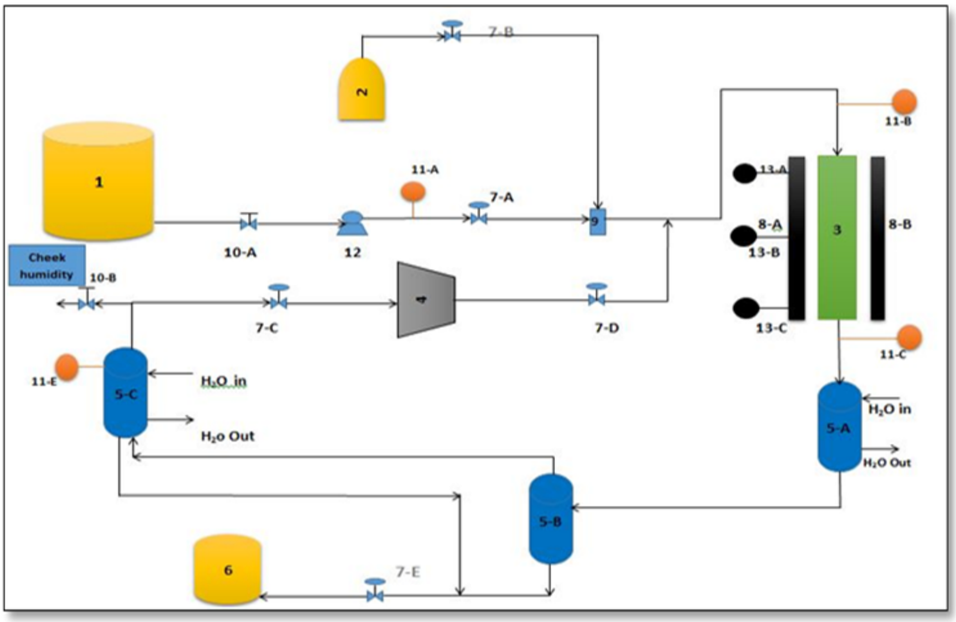

Fig. (2): Flow diagram of catalytic reforming pilot unit.

NO.	Deception
1	Tank of sweet heavy naphtha
2	Source of hydrogen gas
3	Reactor of catalytic reforming pilot unit
4	Compressor recycle of hydrogen gas
5(A, B, C)	Drums
6	Storage of reformate (gasoline)
7(A, B, C, D, E)	Flow control valve
8(A, B)	Heating jacket
9	Mixing point
10 (A, B)	Needle valves
11(A, B, C, D)	Pressure controller system
12	Pump
13(A, B, C)	Temperature controller system

3. Results and Discussion

3.1. Characterization of the Pt -W/ γ -Al_2O_3 Nano Catalyst

3.1.1. Loading Percentage of Platinum and Tungsten

The loading ratio for both the platinum and tungsten metals was tested by the atomic absorption spectrophotometer (AAS) test procedure, where the loading percentages of the platinum metal was 0.3wt% and the tungsten is 0.3wt% over the alumina support.

3.1.2. Particle Size and The Morphology of the Catalyst

To find the morphology of the surface and average particle size of the prepared P-W/γ-Al_2O_3 Nano catalyst, AFM analysis was performed. In Figure (3) displays the two-dimensional surface, irregular hexagonal structure of the prepared catalyst crystal. As revealed in Figure (4) Three-dimensional surface images, indicate that low agglomerated of prepared Nanocrystal are in the form of irregular hexagonal layers, as well spherical and scratchy in shape[25] [26]. The prepared Pt-W/γ-Al_2O_3 catalyst composed of particles with diameters ranged between 40-90 nm and the average diameter was 64.64 nm. The increase in average particle diameter and crystal centering during wet-impregnation technique of the synthesized catalyst happens due to loading Pt, W, on the support Nano γ-alumina which has average particle diameter was 42.71 nm[27] [28] [29].

