



Investigating the Influence of the Cerium loading in prepared Y zeolite from Iraqi kaolin on its Catalytic Performance

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ABSTRACT

In this study, the effects of different loading doses of cerium in the prepared NaY zeolite from Iraqi kaolin were investigated. Al-Duara refinery atmospheric residue fluid catalytic cracking was selected as palpation reaction for testing the catalytic activity of cerium loading NaY zeolite. The insertion of cerium in NaY zeolites has been synthesized by simple ion exchange methods. Three samples of modified zeolite Y have been obtained by replacing the sodium ions in the original sample with cerium and the weight percent added are 0.35, 0.64, and 1.06 respectively. The effects of cerium loading to zeolite Y in different weight percent on the cracking catalysts were studied by employing a laboratory fluidized bed reactor. The experiments have been performed with weight hourly space velocity (WHSV) range from 6 to 24 h⁻¹, and the temperature range from 450 to 510 °C.

The activity of the catalyst with 1.06 wt% cerium has been shown to be much greater than that of the sample parent NaY. Also it was observed that the addition of the cerium causes an increase in the thermal stability of the zeolite.

Key words: cerium exchange NaY, rare earth elements, fluid catalytic cracking.

دراسة تأثير تحميل السيريوم في زيوليت Y المحضر من الكاولين العراقي على أدائه التحفيزي

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الخلاصة

في هذه الدراسة. تم بحث تأثير تحميل السيريوم في الزيوليت Y المحضر من الكاولين العراقي. تفاعل التكسير الحفازي المائع لمتبقي التقطير الجوي تم اختياره كمجس لاختبار الفعالية الحفازية للزيوليت المحسن والمحضر نوع NaY. الزيوليت المطور تم تصنيعه بطريقة التبادل الايوني البسيط. ثلاثة نماذج من الزيوليت المطور تم الحصول عليها عن طريق استبدال ايون الصوديوم في النموذج الاصلي بالسيريوم وبالنسب الوزنية المئوية التالية 0.35 ، 0.64 ، 1.06 تأثير تحميل السيريوم للزيوليت وبنسب وزنية مختلفة على الفعالية التفسيرية تم بحثه باستخدام منظومه مختبريه تجريبية لمفاعل الطبقة المميعة. التجارب المختبرية اجريت عند سرع وزنية فراغيه بين 6 الى 24 ساعة⁻¹ ودرجة حرارة من 450 الى 510 درجة مئوية.

الفعالية الحفازية للعامل المساعد المحتوي على نسبة وزنية من السيريوم بمقدار 1.06 كانت اكبر بكثير من الفعالية الحفازية للعامل المساعد الاصلي. وكذلك تم ملاحظة ان اضافة السيريوم ادت الى زيادة الثباتية الحرارية للزيوليت. **الكلمات الرئيسية:** استبدال السيريوم بالزيولات NaY، عناصر الارض النادرة، التكسير الحفازي المائع.



1. INTRODUCTION

The atmospheric distillation residue oil is gained as a byproduct from the refineries through atmospheric distillation of crude oil to produce light hydrocarbon fraction **Jakob and Peter, 1977**. Approximately half of the crude oil leaving the behind atmospheric distillation is the atmospheric distillation residue. That it is a necessary to find a possible economic process to convert low value product such as residual distillation to high value products such as gasoline and middle distillate, **Pappal, et al., 2003, Gray, 1994.**

The important process in the refinery industry to convert heavy fraction low value product of the crude oil into an assortment of high value, light products is the fluid catalytic cracking (FCC). The operation conditions of this process are preferable achieved with a pressure 1 to 10 bar, weight hour space velocity (WHSV) from 3 to 200 h⁻¹, and temperature from 400 to 550 °C, **Chen, and Cao, 2005**. Fluid catalytic cracking process produces a raw material for many petrochemical processes such as light olefins increasing the its importance, in addition to its main purpose of producing of diesel fuel and gasoline, **Biswas and Maxwell, 1990**

The FCC catalyst is a key point in the optimization of FCC unit. Basically, the FCC catalysts could be divided into three main categories based on the refineries objective i) maximization of valuable product, ii) improving the gasoline quality and, iii) minimization of the residue. In order to achieve those goals, the FCC catalyst has two components, the zeolite and the matrix. Besides these components the catalyst can also have other functional ingredients and or /additives for specified function like enhancing gasoline octane, upgrading the conversion of residue feedstock, enhancing the resistance to metallic poisons, promoting the CO combustion, reduction the NO_x and SO_x emission, among there, **Costa, et al., 2004**.

