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هيئـــــــة التحريــر

بحوث العدد

- "تحفة الأنام بتوريث ذوي الأرحام" دراسة وتحقيقاً
- الاستفهام ودلالاته في شعر خليفة التليسي
- قراءة في التراث النقدي عند العرب حتى أواخر القرن الرابع الهجري
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Catalytic Cracking of Heavy Gas Oil (HGO) Fraction over H-Beta, H-ZSM5 and Mordinite Catalysts

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Abstract

The objective of Catalytic cracking is a process which breaks down the larger, heavier, and more complex hydrocarbon molecules into simpler and lighter molecules by the action of heat and aided by the presence of a catalyst but without the addition of hydrogen. Heavy Gas Oil (HGO) was chosen as a feedstock to investigate in by catalytic cracking process on three different of zeolite catalysts (H-Beta, ZSM-5, and Mordinite) at different reaction temperatures (400, 425, 450 ^{o}C) using autoclave batch reactor. The results showed that the catalytic cracking of HGO over H-Beta, H-ZSM5, and Mordinite catalysts was successfully catalytically cracked into gases, gasoline and distillate products. Simulated Distillation (SIMDIST), Gas Chromatograph (GC) and Thermal Gravimetric Analysis (TGA) techniques were used to analysis the feedstock and reaction products. Catalytic cracking of HGO was studied increase, the yield of gases; gasoline and coke are increases, whilst the yields of distillate (207+ ^{o}C) are decreases. Catalytic cracking of HGO in the present of H-ZSM-5 catalyst at 450^{o}C cracking temperature shows the most interesting results with high Selectivity toward gasoline range (33.9 wt%), and the reaction products formed during the catalytic cracking of HGO are formed to be within the carbon distribution ranging from (C_5-C_{12}). It has been found that the cracking temperature has strong influence on the conversion and product distribution. When H-Beta, H-ZSM5, and Mordinite catalysts were used as a catalyst, it was noted that as the cracking temperature increase from 400-450 ^{o}C, the liquid yield was decreased from 76 to 69%, 73 to 68%, and 80 to 72% by weight ,respectively. TGA was used in this research to quantify the coke deposited on the surface of the catalysts tested. The percentage weight loss of coked catalysts resulting from catalytic cracking of HGO over H-Beta, H-ZSM5, and Mordinite catalysts are 28.6%, 30.6%, and 30% ,respectively.

Keywords: Catalytic Cracking; Heavy Gas Oil; Zeolite Catalysts (H-Beta, H-ZSM5 and Mordinite).

Introduction

In this way, heavy oils (fuel oil components) can be converted into lighter and more valuable products (notably LPG, gasoline and middle distillate components).

Catalytic Cracking of Heavy Gas Oil (HGO) Fraction over H-Beta, H-ZSM5 and Mordinite Catalysts

The catalytic cracking unit is known as the Fluidized Catalytic Cracking (FCC). The FCC is the most widely used secondary conversion process in the refinery industry.

The development of fluid catalytic cracking processes for the production of high octane gasoline is considered as one of the outstanding technological achievements of the oil industry in recent years. Through the advantages of the new technique, the oil industry has been able to meet the vital demands of the war effort for an unlimited supply of high-octane gasoline. The product so produced is of such strength that it must be cut with gasoline of lower octane rating in order to be used as fuel in combustion engines of present-day design. It is truly a super fuel. The FCC process upgrades a variety of heavy feedstocks to lighter products.

The use of catalysts in chemical reactions has been regarded as common practice by chemical operators for many years, yet knowledge of exact catalytic mechanism is not great. A catalyst is a substance which takes part in a chemical reaction without itself being altered or consumed. When properly chosen its presence will cause a reaction which normally proceeds at a low rate to accelerate tremendously. Catalysts, therefore, do not sponsor impossible reactions but instead select a low speed reaction and hasten the attainment of its equilibrium. By correct application of these catalytic principles, the oil industry is able to convert low grade furnace oil into super-octane gasoline, and conversion takes place readily at low temperature and atmospheric pressures. Such a conversion is known as a cracking process. Solid materials are usually used as catalysts in cracking processes. Since experimental data have indicated that reaction stimulation takes place on the surface of a catalyst, industrial research has commissioned many projects concerned with the study of cracking reactions in relation to catalytic surface variables.

1. Experimental Work

The feedstock used in the current experimental work was gas oil fraction. Gas oil is a middle distillate of crude oil distillation that boils in the range of 275-375 ^{o}C and contains a mixture of paraffins, naphthenes, aromatics and olefins. Libyan gas oil derived from the Hamada, Feil and Shrara fields supplied by the Zawia Oil Refinery Company were used as a feedstock in this research.. Three different types of zeolite catalysts namely H-beta, H-ZSM-5, and Mordinite were used as a catalytic material in the present work. The Batch autoclave reactor used in this research.

Catalytic Cracking of Heavy Gas Oil (HGO) Fraction over H-Beta, H-ZSM5 and Mordinite Catalysts

1.1. Experimental Procedures:

The reactor with 130 g feed was gradually heated at the rate of 10 ^{o}C/min and temperature was kept at 400 ^{o}C for 1 h under the nitrogen blanket. All runs were made at reaction time of 1 h and at temperatures of 400, 425, and 450 ^{o}C.

1.2. Feedstock and Reaction Products Analysis.

The reaction products collected from the outlet of the batch reactor were classified into gases, liquid hydrocarbons, and coke deposited on the catalyst surface. The feedstock (heavy gas oil) and liquid reaction products were subjected to detail analysis using GC, and SIMDIST techniques whilst, gas products were identified using GC technique. Thermal gravimetric analysis (TGA) was used to quantify the carbon deposited on the catalyst.

2. Results and Discussion

In this study, the results of catalytic cracking of HGO over H-Beta, H-ZSM5, and Mordinite catalysts are presented. GC and SIMDIST techniques were used to identify the hydrocarbon and boiling point distribution of feedstock (gas oil) and reaction product respectively.

2.1. Catalytic Cracking of Heavy Gas Oil (HGO).

The Catalytic cracking of HGO over H-BETA, H-ZSM5, and Mordinite catalysts was investigated. The cracking temperature was fixed at 400, 425 and 450 ^{o}C for all experiments with reaction time 1 hour. Gases, liquid and coke products were collected after completion each run. GC and SIMDIST techniques have been used to identify the hydrocarbons present in the reaction products, whilst TGA technique was used to quantify the coke deposited on the catalyst.

2.2. Yields and Conversion:

The yields of reaction product gases, liquids and coke obtained from the catalytic cracking of HGO at 400, 425 and 450 ^{o}C are given in Table (1). It is clear from this table that both reaction temperature and catalyst type have strong influence on the reaction products. Table 1 shows the results of conversion and yield of gases (C_1-C_4), gasoline (IBP-207), condensate 207^{+} ^{o}C and coke over H-BETA, H-ZSM5, and Mordinite catalysts. It has been found that the yield of liquid produced from the catalytic cracking of HGO decreases as the reaction temperature increases whereas the yield of gases increase with decrease in reaction temperature.

Table 1: Yields of gases, liquid and coke from catalytic cracking of HGO.