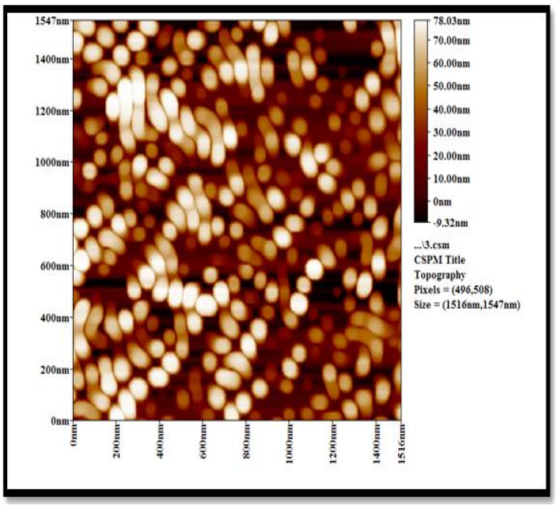

Fig. (3): Two-dimensional surface of prepared Nano Pt-W/γ-Al_2O_3 catalyst.

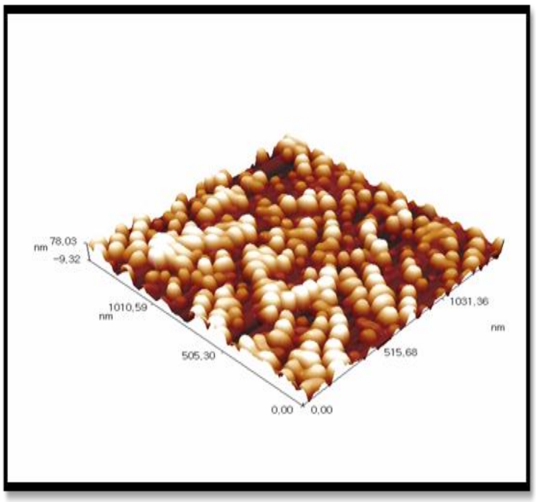

Fig. (4): Three-dimensional surface of prepared Nano Pt-W/γ-Al_2O_3 catalyst.

3.1.3. Field emission Scanning Electron Microscopy (FE-SEM)

Figure (5) shows the field emission scanning electron microscopy (FE-SEM) images of the prepared Nano Pt-W/γ-Al_2O_3 catalyst. increase particle diameter and growth crystal centering of the prepared catalyst after loading 0.3wt% platinum and 0.3wt% tungsten over Nano γ-alumina[29] [27]. It is clear that both (Pt, W) metals were distributed almost regularly on surface Nano γ-alumina, maybe which is explained by the regular increase in the size of the particles after the loading process. These results had an agreement with the results obtained from AFM analysis and test of BET surface area.