In the recent years the replacement of rare earth zeolite has become more interesting as stable solid acid catalyst due to the high thermal stability **Magee, et al., 2002**. Modifying zeolites Y by ion exchange of commutable cations provides a beneficial properties to particular application which become an integral portion of the manufacture process catalyst. The performance of cracking catalyst such as catalyst selectivity, hydrothermal stability, catalyst activity has been enhanced when rare earth element was introduced. The faujasite Y zeolites modified process can be achieved by replacing the native sodium ions with rare-earth ions. For example Ce NaY, LaNaY plays an significant part in the production of FCC catalysts, **Nery, et al., 1997 and Thomas, et al., 2006**.

The sodium ions in the zeolite crystal exchange with rare earth element, such as praseodymium, cerium and lanthanum being trivalent which make "bridges" among three or two acid sites in the framework of zeolite, these bridges stabilizes the structure of zeolite and protect acid sites from ejection. The hydrothermal and thermal and activities of zeolite increases with exchange rare earth components due to the "bridge" stabilizes and prevents aluminum atoms from dissociation from the zeolite structure whenever the catalyst exposure to high temperature in the reactor and regenerator. The rare earth element is generally ion exchange with NaY zeolite, **Sadeghbeigi, 2000**.

The aim of present study is to modify the zeolite Na Y prepared from Iraqi kaolin with various weight percents cerium exchange. Also the activity of the resulting catalyst were evaluated by employing a laboratory fluidized bed reactor by using atmospheric distillation residue as a feed, and the influence of the addition of cerium on the thermal stability of the NaY was investigated.



2. EXPERIMENTAL

2.1 Feedstock

Atmospheric residue with boiling range 278°C to 450°C was supplied from atmospheric distillation unit of Al-Duara refinery as a raw material for the catalytic performance of the zeolites synthesized, and its detailed properties are listed in **Table 1**.

2.2 Catalyst

Parent NaY zeolite were prepared from Iraqi kaolin as follows: Kaolin was finely divided to powder and mixed with sodium hydroxide solution was prepared at concentration 45% wt with weight ratio $\left(\frac{\text{Kaolin}}{\text{pureNaOH}} = \frac{1}{1.5}\right)$. The mixture fused at 850°C by programmable electrical furnace, then ten grams of powder resulting mixed with twelve point sixty seven gram of sodium silicate and disseminate in one hundred and fifty milliliter of deionized water with stirring for one hour. The resultant slurry which has pH 13.6 was subjected to aging at 50 °C for 24 hr in a programmable electrical furnace, then the gel slurry was subjected to hydrothermal crystallization at 100 °C for 48 hr in the same furnace. After that the slurry was filtrated to separate the precipitate from mother liquid. The crystalline precipitate washed with deionized water to reduce a pH to 11.5. After that the slurry was filtrated to separate the precipitate from mother liquid. The precipitate subjected to dry at one hundred centigrade for sixteen hours. The dried powder was activated by calcinations in a programmable electrical furnace at five hundred centigrade for one hour. **Htay and Oo, 2008**

2.3 Modified Zeolite Procedure.

Cerium exchanged was obtained by contacting Na-Y with 0.5M cerium nitrate solution (0.025 moles of cerium nitrate / g of zeolite) at 80 °C for 24 hours ,then filtrate, and was washed to obtained 0. 35 wt% Ce Na-Y . This exchange cycle was repeated two times with fresh cerium nitrate to obtain 0.64 wt% CeNa-Y. Na-Y was mixed with fresh 0.5M cerium nitrate solution four times to obtained 1.06 wt% Ce Na-Y. All samples were calcined after each exchange at a temperature of 150 to 500 °C with heating rate 12 °C /minute and at 500 °C for 5 hours **Thomas, et al., 2006**. The degree of cerium exchange in the zeolite was determined by the analysis of each zeolite sample to determine the sodium weight percent in the starting and those remaining after the cerium exchange. The sodium weight percent loss converted to equivalent cerium

2.4 Catalytic activity test

Catalytic Cracking experiments achieved by employing a laboratory fluidized bed reactor, , shown in **Fig. 1**. This unit was found in the laboratories of Chemical Engineering Department, Baghdad University, it includes four parts: water and atmospheric residue pumping system, catalytic cracking reaction, control of temperature system, and separation and collection system.

Water pumped preheater at specified flow rate; the water was converted to steam by heating system. Steam was used to remove the air from reactor. After that atmospheric residue was pumped to a pre-heater to increase temperature before reaching catalytic reactor



which reactions was occurred. The products from reactor cooled, and then separated to gas and liquid products. Cracking liquid product was transferred to distillation unit for separation catalytic cracking gasoline from heavy stock. The chemical composition of the gases and catalytic cracking gasoline was determined by using gas chromatography.