Catalyst/Feedstock	H-ZSM5 HGO	Mordinite HGO	H-BETA HGO
Reaction temperature, 400°C Yields, wt%			
Gas	26.30	19.69	23.45
Liquid	73.54	80.20	76.43
Coke	0.16	0.11	0.12
Reaction temperature,425°C Yields, wt%			
Gas	29.86	23.72	25.43
Liquid	69.93	76.13	74.38
Coke	0.21	0.15	0.19
Reaction temperature,450°C Yields, wt%			
Gas	31.03	27.57	30.17
Liquid	68.69	72.19	69.6
Coke	0.28	0.24	0.23

2.3. GC Results.

As stated earlier that GC has been used to identify the hydrocarbon present in feedstock (HGO) and reaction products. Carbon distribution of feedstock (HGO) and reaction products at different cracking temperature using H-Beta, H-ZSM5, and Mordinite catalysts are shown in Figures 1, 2, and 3 . As it is clear from Figure 1 that it is obvious from the mentioned figures that as the cracking temperature increase, the yield of light products increases. The most dominated hydrocarbon present in feedstock is $C_{20,}$ as the cracking temperature increase the light products formed during the reaction.

Catalytic Cracking of Heavy Gas Oil (HGO) Fraction over H-Beta, H-ZSM5 and Mordinite Catalysts

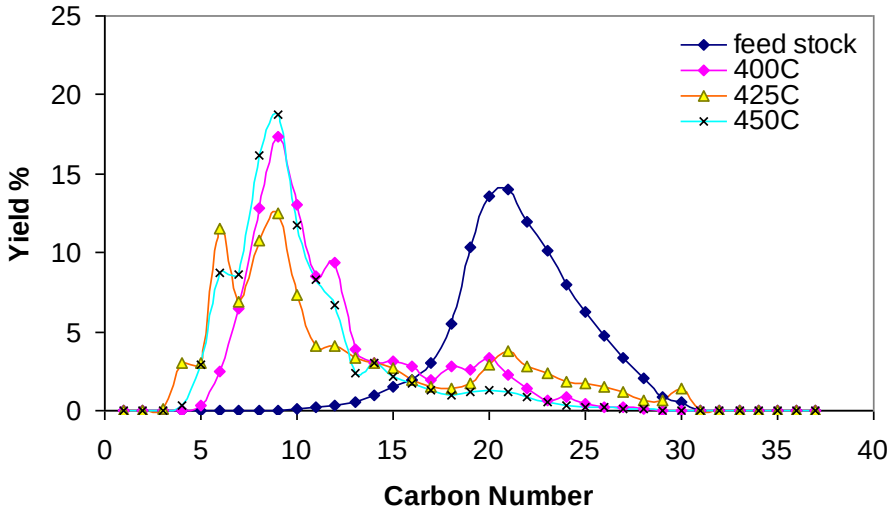

Figure 1: Carbon distribution of feedstock (HGO) and reaction products at different temperatures, and 1 hr reaction time using H- Beta catalyst.

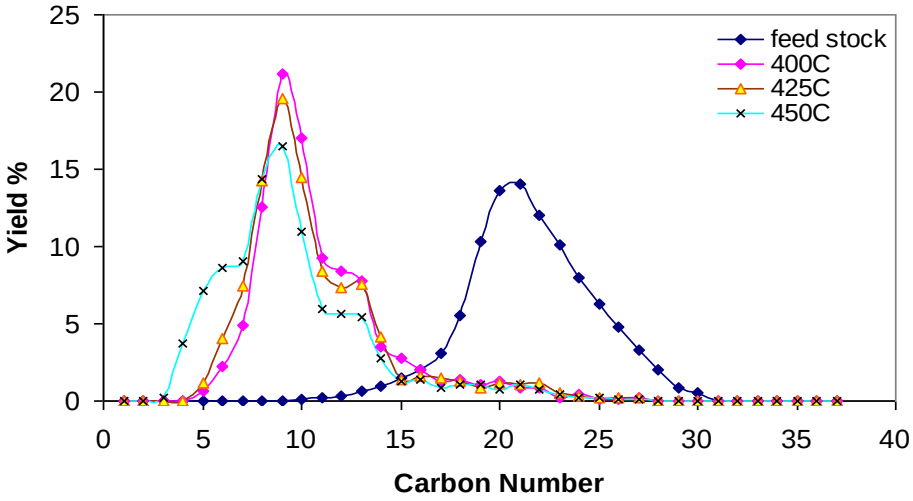

Figure 2: Carbon distribution of feedstock (HGO) and reaction products at different temperatures, and 1 hr reaction time using H-ZSM5 catalyst.

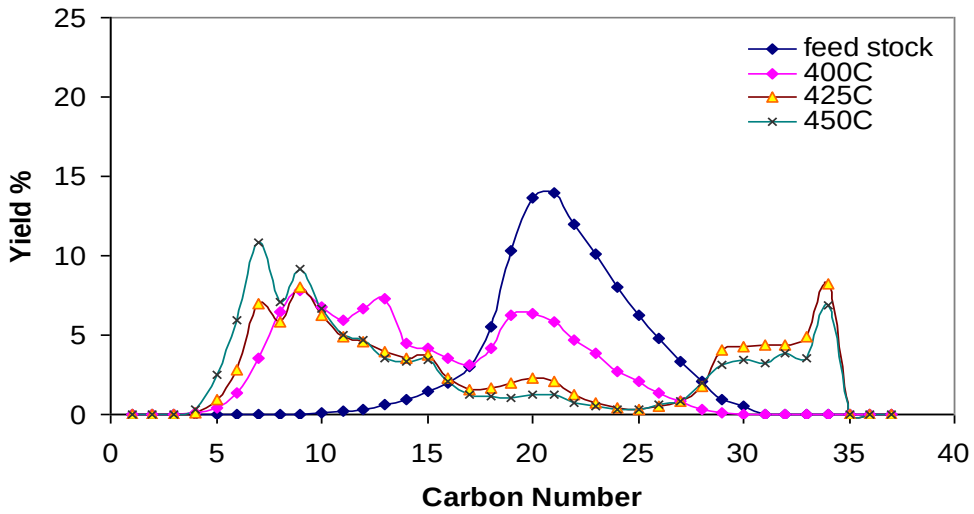

Figure 3: Carbon distribution of feedstock (HGO) and reaction products at different temperatures, and 1 hr reaction time using Mordinite catalyst.

3.4. Simulated Distillation (SIMDIST) Results:

As mentioned earlier that SIMDIST technique was used in the current research to identify the boiling point distribution of the feedstock (HGO) and cracking products at different cracking temperature using H-Beta, H-ZSM5, and Mordinite catalysts.

The results of this test are presented in Figures 4, 5, and 6. In case of H-Beta catalyst, during the cracking reaction the I.B.P was reduced to 64, 70.6 and 50.6^{o}C at cracking temperature varies from 400-450 ^{o}C. The gasoline fraction obtained from the cracking reaction was ranged from (16.06% to 54.88%) at the same cracking temperature. When was used H-ZSM5 as a catalyst, the I.B.P was reduced to 65.3, 56.3 and 50.8 ^{o}C at cracking temperature varies from 400-450 ^{o}C. The gasoline fraction obtained from the cracking reaction was ranged from (24.12% to 51.98%) at the same cracking temperature. When was used Mordinite as a catalyst, the I.B.P was reduced to 73, 53.9 and 50.6 ^{o}C at cracking temperature ranging from 400-450^{o}C. The gasoline fraction obtained from the cracking reaction was ranged from (9.68% to 53.98%) at the same cracking temperature.

However, at 450^{o}C, which is the highest cracking temperature used in this study, the gasoline yields were found 54.88% for HGO/H-Beta, 51.98% for HGO/H-ZSM5, and 53.98% for HGO/Mordinite. It has been found that the

catalysts type have slightly effect on the boiling point distribution of the reaction product whereas the cracking temperature has strong influence on the boiling point distribution.