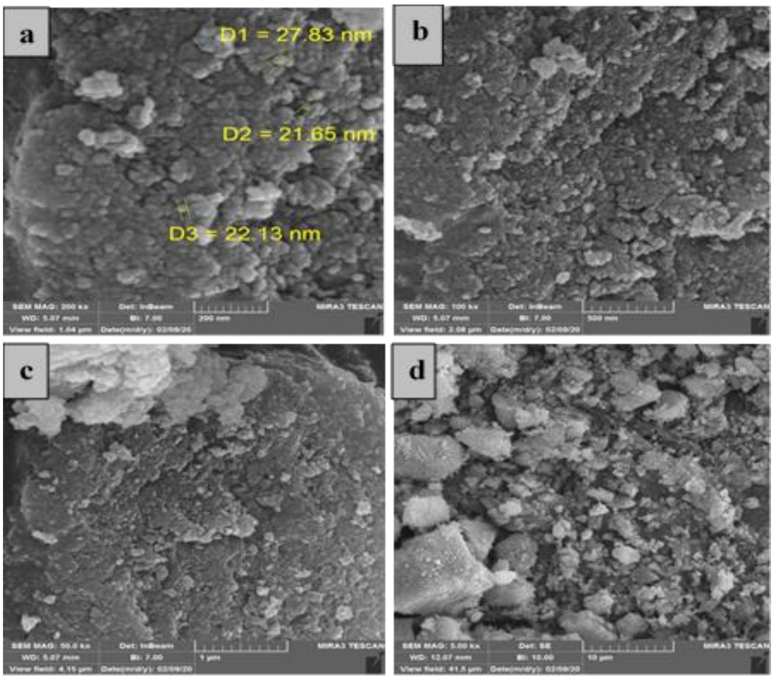

Fig. (5): FE-SEM images of the Pt/W γ -alumina Nano catalyst; (a-d).

3.1.4. Surface Area, Pore Volume and Pore Size

The surface area of Pt-W/γ-Al_2O_3 Nano catalyst was determine using the BET method by nitrogen adsorption/desorption isotherms. Pt-W/\γ-Al_2O_3 Nano catalyst surface area was 340.1m^2/g, pore volume 0.326 cm^3/g and pore size 6.139nm while Nano γ-alumina was previously prepared which had a surface area of 389.34 m^2/mg, pore volume 0.468 cm^3/g and pore size 8.07nm. The decrease in surface area, and pore volume probably due to blockage some pores due to loading platinum and tungsten on Nano γ-alumina and maybe the reason for the decrease in pore size is due to the deposition of both platinum and tungsten on the surface of the inner wall of the pore size during wet-impregnation method[30] [31].

3.1.5. Crushing Strength of Catalyst

The crushing strength of the prepared Pt-W / γ-Al_2O_3 nanocatalyst was 1.45 kg it indicates the stability and good mechanical resistance of the prepared catalyst [32].

3.2. Activity Testing of the Pt -W/ γ -Al_2O_3 Nano Catalyst

The activity of the synthesized catalyst sample was investigated for sweet heavy naphtha reformer reactions to produce high octane gasoline.

3.2.1 Effect of Temperature on Catalytic Reformer

It can be observed in Figure (6) that the prepared catalyst exhibits distinctive high activity with increasing temperature the proportion of aromatic compounds produced increased this due to happen dehydrogenation and dehydrocyclization reactions which are the best reaction at catalytic reformer increase octane number also the proportion of paraffin compounds decreased and decreasing of the percentage of naphthenic compounds. These are results agreeing with those obtained by the previous authors[4] [5] [15]. The quality of product gasoline symbolized by octane number and the proportion increase in an octane number range from (45.45 % to 60.90 %) at reaction temperature range (470-500°C), as revealed in Table (2). Nevertheless, when the reaction temperature increases to (510°C and 520°C), the percentage increase in an octane number decreases to 57.27, 55.45 respectively, and this is due to the catalyst activity reduced progressively with time due to the carbon deposition that occurred as a result of hydro cracking reactions, and thus coke covers the active sites of the catalyst thus decreasing the catalytic activity. these results are agreeing with the study of [33]. To reduce and control the accumulation of carbon materials, hydrogen gas has been used and however, hydrocracking is an undesirable reaction since it produces gaseous product but it may participate to octane enhancement via cracking of large molecular to such as normal-decane to C3 and C7 and the last will be transformed to aromatic[34]. The octane number increased from an initial value of 55 for sweet heavy naphtha to range from (80 to 88.5) of product gasoline at reaction temperature range (470-500°C) as revealed in Figure (7) and Table (2), and this is a result of the occurrence of reforming reactions on the surface of a prepared catalyst for sweet heavy naphtha that has been transformed into gasoline. The best result obtained was octane number = 88.5 for product gasoline at temperature 500ºC with an increase in the ratio of the octane number 60.90 % this is due to metallic function of the catalyst symbolized via (Pt&W) which increased the dehydro genation reactions of naphthenes and parraffins leading to increase the amount of aromatic compounds produced, and the last is the essential source of high-octane gasoline production as revealed in Figure (7) and Table (2), but when the temperature increases to (510°C and 520°C), the octane number of the gasoline product decreases to 86.5,85.5 respectively, and this decrease may be due coke is formed in the active sites of the catalyst, as well as high temperature leads to occurred increase in undesirable reactions such as hydrocracking, which reduces the octane number of the gasoline product [35] [36]. This shows that the reformer reaction is prefer using the affective metal loading and also via the high surface area of the carrier which leads to the desired increase of octane number for the product gasoline.