Coke content on catalysts was measured by the following method. A sample of catalyst from reactor was weighed and dried by electrical programmable furnace at 100 °C for 16 hr. The dry sample was weighed and returned to electrical programmable furnace to restore catalyst activity by burning off coke deposition on spent catalyst at 650 °C for one hour. The calcined sample after burning coke was weighed. The differences between weight 1 and weight 2 represents moisture and difference between weight 2 and weight 3 represents delta coke. Delta coke is defined as the difference between coke on the spent catalyst after stripping and on the regenerated catalyst. The catalyst activity was determined by the weight percent of feedstock was converted to products ,the products included gas ,gasoline ,and coke. The part of liquid product which has a boiling point between ambient temperatures and 220 °C was represented a gasoline.

2.5 Operating Conditions

The experiments of catalytic cracking of atmospheric residue were achieved on parent NaY zeolite and modified zeolite catalysts sampled in the laboratory fluidized bed reactor system. Operating conditions were catalyst weight hourly space velocity was 6 , 12 ,18,and 24 h⁻¹ , reaction temperature was 450 ,470,490,and 510 °C. The effect of regeneration number (it refers to restoring catalyst activity after each complete reaction) on the catalyst activity of the parent NaY zeolite and modified zeolite(1.06wt%) was carried out at 510 °C, WHSV= 6 h⁻¹ , and atmospheric pressure.

2.6 Analytical Apparatus

The digital flame analyzer by flame photometer Gallen Kamp in The State Company for Geological Survey and Mining was used to determine the sodium content the parent catalyst and modified catalyst after cerium ion exchange.

The gas chromatograph Agilent Technologies 6890N equipped with flame ionization detector (FID) was used to determine the composition of the gases produced from experiment. The volume data converted to mass data by using equation of state for ideal gases.

The composition of cracked gasoline measured by using simulated distillation gas-chromatography Agilent Technologies 6890N .

Chemical analysis according UOP Method 172- 59 was used to measure the concentration of the hydrogen sulfide.

Gas Chromatograph 373 GASUKURD KDGYO was used to determined the concentration hydrogen gas in the gases mixture produced. All above device was located in Al- Duara Refinery.

3. RESULT & DISCUSSION

3.1 The Effect of Cerium Exchange on the Atmospheric Residue Conversion.

The effects of cerium exchange and WHSV on the atmospheric residue conversion were studied at different reaction temperatures. **Fig. 2** and **Table 2** show the dependence of conversion upon cerium weight percent loading and catalyst hour space velocity when catalytic cracking was over zeolite catalysts containing different weight percent cerium.

At a given catalyst hour space velocity with constant temperature, the zeolite catalysts containing more cerium weight percent loading show greater conversion of atmospheric residue and gives more products (gasoline, gases, and coke) in comparison with the parent Na-Y zeolite (fig. 2 and table 2). This means that the cerium indeed contributes to the conversion, which may be explained as follows: the sodium ion reduced the acidity of zeolite then reduced the activity of zeolite due to that the residual sodium ion has a poisoning effect on the acidity especially Bronsted type of faujasite zeolites. Low acid amount of Na-Y zeolite might reduce catalyst activity, which is the crucial step in the formation of the desired product.

The total acid amount increased after introducing rare elements (RE) to zeolite sample. There were two possible reasons for the result. Firstly, the hydrothermal stability had been strengthened on account of the addition of the rare elements (RE) cation, which coordinated with the oxygen atoms in the pore channel. Therefore, the dealumination of the framework was restrained during the hydrothermal disposal. So the loss of acidic amounts would reduce. Secondly, silicon hydroxyl and aluminum hydroxyl in the zeolite framework were polarized due to the insertion of the RE cation. Consequently the electron density increases in the framework of zeolite, appeared vigorous acidity of the acidic center. And because of the existence of an empty f orbit RE^{3+} , the amount of Lewis acid would also increase, **Xiaoning, et al., 2007**. The zeolite activity comes from these acid sites, **Hayward, and Winkler, 1990**. From the above the increasing cerium exchanged decreases Na^+ ion in the zeolites and gives more conversion of atmospheric distillation residue.