At 400 and 425ºC, catalytic cracking of HGO/H-ZSM5 gave the highest liquid containing 24.12% and 45.8% gasoline (bp<207ºC), respectively, in case of catalytic cracking of HGO/H-BETA, gasoline yields were 16.06% and 42.23%, at the above mentioned temperatures whilst in case of catalytic cracking of HGO/ Mordinite, gasoline yields were 9.68% and 36.34%, respectively.

HGO/H-ZSM5 produced a much greater amount of material boiling below 450ºC. HGO/H-BETA had less recovery of material boiling below 450ºC than HGO/H-ZSM5, and HGO/ Mordinite. In general, as the temperature increased from 400 to 425ºC, light fractions (bp<180ºC) obtained from cracking increased.

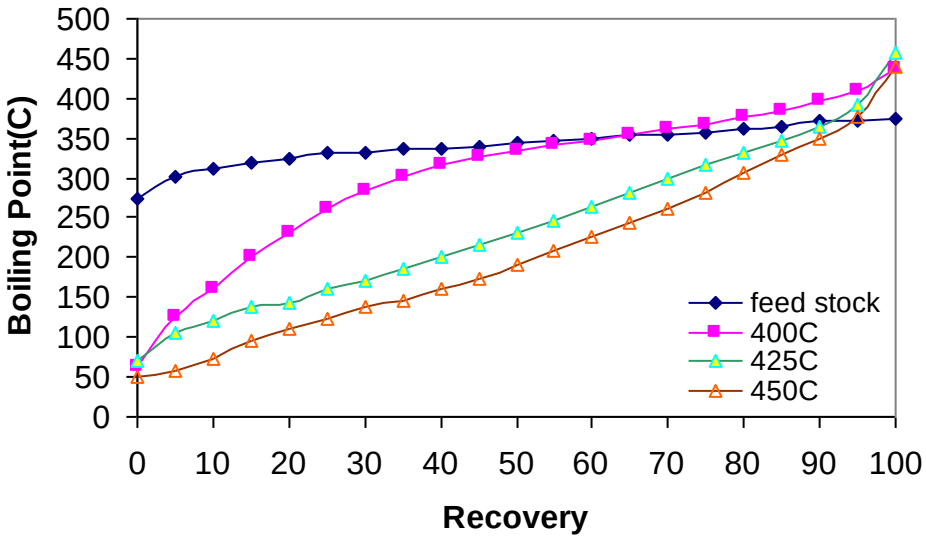

Figure 4: The effect of cracking temperature on the boiling point distribution range of liquid products from HGO cracking using H-Beta catalyst.

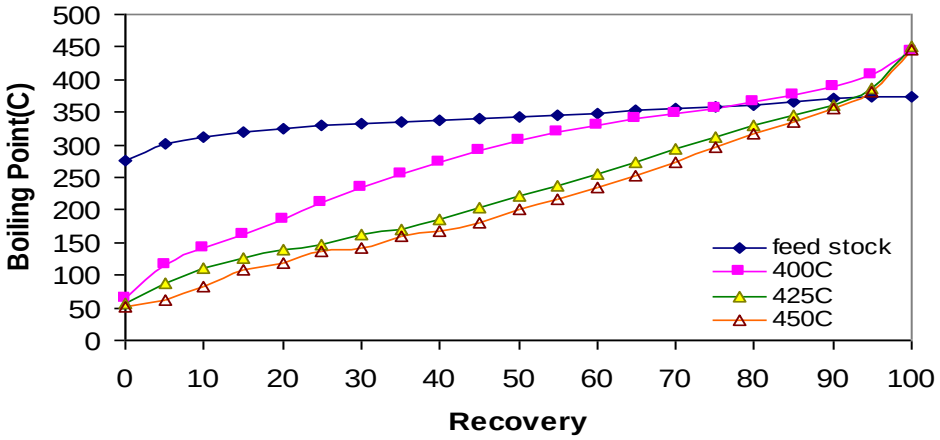

Figure 5: The effect of cracking temperature on the boiling point distribution range of liquid products from HGO cracking using H-ZSM5 catalyst.

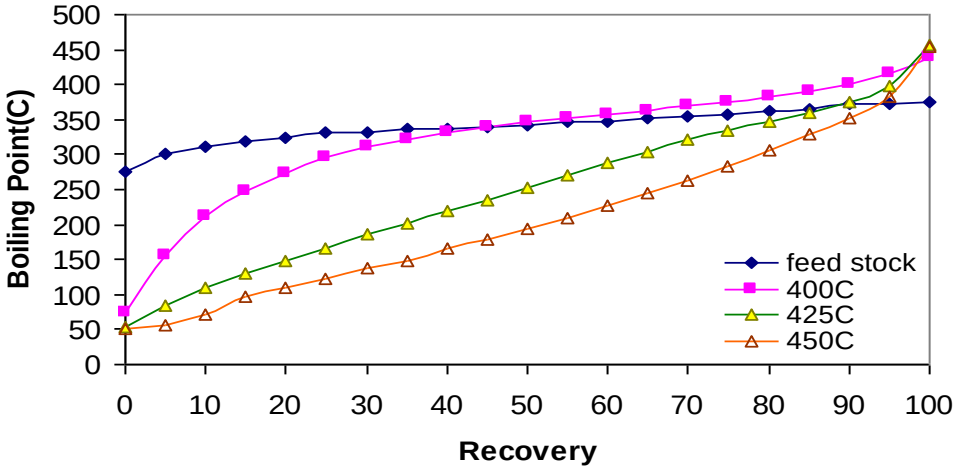

Figure 6: The effect of cracking temperature on the boiling point distribution range of liquid products from HGO cracking using Mordinite catalyst.

3.5. Gas Analysis:

The gas compositions produced from the catalytic cracking of HGO/H-Beta, HGO/H-ZSM5, and HGO/Mordinite at different cracking temperatures are presented in Figures 7, 8, and 9. The gas products from HGO/H-BETA, HGO/H-ZSM5, and HGO/Mordinite consisted of mostly C_3-C_4 hydrocarbons with a small amount of C_5-C_6. Both the cracking temperature and the catalyst type are effective on the composition of gas products. The gas contains the highest percentage of C_3, C_4 when H-ZSM5 (Figure 8) was used as a catalyst, with comparison in case

of H-Beta (Figure 7) and Mordinite (Figure 9) catalysts. Both cracking temperature and catalyst type were found to have strong effect on the composition of gases produced from catalytic cracking of heavy gas oil. C_3, C_4 hydrocarbons increased with decreasing temperature.

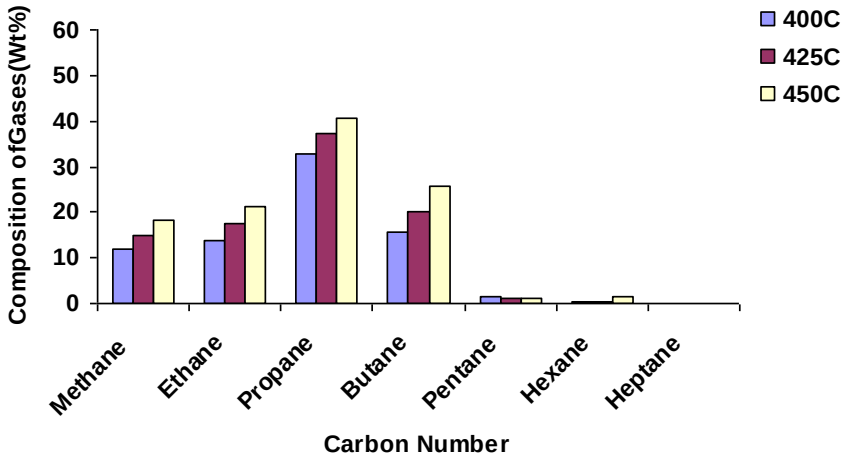

Figure 7: Composition of the gas produced from catalytic cracking of HGO using H-Beta as a catalyst at different cracking temperatures.