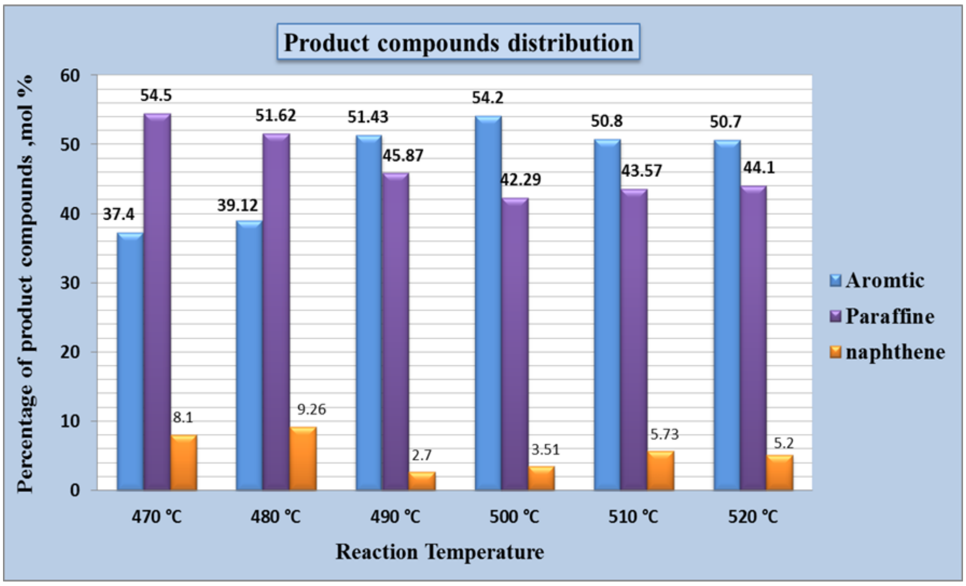

Fig. (6): Product compounds distribution for Pt-W/γ-Al_2O_3 Nano catalyst at pressure 15bar and different reaction temperature.