3.2 The Effect of WHSV on the Atmospheric Residue Conversion.

At a constant temperature with a given cerium weight percent loading **Fig. 3** and **table 2** shows that the atmospheric residue conversion increases with decreasing of WHSV. This means that the conversion of reduced crude is a function of reaction time for all catalysts, when the contact time between particle of catalyst and the feed molecules increases the atmospheric residue conversion increasing in direct proportion to the amount of the catalyst and inversely proportional to the feed flow rate. These results are a good agreement with those obtained by **Ancheyta and Sotelo, 2002, Wallenstein, et al., 2002** about the effect of WHSV on the atmospheric distillation residue conversion, gasoline, gases, and delta coke yield.

3.3 The Effect of Temperature on the Atmospheric Residue Conversion.

At a given catalyst hour space velocity with constant cerium weight percent loading **Fig. 2** and **Table 2** show that the atmospheric residue conversion increases with increasing the temperature. This may be imputed to the intermolecular motions accelerated with the increasing of temperature contributing to the conversion of the reactants to new products and consequently increases the chemical reaction rate. Also the increasing of temperature accelerates thermal activation which conjuncts with catalytic activation to increase the conversion of atmospheric residue. The feed molecules are better vaporized at the higher temperature that decreased the coke formation from condensation reactions which occurred when poor feed molecules vaporization at low temperature. The diffusion of feed molecules increased at coke decreasing and high temperature which consequently increases the feed conversion as mentioned by **Decroocq, 1984**. These results are a good agreement with those obtained by **Lan, et al., 2009** about the effect of temperature on the atmospheric distillation residue conversion, gasoline, gases, and delta coke yield.

3.4 The Effect of Cerium Exchange on the Gasoline Yield.

Fig. 4 and **Table 2** show that the gasoline produced from the different catalysts when cracking atmospheric residue depends on the conversion, when the catalysts contain more cerium weight percent loading it gives a higher gasoline yield. Also increasing temperatures and decreasing weight hours space velocity shows more gasoline yield obtained. **Table 3** shows chemical composition of cracked gasoline for parent NaY and 1.02 CeNa. This table shows that olefins and naphthenes decrease while paraffins and aromatics increases for 1.06 CeNaY with respect to the parent NaY, due to the hydrogen transfer reactions increases with increasing cerium weight percent loading. In this reaction two molecules reacted (bimolecular reaction) one of them is olefin and the another either naphthenes or also olefin. In case of both molecules reacted being olefin, the reaction is chain reaction and ultimately converts olefins molecules to paraffin's and aromatics molecules. In the reacted olefins molecules with naphthenes molecules, this reaction produces aromatics and paraffin's.

In comparing **Fig. 2 and 4** for the catalyst 1.06 CeNaY at 6 WHSV the reduced crude conversion increases with increasing of temperature at constant WHSV within the limit of study, while, the gasoline yield increases with conversion, with a tendency to reach a maximum (at 490 °C) and after that the gasoline yield remain approximately constant. This is due to the fact that gasoline yield undergoes the secondary cracking (over cracking) to gaseous products and coke.

From **Table 2** the gases yields and coke yield are dependent on conversion when catalytic cracking of atmospheric residue over different catalysts. This table shows that the coke yields and the gases yield as function of the cerium weight percent loading, temperatures and weight hours space velocity. The composition of the gas produced at a conversion of 82.3 wt % (as a sample), is shown in **Table 4**. The above result agreement with the results obtained by **Al-Khattaf, 2002, Ancheyta and Sotelo, 2002** about the relation between WHSV and gasoline yield with occur the phenomenon of secondary cracking (over cracking) of gasoline to gaseous products and coke

3.5 The Effect of Cerium Exchange on the Thermal Stability.

Fig. 5 shows the relation between the conversion of atmospheric residue and the number of regeneration for NaY and 1.06CeNaY catalysts. From this figure the difference in the activity between NaY and 1.06 CeNaY for zero regeneration (fresh catalysts) was about 15 wt%. For first regeneration it was about 37wt%, while for second regeneration it was about 43wt%. This means that the catalyst 1.06 CeNaY decreases the activity less than the catalyst NaY due to the fact that the sodium ion reduces the zeolite hydrothermal stability and reacts with acid sites of zeolite to decrease the activity of catalyst, **Suchuchchai, 2004**. In the regenerator the temperature increases which causes the mobility of sodium and it's neutralizes the stronger acid sites, **Scherzer, 1990**, while the cerium is insensitive to the temperature increase, its immobile component, **Lottermoser, 1992**.

To explain this positive effect of the RE elements on zeolites thermal stability, two aspects may be considered: (i) cation valence, and (ii) formation of RE–O–RE bonds in zeolite cavities. the divalent cations have a more stabilizing influence than monovalent cations, the exchanged trivalent cations with zeolites gives the more stable ones. Moreover, the enhancement on thermal stability has also been attributed to the existence of RE–O–RE bonds in the interior of sodalite cavities, which form bridges with the zeolite tetrahedra, stabilizing the structure, **Trigueiro, et al., 2002**.