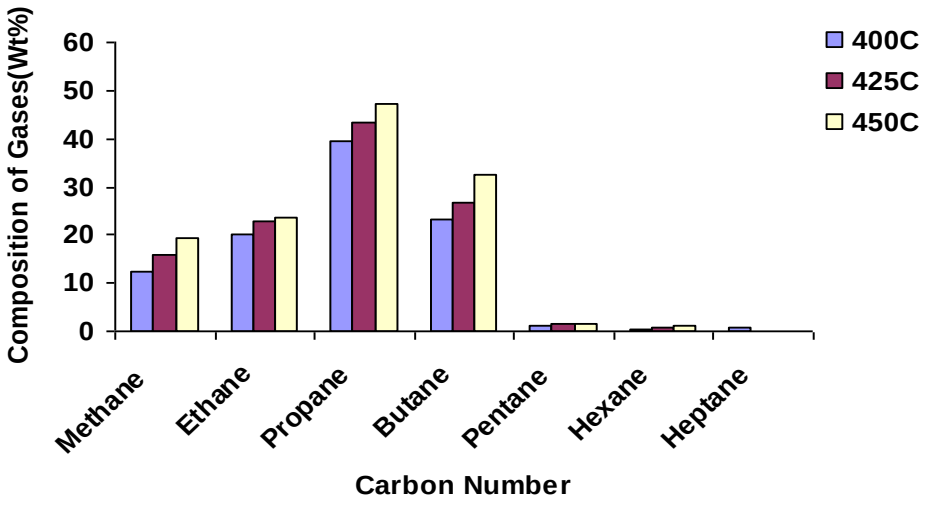

Figure 8: Composition of the gas produced from catalytic cracking of HGO using H-ZSM5 as a catalyst at different cracking temperatures.

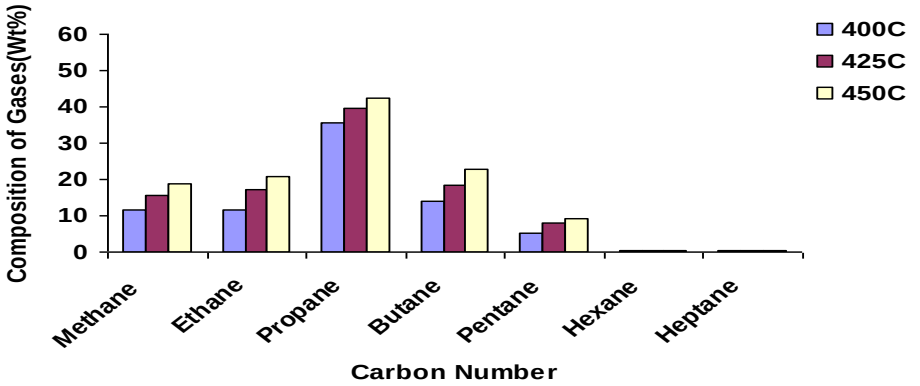

Figure 9: Composition of the gas produced from catalytic cracking of HGO using Mordinite as a catalyst at different cracking temperatures.

3.6. Effects of Cracking Temperature and Catalyst Type on the Product Distribution:

The influence of cracking temperature on the yield of gases, gasoline, 207^{+} distillate and coke are investigated using H-Beta, H-ZSM5, and Mordinite catalysts.

Table 2: Products distribution of catalytic cracking experiments of HGO.

Catalysts	Temperature	Gas	Gasoline	207+	Coke	Conversion
H-BETA	400°C	23.45	21.90	54.53	0.12	45.35
	425°C	25.43	27.45	46.93	0.19	52.88
	450°C	30.17	30.45	39.15	0.23	60.62
H-ZSM5	400°C	26.30	22.94	50.60	0.16	49.24
	425°C	29.86	28.94	40.99	0.21	58.80
	450°C	31.03	30.92	37.77	0.28	61.95
Mordinite	400°C	19.69	21.60	58.60	0.11	41.29
	425°C	23.72	25.66	50.47	0.15	49.38
	450°C	27.57	29.22	42.97	0.24	56.79

Figures 10 and 11 show the effect of cracking temperature on the yield of gases, gasoline, distillate and coke using H-Beta catalyst. It is obvious that as the cracking temperature increase, the yield of gas and gasoline increase but the distillate decrease. The coke obtained from the cracking reaction in Figure 11 was ranged from (0.12% to 0.23%) at the same cracking temperature.

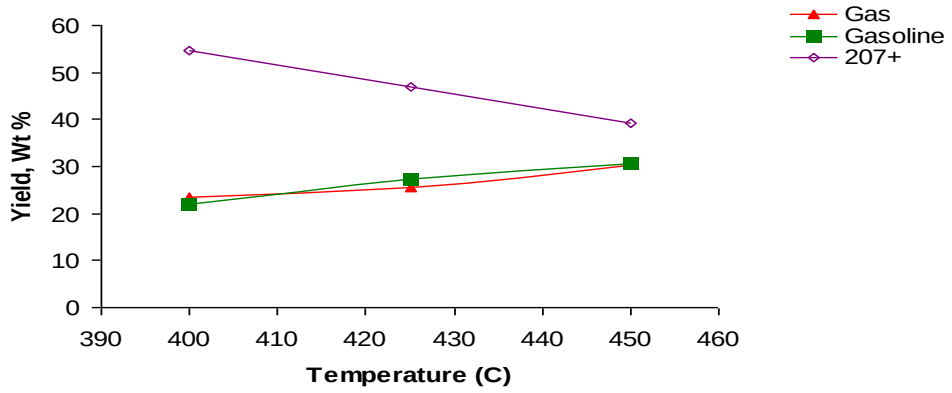

Figure 10: Effect of cracking temperature on the yields from catalytic cracking of HGO over H-Beta catalyst.

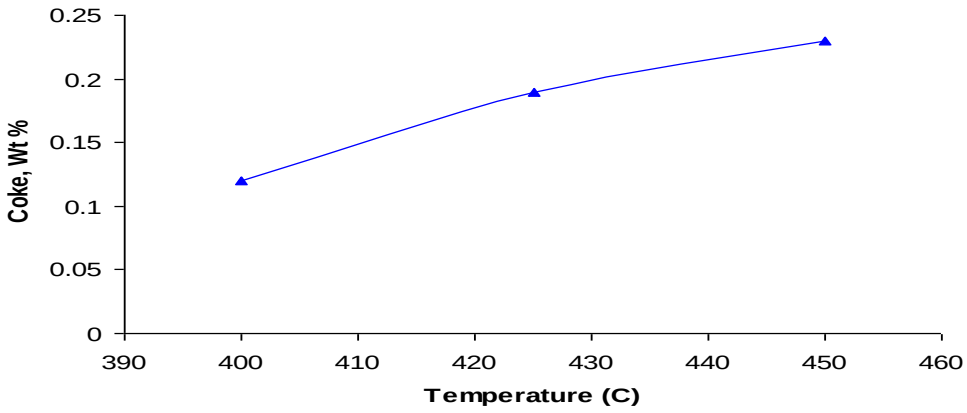

Figure 11: Effect of cracking temperature on percent of coke formed during catalytic cracking of HGO over H-Beta catalyst.