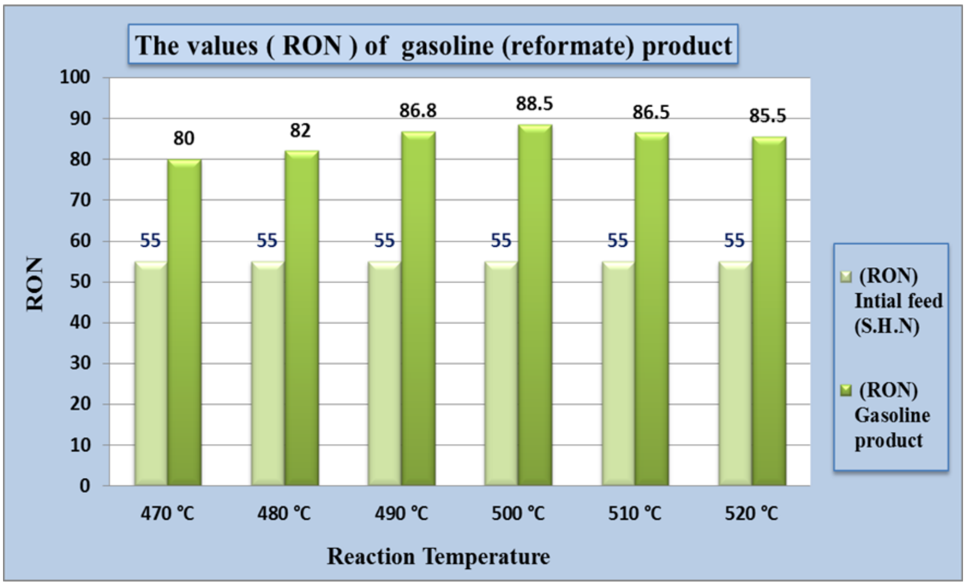

Fig. (7): The octane number for Pt-W/γ-Al_2O_3 Nano catalyst at pressure 15 bar and different reaction temperature.

Table (2): Percentage increase in octane number from initial value of sweet heavy naphtha to gasoline product for Pt-W/γ-Al_2O_3 Nano catalyst.

Temperature °C	(RON) of Reformate	(RON) of sweet heavy Naphtha	(RON) %
470	80	55	45.45
480	82	55	49.09
490	86.8	55	57.81
500	88.5	55	60.90
510	86.5	55	57.27
520	85.5	55	55.45

3.2.2 Effect Adding Second Metal (Tungsten) on Catalytic Reforming

To compare the prepared catalyst Pt-W/γ-Al_2O_3 with commercial catalyst Pt-Re/Al_2O_3 in reformer unit at Dura Refinery, it was found that the prepared catalyst more thermal stability works with temperature limits of (490-500)°C and high the octane number ranges (86.8-88.5), while commercial catalyst works with temperature limits of (480-490)°C and the octane number ranges (82-86) with less yield. After addition second metal tungsten as a promoter to Pt/Al_2O_3 catalyst, became the bimetallic catalyst more efficient, in terms of the resistance of Pt crystallites against sintering at high the temperature, the prepared catalyst has better thermal stability towards deactivation by reduced hydrocracking reactions and increase the selectivity and yield for aromatics compounds as revealed in Figures (9) and (10), depending on the previous study[10][37]. Catalytic naphtha reformer showed to progress a typical bi functional reactions happened on metallic site symbolized by (Pt&W) and the the acidic site represented by alumina (γ-Al_2O_3) as support[9]. Universal Oil

Product (UOP) has combined catalytic naphtha reforming reactions in an overall scheme showing pathways for the reactions and the specific catalyzing actions of metal and acid functionalities as shown in Figure (8). As it explains that some reactions are corroborative by metallic loading such as dehydro genation reactions whilst hydrocracking and dehydrocyclization reactions are corroborative via metallic and acidic properties of the catalyst. In whole cases, it can be concluded that metallic loading obtained an important role in whole catalytic reforming reactions[19].