4. CONCLUSION

- 1- The Na–Y modified with cerium increased cracking activity, which show an increasing the gasoline, gas ,and coke yields with increasing cerium weight percent.
- 2-The secondary cracking of gasoline to gaseous products and coke occur at 6 WHSV and 490°C
- 3- Cerium substituted zeolite show far best stability toward reaction they lost 12.1 % of the initial activity in 1.06CeNaY whereas, NaY lost 33.6% activity with same condition (6WHSV,510°C) for first regeneration and 21.2 % to 1.06 CeNaY, 50.2% to NaY respectively for second regeneration .

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Table 1. Atmospheric residue Properties.

Density (20 °C ,gm / cm ³)	0.908
Molecular weight	486
Carbon residue(wt%)	4.67
ASTM distillation (°C)	
IBP	278
10%	291
30%	352
50%	396
70%	417
90%	450

Table 3. A comparison between the chemical compositions of cracked gasoline at 6 WHSV and 490 °C.

Chemical Composition of cracked gasoline(vol. %)	NaY (C ₅ -220°C)	1.02 CeNaY (C ₅ -220°C)
Paraffins	19.4	27.8
Naphthenes	5.7	3.2
Olefins	40.1	32.3
Aromatics	34.8	36.9

Table 4. Chemical analysis of gases at 6 WHSV and 510°C.

Gases	Yield(wt%)from the total product
Hydrogen	0.5375
Hydrogen sulfide	0.7485
Methane	2.4820
Ethane	2.8345
Ethylene	2.0677
Propane	4.3310
Propylene	3.4025
Iso Butane	2.5930
N. Butene	3.4835
N. Butane	3.2690
Iso Pentane	0.4813
N. Pentane	0.1695

Table 2. Cracking experiments results of the atmospheric residue at different cerium weight percent loading.

Catalyst type	Reaction temperature	WHSV (h ⁻¹)	Conversion	Gasoline(wt%)	Gases(wt%)	Coke(wt%)	Heavy oil(wt%)
NaY	510	24	46.2	20.1	16.0	10.1	53.8
	490		43.8	19.3	15.0	9.5	56.2
	470		42.3	19.1	14	9.2	57.7
	450		41.4	18.3	14.2	8.9	58.6
	510	18	53.7	23.5	18.1	12.1	43.3
	490		52.4	23.1	17.3	12	44.6
	470		50.8	22.2	17.1	11.4	46.2
	450		48.6	21.4	16.2	11	47.4
	510	12	61.9	29.2	18.7	14.1	38.1
	490		59.8	28.4	18.1	13.5	39.2
	470		58.4	28.1	17.9	12.4	41.6



	450	6	55.9	27.1	17.3	11.5	44.1	
	510		70.2	33.2	21	16	29,8	
	490		68.7	32.4	20.1	15.2	31.3	
	470		65.1	31.0	19.1	15	33.9	
	450		63.2	30.0	19	14.2	36.8	
0.35Ce NaY	510	24	50	24.9	15.1	10	50	
	490		47.2	24.1	14	9.1	51.8	
	470		45.7	23.0	13.3	8.4	54.3	
	450		43.3	22.0	13.1	8.2	56.7	
	510	18	57.1	28.2	16.9	13	42.9	
	490		55.8	27.8	16.5	12.5	44.2	
	470		54.1	26.9	16.2	11	45.9	
	450		51.6	26.2	15.3	11.1	48.4	
	510	12	64.9	31.2	19.9	13.8	35.1	
	490		63.1	30.3	19.1	13.7	36.9	
	470		60.8	29.1	18.5	13.2	39.2	
	450		59.2	28.3	17.9	13.0	40.8	
	510	6	74.1	34.9	23.1	16.1	25.9	
	490		71.9	33.8	22.3	15.8	28.1	
	470		70.2	33.0	21.9	15.3	29.8	
	450		67.5	32.8	20.7	14.0	32.5	
	0.64Ce NaY	510	24	54.3	24.2	18.0	11.1	45.7
		490		50.5	23.0	17.3	10.2	49.5
		470		48.1	22.2	16.4	9.5	51.9
		450		46.4	21.6	15.7	9.1	53.6
		510	18	60.8	27.5	20.1	13.2	39.2
		490		59.5	27.1	19.6	12.8	40.5
		470		57.3	26.2	19.0	12.1	42.7
		450		55.6	25.7	18.4	11.5	54.4
510		12	68.7	31.2	22.4	15.1	31.3	
490			66.4	30.1	21.5	14.8	33.6	
470			64.6	29.4	21.0	14.2	35.4	
450			61.1	28.2	20.3	13.6	37.9	
510		6	77.6	36.2	24.0	17.4	22.4	
490			75.8	35.4	23.5	16.9	24.2	
470			73.9	34.6	23.1	16.2	26.1	
450			70.5	32.4	22.2	15.9	29.5	
1.06Ce NaY		510	24	58.8	29.1	18.4	11.3	41.2
		490		55.2	27..2	17.1	10.9	44.8
		470		53.6	26.7	16.5	10.4	46.4
		450		51.6	25.8	16.1	9.7	48.4
		510	18	65.8	32.3	20.4	13.1	34.2
		490		63.5	31.2	20.0	12.3	36.5
		470		61.7	30.4	19.5	11.8	38.3
		450		60.1	30.0	18.8	11.3	39.9
	510	12	73.4	35.3	23.0	15.1	26.6	
	490		70.6	34.2	22..4	14.0	29.4	
	470		68.9	33.3	21.9	13.7	31.1	