Figures 12 and 13 show the effect of cracking temperature on the yield of gases, gasoline, distillate and coke using H-ZSM5 catalyst. It is obvious that as the cracking temperature increase, the yield of gas and gasoline increase but the

distillate yield (207^{+} ^{o}C) decrease. The coke obtained from the cracking reaction in figure 13 was ranged from (0.16% to 0.28%) at the same cracking temperature.

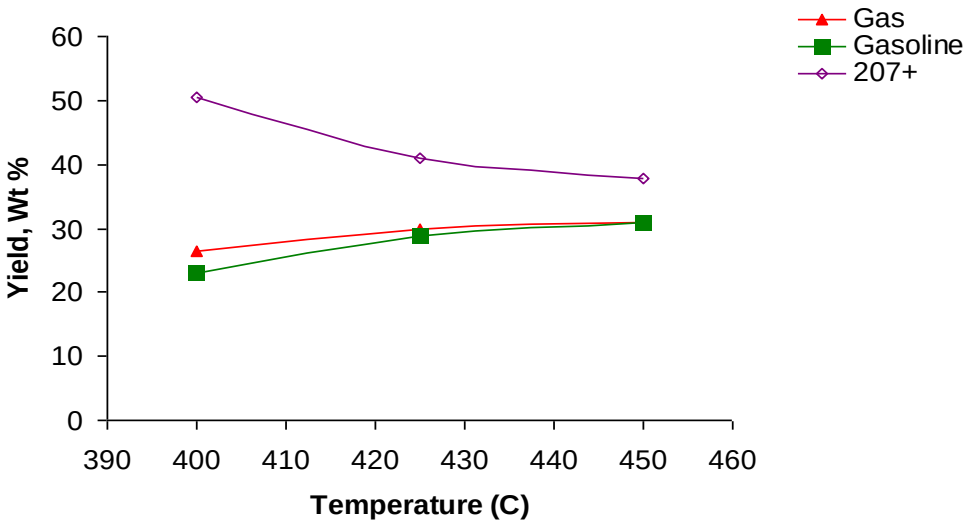

Figure 12: Effect of cracking temperature on the yields from catalytic cracking of HGO over H-ZSM5 catalyst.

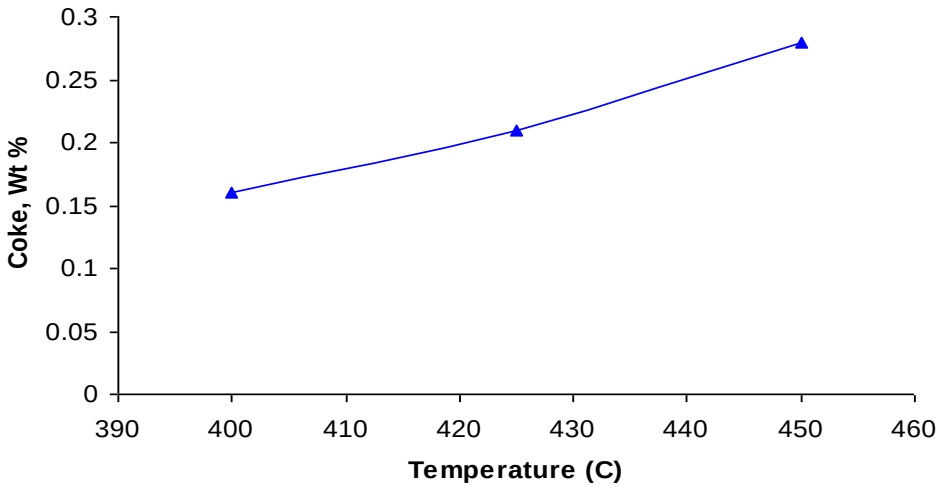

Figure 13: Effect of cracking temperature on percent of coke formed during catalytic cracking of HGO over H-ZSM-5 catalyst.

Figures 14 and 15 show the effect of cracking temperature on the yield of gases, gasoline, distillate and coke using Mordinite catalyst. It is obvious that as

the cracking temperature increase, the yield of gas and gasoline increase but the distillate yield (207^{+} ^{o}C) decrease. The coke obtained from the cracking reaction in figure 15 was ranged from (0.11% to 0.24%) at the same cracking temperature.

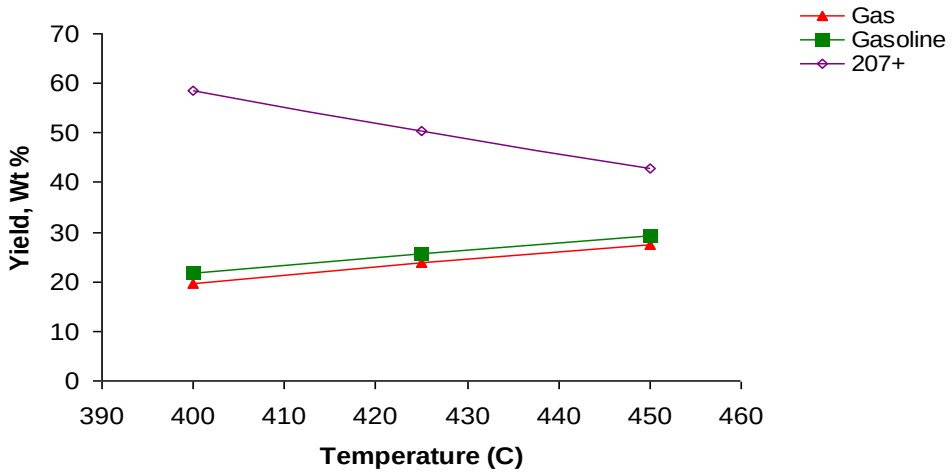

Figure 14: Effect of cracking temperature on the yields from catalytic cracking of HGO over Mordinite catalyst.

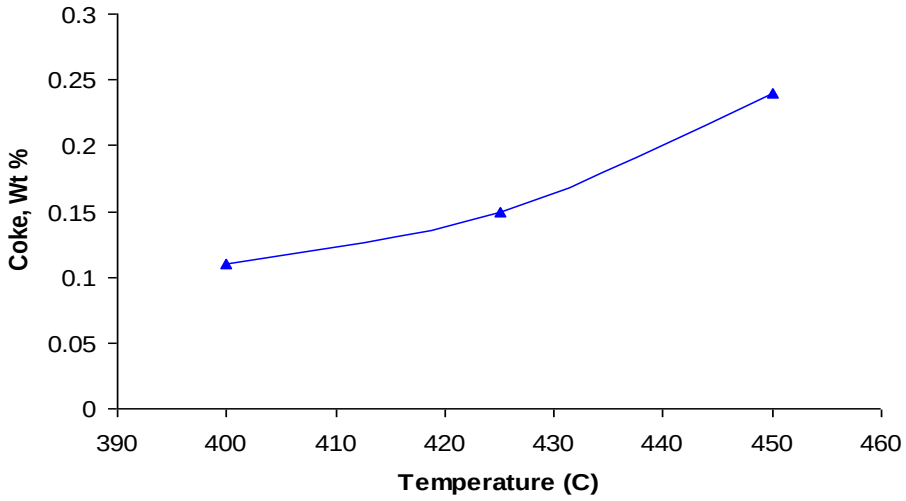

Figure 15: Effect of cracking temperature on percent of coke formed during catalytic cracking of HGO over Mordinite catalyst.