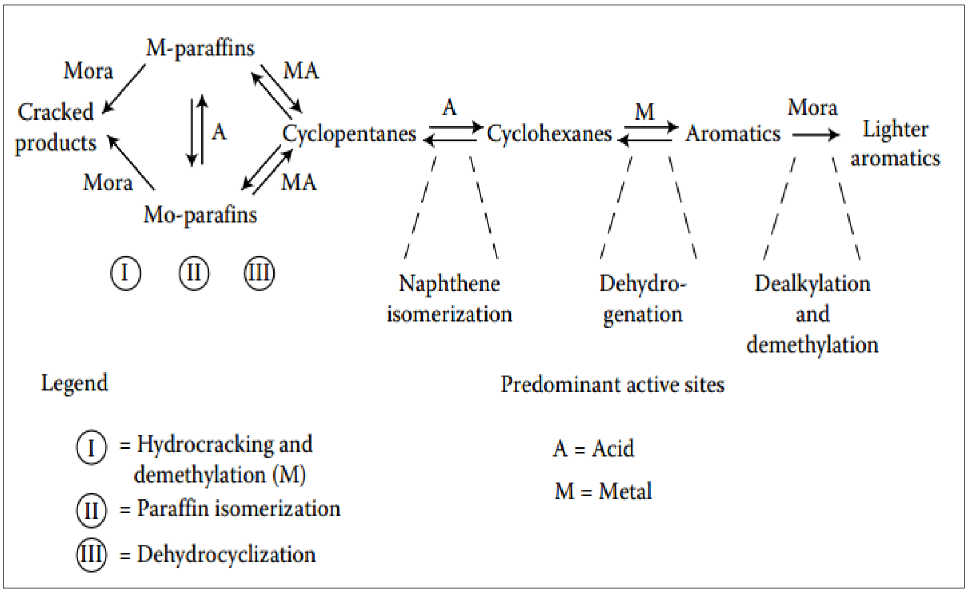

Fig. (8): Catalytic reformer reaction scheme over metallic [M]and acidic [A] sites of Pt-W/γ-Al_2O_3 bi functional[19].

3.2.3 Catalyst Performance and (Selectivity, Yield) of Gasoline

Commonly, the catalyst activity means how it well performed its duty in relation to the reaction rate which was mean that higher reaction rate, higher activity of the catalyst and catalytic reforming higher yield of aromatic was as well a good selectivity[15]. Gasoline yield means the volume of product gasoline to the volume of (SHN) as feedstock[38]. The acidic properties of the carrier alumina are affected via the metal loading and this leads to effect on the activity and selectivity of the synthesized catalysts, as the metal loading of (0.3wt%Pt and 0.3wt%W) showed a balance in the performance of the acidic and metallic function, according to the yield of reformate product at the different reaction temperature as revealed in Figure (9) corresponds to [39]. The yield of gasoline (reformate) depends on the extent to which the conversion of sweet heavy naphtha to reformate and increase or decreases the quality of product reformate as revealed in Figures (8) and (10**)**. The results showed that the gasoline yield between (80.5% to 90%), but the best yield was 85% at the highest octane number = 88.5 with reaction temperature 500°C for prepared catalyst investigated and the results have been inserted in Figure (9).

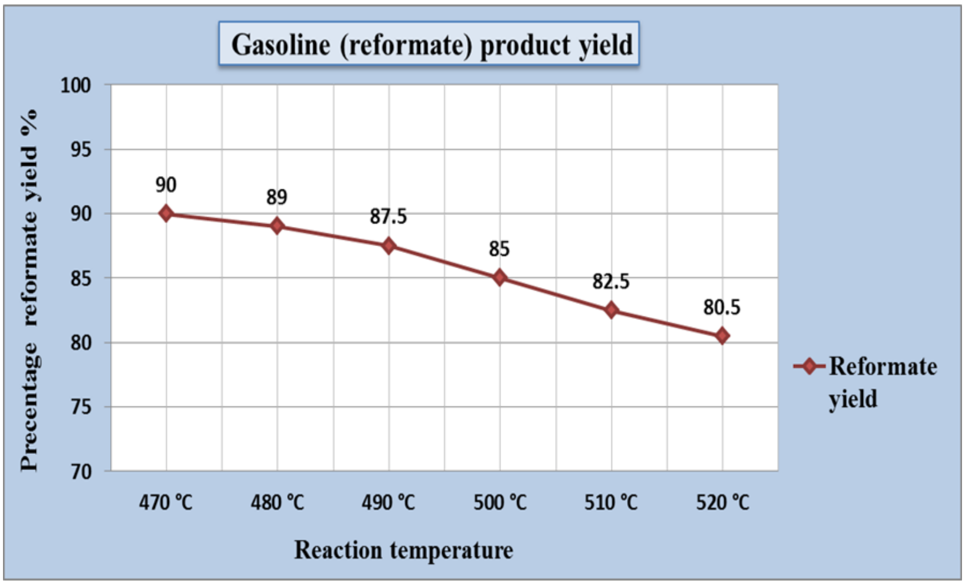

Fig. (9): Gasoline (reformate) yield for prepared Pt-W/γ-Al_2O_3 Nano catalyst.