	450		66.0	31.8	21.1	13.1	33.7
	510	6	82.3	37.1	26.4	18.8	17.7
	490		80.6	37.2	25.6	17.9	19.4
	470		77.8	36.5	24.1	17.3	22.2
	450		75.5	35.1	23.2	16.2	24.5

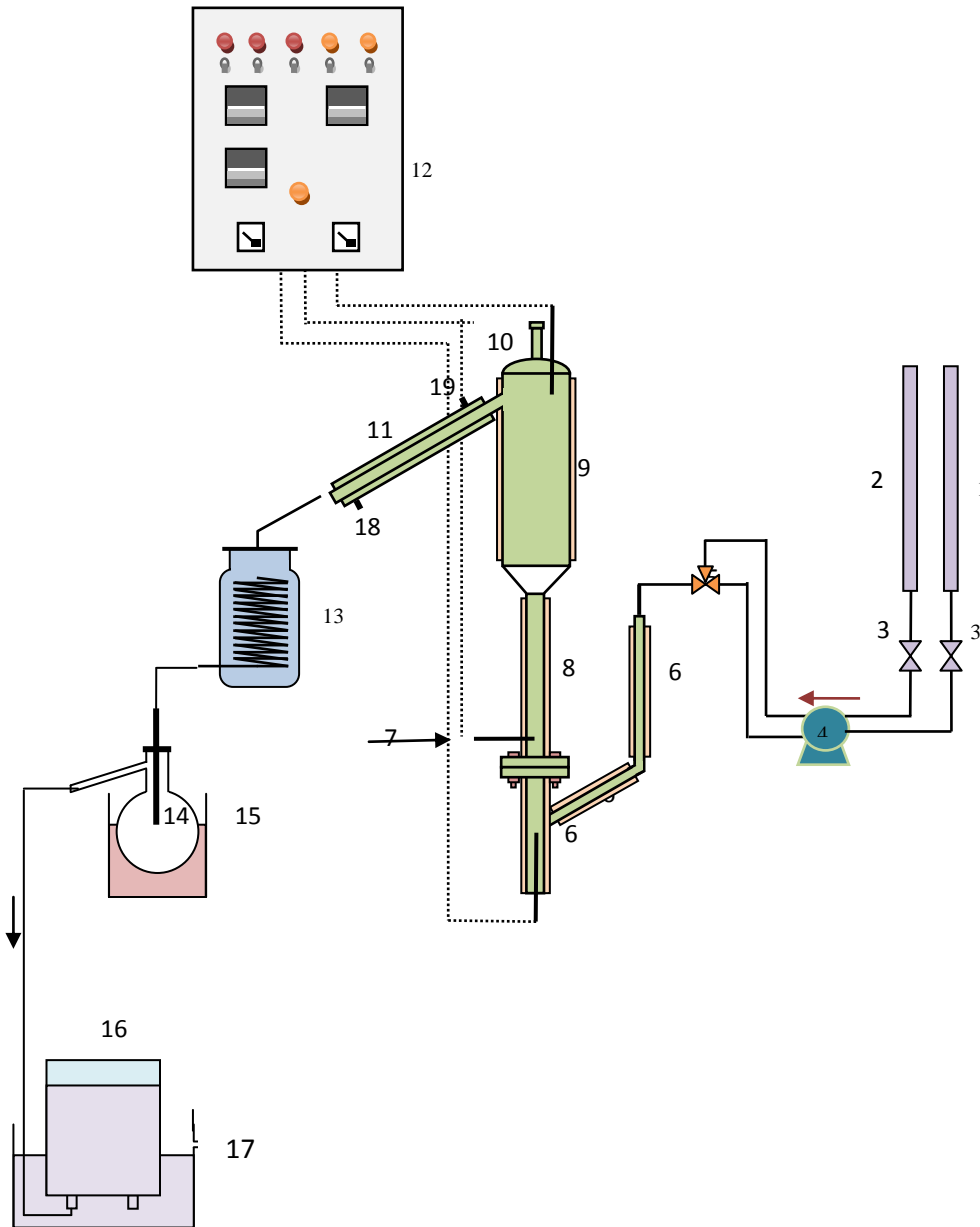


Figure 1. Schematic flow diagram of the fluidized catalytic cracking system:

(1) Burette atmospheric residue supplying; (2) Burette water supplying; (3) Valve; (4) Feeding pump; (5) Three way valve; (6) Preheated part ; (7)Distributor (8)Fluidized bed reactor