The influence of cracking temperature on the yield of gases and gasoline using H-Beta, H-ZSM5, and Mordinite catalysts are given in Figures 16 and 17. As clear

from the above mentioned figures that H-ZSM5 gives higher yield of gasoline, and gas than H-Beta, and Mordinite catalysts.

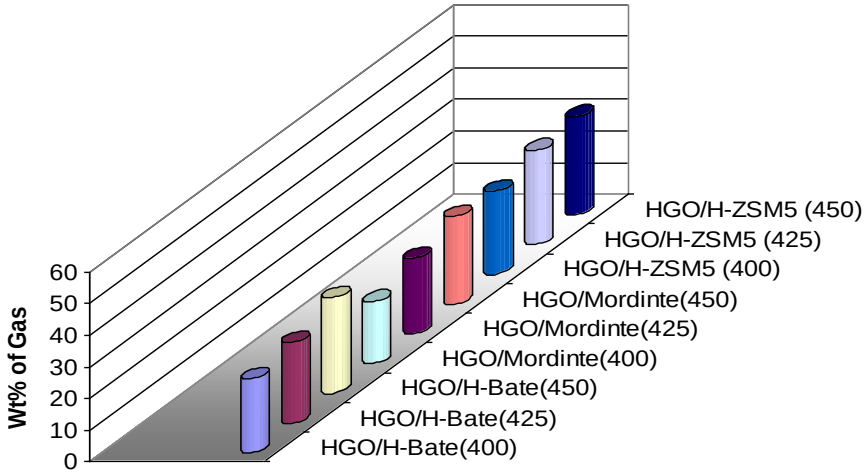

Figure 16: The effect of cracking temperature on the gas yield using H-Beta, H-ZSM5 and Mordinite catalysts.

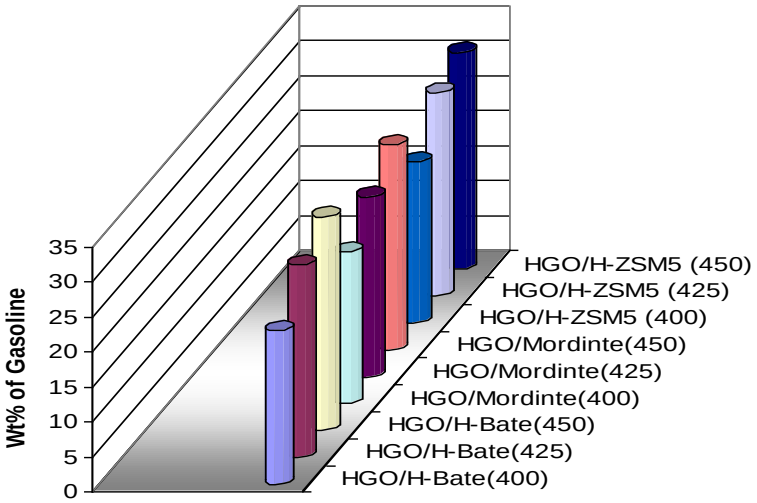

Figure 17: The effect of cracking temperature on the gasoline yield using H-Beta, H-ZSM5, and Mordinite catalysts.

Figure 18 shows the effect of cracking temperature on the yield of 207^{+} ^{o}C using H-Beta, H-ZSM5, and Mordinite catalysts, it is obvious from this figure that the yield of 207^{+} using tested catalysts are identical.

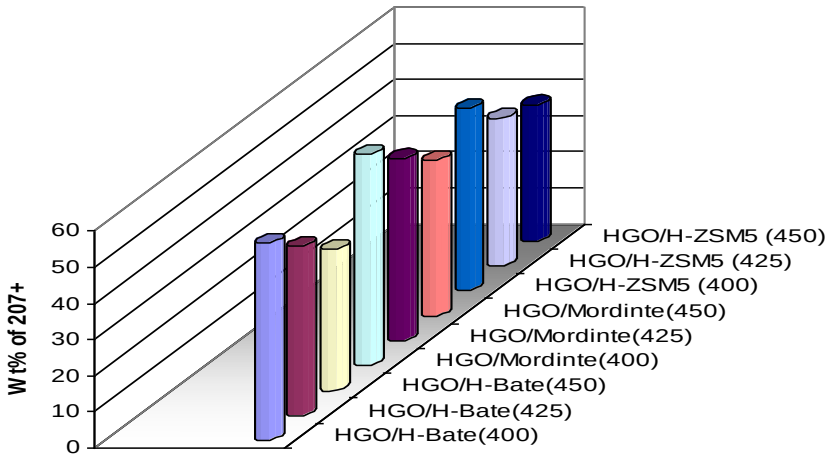

Figure 18: The effect of cracking temperature on the yield of 207^{+} ^{0}C at different cracking temperature using H-ZSM5, H-Beta, and Mordinite catalysts.

Figure 19 shows the effect of cracking temperature on the formation of coke deposited on the surface of H-Beta, H-ZSM5, and Mordinite catalysts, it is clear from this figure that the quantify of coke deposited on the H-ZSM5 catalyst is higher than the coke deposited on H-Beta, and Mordinite catalysts at the cracking temperature studied. This can be explained due to the pore size of the catalysts. The effect of cracking temperature on the conversion using tested catalysts is presented in Figure 20, it is noted that the conversion obtained using tested catalysts are identical.

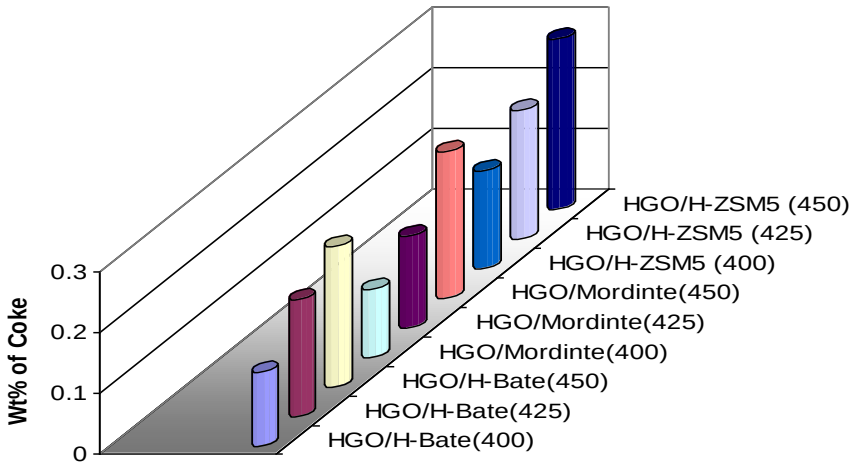

Figure 19: The effect of cracking temperature on the coke using H-Beta, H-ZSM5, and Mordinite catalysts.

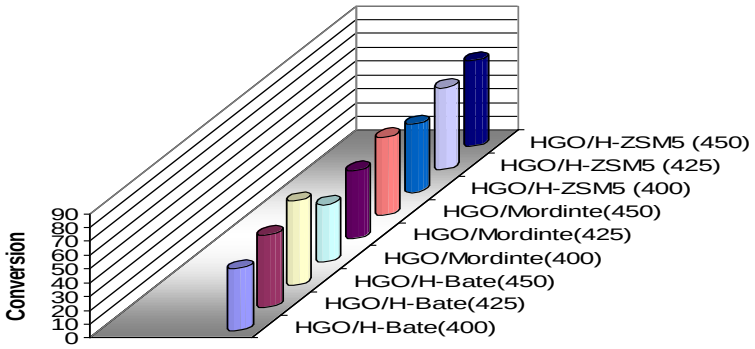

Figure 20: The conversion for catalytic cracking of HGO over H-Beta, H-ZSM5, and Mordinite catalysts.