Catalyst selectivity counts on the conversion ratio and in reforming reaction group selectivity for aromatic compounds is of great importance because it is responsible for the high quality of product gasoline due to their high octane number. The highest selectivity was obtained at 500°C, but when the temperature increased to 510°C and 520°C leads to a decrease in the selectivity, this is due to the low percentage of the aromatic compounds produced, Figure (10) shows that at a relatively high temperature the selectivity towards aromatics increases and as well the best performance was gained a low percentage of metal loading this corresponds to the study [39].

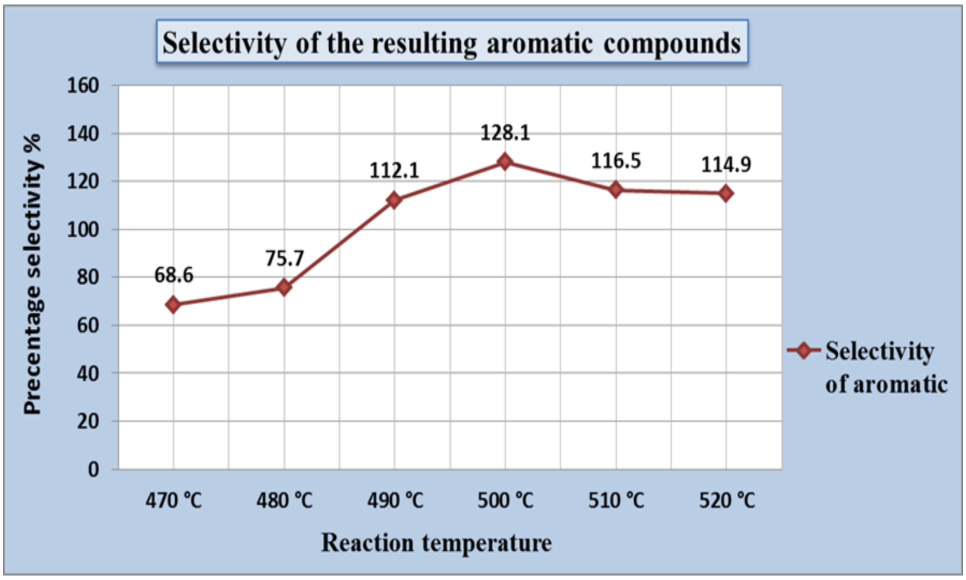

Fig. (10): Aromatic selectivity % for prepared Pt-W/γ-Al_2O_3 Nano catalyst.

4. Conclusions

The Nano Pt-W/γ-Al_2O_3 catalyst has been prepared successfully using wet-impregnation technique. The activity of prepared catalyst was tested in the reformer pilot unit, experiments showed a good activity level, stability and selectivity, at pressure 15 bar and temperature (470 to 520°C). A high octane number was obtained 88.5 for the gasoline product at pressure 15 bar and temperature 500°C. It's encouraging results can be the future nominee for the catalyst reformer unit in oil refineries after comparing the prepared catalyst Pt-W/γ-Al_2O_3 with commercial catalyst Pt-Re/Al_2O_3 as the prepared catalyst has better thermal stability towards deactivation by reduced hydrocracking reactions and increases the selectivity and yield for aromatics compounds.

Author Contributions Statement: Karrar A. M. Deabl contributed to the contributed to the Conception of the study; Methodology; Investigation and experiments; Data curation and analysis; Original draft preparation; and review and editing of the manuscript. Usama A. Saed Conception of the study; Methodology; Data analysis and interpretation; Original draft preparation; and review and editing of the manuscript. Huda H. M. Al-Muqdadi contributed to the contributed to the Conception of the study; Methodology; Investigation and experiments; Data curation and analysis; Original draft preparation; and review and editing of the manuscript. All authors have read and approved the final version of the manuscript.

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