part; (9)Reactor separation part; (10) Catalyst charge inlet; (11) Double pipe heat exchanger; (12) Control panel; (13) Internal tube ice water bath; (14) Collection and



separation flask; (15) Ice water bath ; (16) Collection of gas ; (17) Water tank; (18) Chilled water in; (19) Chilled water out.

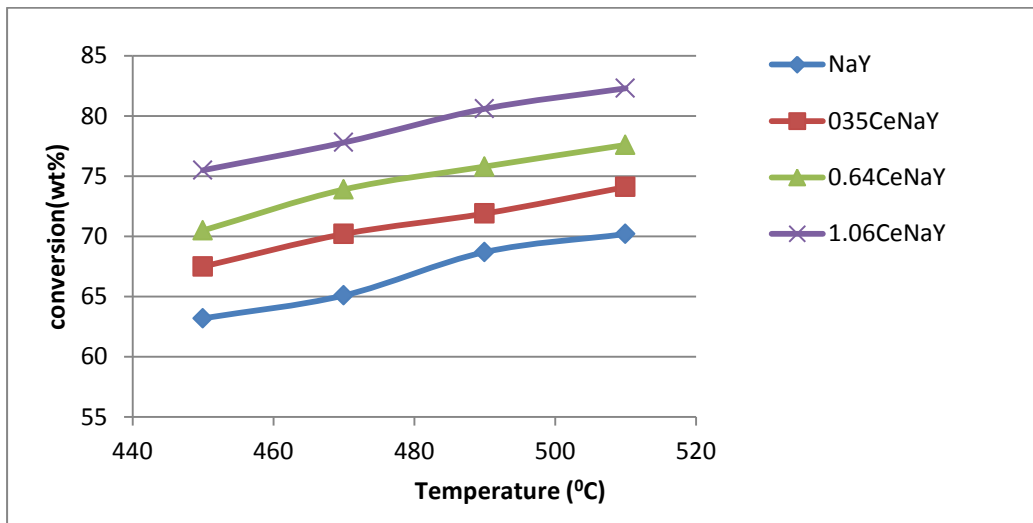


Figure. 2 Effect of cerium weight percent loading on the atmospheric residue conversion at different temperatures and 6 WHSV.