3.7. Thermal Gravimetric Analysis (TGA) Technique.

This technique was used in this research to quantify the coke deposited on the surface of catalyst. The results of these tests are presented in Figures 21 to 26**.** In this work, the percent of coke formed during cracking reactions of HGO using over tested catalysts has been determined using TGA technique. Figure 21 shows the TGA curves for fresh H-Beta used in this study. Figure 22. shows the TGA

curves for H-Beta at 450ºC cracking temperature with percentage of coke is 30% Figure 23 shows the TGA curves for fresh H-ZSM5and Figure 24 shows the TGA curves for used H-ZSM5 at 450ºC cracking temperature with percentage of coke is 28.6%. Figure 25 shows the TGA curves for fresh Mordinite and figure 26 shows the TGA curves for used Mordinite at 450ºC cracking temperature with percentage of coke is 30.6. The coke deposited on the surface of the catalysts has been calculated based on heat flow chart and mass loss curve ranged from 450 to 550ºC at exothermic reaction.

The combustion of coke deposits in the zeolites shows that the Mordinite zeolite produces more coke than H-Beta, and H-ZSM5. This fact has been attributed to the different pore structure. Table 3 presented coke deposited on the surface of the tested catalyst in this research.

Table 3: The coke deposited on the surface of tested catalysts.

Catalyst	Coke (Wt%)
H-ZSM5	30.6
Mordinite	30.0
H-BETA	28.6

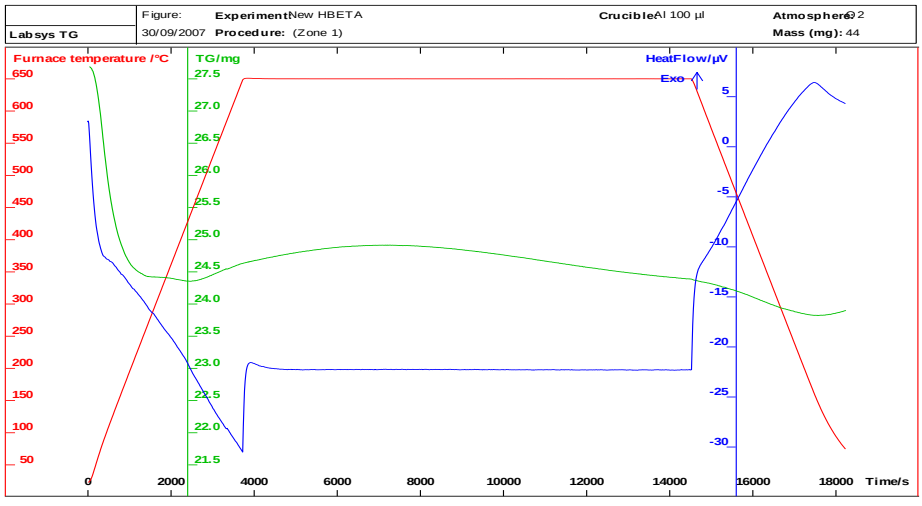

Figure 21: TGA curves of fresh H-Beta.

Catalytic Cracking of Heavy Gas Oil (HGO) Fraction over H-Beta, H-ZSM5 and Mordinite Catalysts

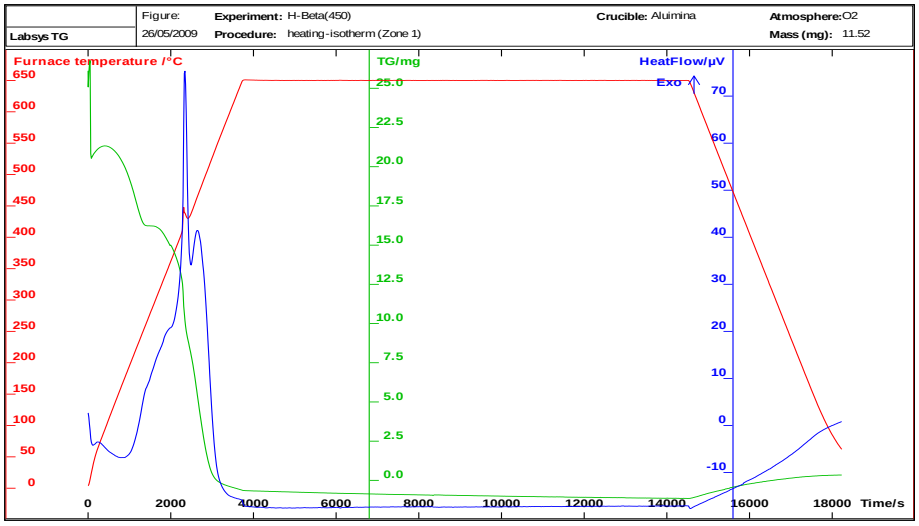

Figure 22: TGA curves of used H-Beta catalyst, cracking temperature (450 °C).

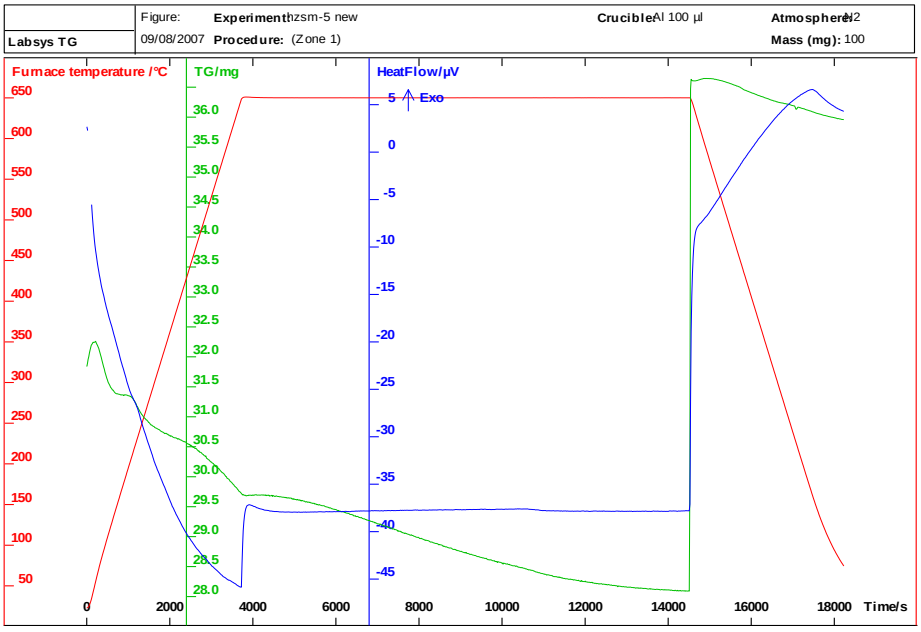

Figure 23: TGA curves of fresh H-ZSM5.

Catalytic Cracking of Heavy Gas Oil (HGO) Fraction over H-Beta, H-ZSM5 and Mordinite Catalysts

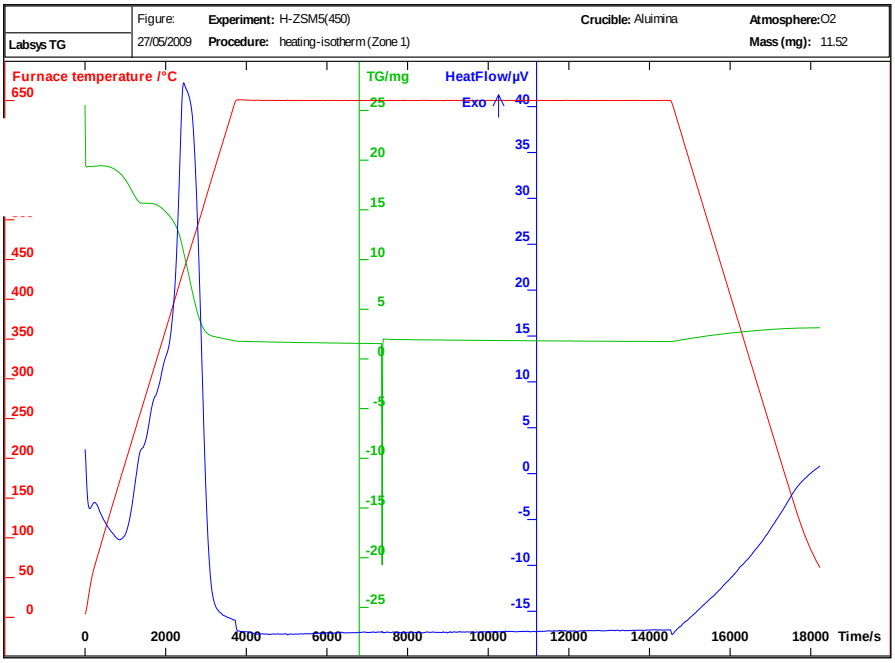

Figure 24: TGA curves of used H-ZSM5 catalyst, cracking temperature (450 ºC)

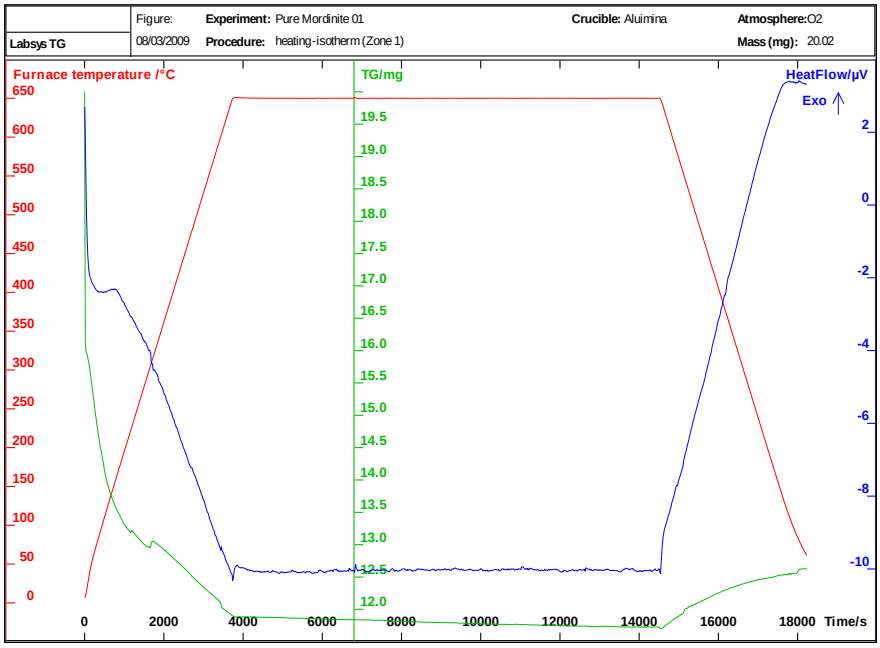

Figure 25: TGA curves of fresh Mordinite.

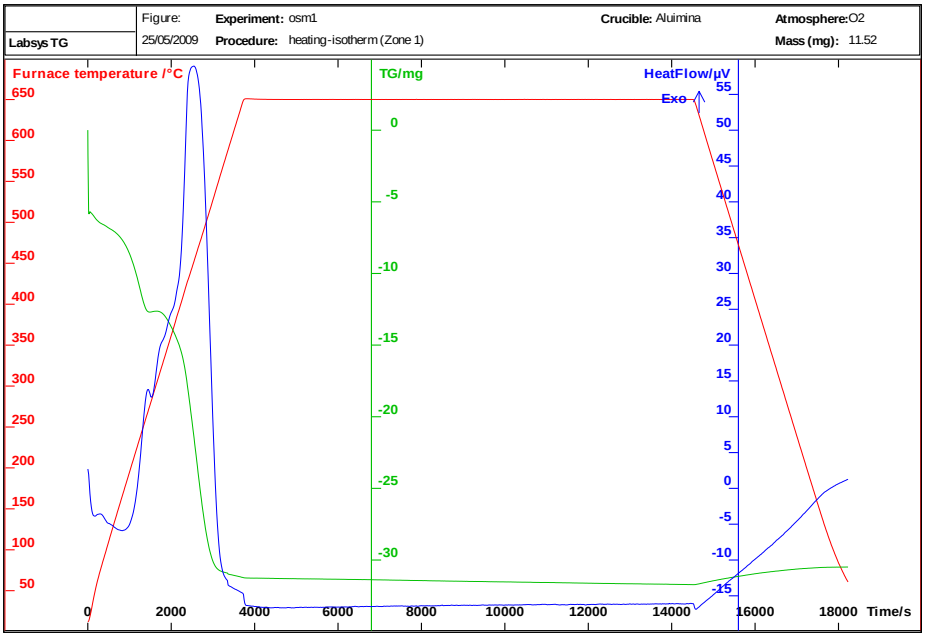

Figure 26: TGA curves of used Mordinite catalyst, Cracking temperature (450 ^{o}C).

4. Conclusions:

In the present study, catalytic cracking of HGO over H-Beta, H-ZSM5, and Mordinite catalysts were investigated.

1. Catalytic cracking of HGO over H-Beta, H-ZSM5, and Mordinite catalysts was successfully catalytically cracked into gases, gasoline and distillate products. SIMDIST, GC and TGA techniques were used to analysis the feedstock and reaction products.
2. Catalytic cracking of HGO in the present of H-ZSM-5 catalyst at 450^{o}C cracking temperature shows the most interesting results with high Selectivity toward gasoline range (33.92 wt%). And the reaction products formed during the catalytic cracking of HGO are formed to be within the carbon distribution ranging from (C_5-C_{12}).
3. Catalytic cracking of HGO was studied increase, the yield of gases; gasoline and coke are increases, whilst the yields of distillate (207+ ^{o}C) are decreases.
4. It has been found that the cracking temperature has strong influence on the conversion and product distribution. When H-Beta was used as a catalyst, it was noted that as the cracking temperature increase from 400-450^{o}C, the liquid yield was decreased from 76 to 69% by weight. On when H-ZSM5 was used as a

catalyst, it was noted that as the cracking temperature increase from 400-450ºC, the liquid yield was decreased from 73 to 68% by weight. On when Mordinite was used as a catalyst, it was noted that as the cracking temperature increase from 400-450ºC, the liquid yield was decreased from 80 to 72% by weight.

5. The results show gas contains the highest percentage of C_3, C_4 when H-ZSM5 was used as a catalyst, with comparison in case of H-Beta and Mordinite catalysts. Both cracking temperature and catalyst type were found to have strong effect on the composition of gases produced from catalytic cracking of heavy gas oil.

6. TGA technique was used to quantify the coke deposited on the surface of H-ZSM5, H-Beta, and Mordinite catalysts. The combustion of coke deposits on the surface of catalyst showed that the H-ZSM-5 catalyst produces more coke than H-Beta, and Mordinite catalysts. This fact has been attributed to the different pore structure.

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الفهـــــــــــرس

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