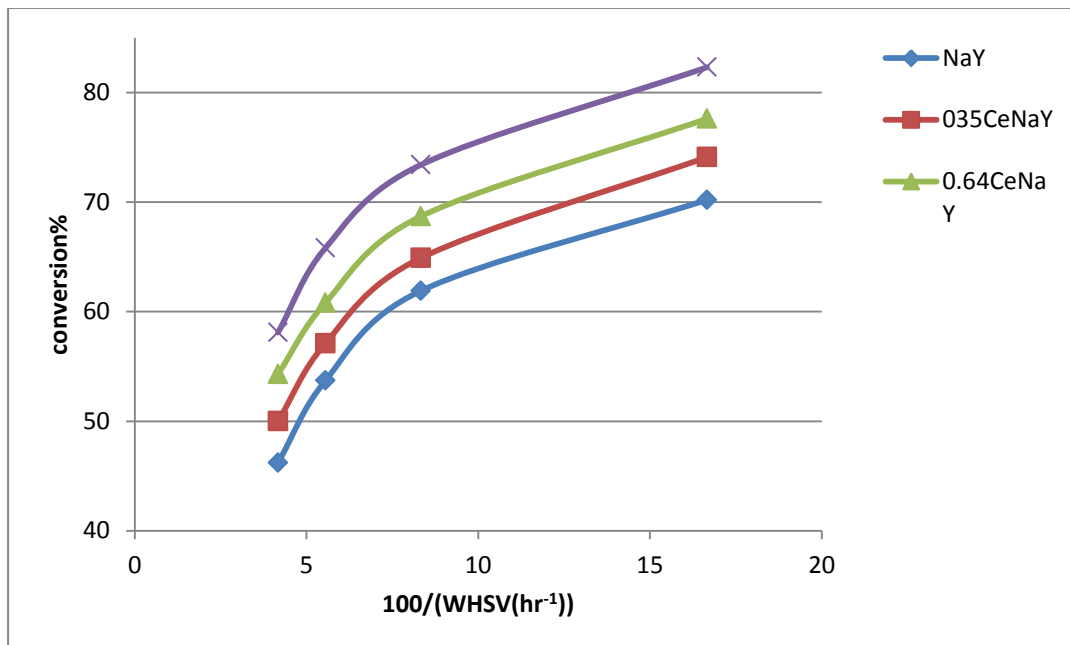


Figure.3 Effect of cerium weight percent loading on the atmospheric residue conversion at different WHSV and temperatures 510°C.

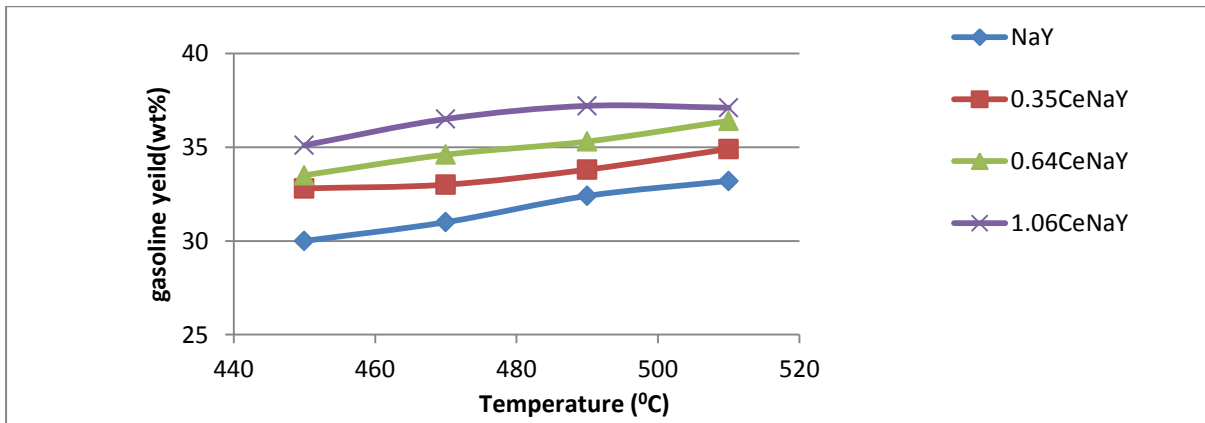


Figure.4 Effect of cerium weight percent loading on the yield of gasoline at different temperatures and 6 WHSV.

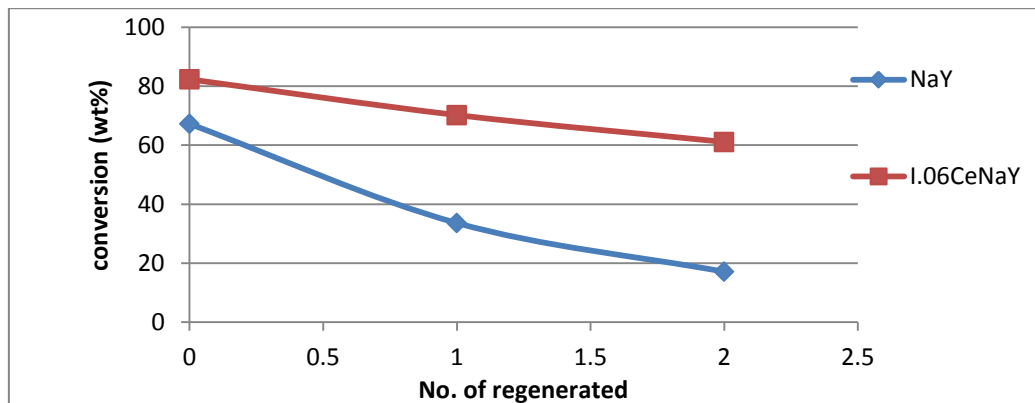


Figure.5 Relation between the conversion and the number of regeneration for NaY and 1.06 CeNaY catalysts.