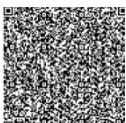# Original Article: Investigation of Thermal and Catalytic Pyrolysis of Polyolefin and Rubbers

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ABSTRACT

In this research, thermal and catalytic pyrolysis and the amount of liquid product obtained from pyrolysis and its composition have been considered. In pyrolysis of each polymer, the amount of liquid product was increased by performing several optimization steps on the reactor. According to studies, among the three catalysts used, the FCC catalyst produces the most liquid product and the HZSM-5 catalyst releases most of the product as gas due to the small pore size. By gas chromatography test, the percentage of liquid product components, aromatic, olefin, paraffin and naphthenic in the product were obtained and also liquid product components were obtained based on the number of carbons. The results of gas chromatography test show that a significant percentage of the product in pyrolysis with FCC catalyst is olefins. Evaluation can be said that in pyrolysis of rubbers (polybutadiene rubber and styrene butadiene rubber) due to the presence of many double bonds in their structure and the formation of many radicals during pyrolysis, a pyrolysis process will be multi-stage and unlike plastic pyrolysis. The hydrocarbon liquid product is extracted separately and in several stages. The trend of temperature changes in pyrolysis of tires is also increasing and decreasing.

Introduction

The use of polymers, plastics and rubbers in everyday life leads to the production of millions of tons of waste, which is one of the most important issues in urban life [1-3]. There are several ways to deal with this waste, the most important of which are: landfill, landfill with energy from it, waste incineration and heat and electricity recovery, recycling and production of compost (fertilizer), reuse and waste conversion to other compounds such as petroleum liquids. Proper use of waste can become one of the main sources of human energy in the not-too-distant future [4-6]. Global production of tires and plastics is increasing day by day, and consequently the production of plastic and rubber waste has an upward trend. The life of this waste is up to months (agricultural film) and years (cars, household appliances) and sometimes even It lasts up to a century (in some construction applications) [7-9].

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The effect of temperature on the pyrolysis process

Temperature is the most important parameter affecting the pyrolysis process because if there is no temperature and heating does not occur, pyrolysis will not occur. Further studies have shown that with increasing temperature, we have a decrease in the amount of liquid product. Another point is that with increasing temperature, the Dils-Alder and dehydrogenation reactions become more intense and the number of aromatic compounds increases, and at higher temperatures, it leads to the formation of heavy fleas [10-12].

On the other hand, with increasing temperature, the heat transfer rate increases and the pyrolysis process occur faster, so the residence time or reaction time decreases. Reaction time is one of the key parameters in the design of pyrolysis production line and reduction of production cost.

At higher temperatures with increasing temperature due to increased chain failure, the percentage of gaseous products increases [13-15]. Various studies have shown that the rate of conversion increases with temperature and with increasing temperature the composition percentage of liquid and gaseous products changes. It should be noted that with increasing pyrolysis temperature, the effect of catalyst on pyrolysis decreases.

By using catalysts, the activation energy for plastic failure and the required temperature are reduced. High temperatures can accelerate the breakdown of carbon chains and significantly improve gasoline efficiency and the rate at which plastics are converted to heavy oils. The required temperature will vary depending on the type of plastic and the desired structure composition for the product.

At temperatures above 600°C, the products are essentially a combination of fuel gases such as 2H, CH4 and light hydrocarbons, while at 400-600°C, wax and liquid fuel are produced [16].

The liquid fuel product consists mainly of naphtha, heavy oil, gasoline, diesel oil and kerosene. In general, polyethylene and polypropylene are converted to fuel oil and gas by pyrolysis, while polystyrene produces most styrene monomer and light hydrocarbons. If a suitable catalyst is used, the failure temperature is reduced to 200-300 °C and on the other hand the efficiency of liquid products is increased [17-19].

The effect of temperature on catalytic failure

The effect of temperature on the catalytic failure of different plastics has been investigated by Huani. As the temperature increases, the conversion rate increases first, but then gradually decreases until it finally reaches a constant value. But the efficiency of gasoline starts to decrease after a certain temperature and large amounts of coke and gas are produced. The optimum temperature varies depending on the type of plastic [20-22]. In general, larger extrusion bands in the side chains make it easier to break down the plastic. Therefore, the temperature order required for pyrolysis of PP, PS and PVC is as follows:

PS <PP <PVC

The main products and conditions of PE, PP, PS and PVC pyrolysis are summarized. The Rgp parameter is a measure of the pyrolysis rate, or product production rate. As we can see in figure (1), with increasing temperature, the pyrolysis rate increases and the diagram has a steeper slope than time. The highest pyrolysis rate occurs at very short times, at the very beginning, and after a period of time, the pyrolysis rate decreases because there is no product to exit [23]. As the temperature decreases, the destruction is completed in longer times [24].
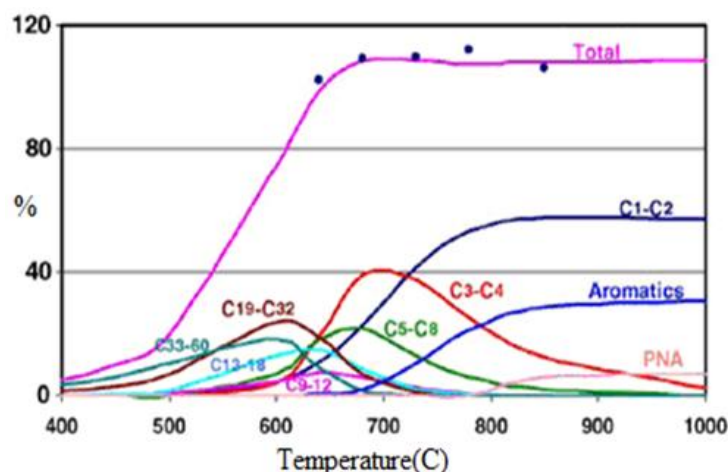
Figure 1: The effect of temperature on the composition percentage of heavy pyrolysis polyethylene products [25]

At lower temperatures, the production of hydrocarbons with high molecular mass is higher. As the temperature rises, the products move towards liquefaction (oil). Then it goes to gasoline products and with the continuation of this process, the production of aromatics begins. When the temperature rises above 700 °C, we move towards the production of hydrocarbon compounds with a molecular mass [26-28].

Effect of catalyst on pyrolysis process

FCC catalysts have been used on an industrial scale in the oil refining industry and have essentially been developed into lighter and more desirable gasoline to break down the heavy oil components from crude oil. The FCC catalyst is composed of zeolite crystals and a non-zeolite acid matrix (usually silica alumina and a binder). Over the last forty years, zeolite Y has been an element of FCC catalysts due to product selectivity and high thermal stability.

This catalyst has a double porosity distribution and is used to optimize the distribution of hydrocarbons in the liquid product.

One of the most important factors of FCC catalyst is that with its control over the product, most products in the gasoline range can be achieved [29].

In the catalytic process, the degradation of polymer chains occurs on the surface of the catalyst, which increases the selectivity of the product to a large extent (Table 1), the passage of products obtained through the pores of the catalyst. Research in this field shows that the products in the absence of catalyst in polyethylene pyrolysis to produce wax and in the presence of FCC catalyst in a reactor with proper heat transfer to the gasoline range. Table (1) investigates the effect of catalyst on pyrolysis in five percent different from zero to sixty percent of catalyst compared to heavy polyethylene polymer at 450°C and stirring speed 50RPM [30].

Table 1: Effect of FCC catalyst on pyrolysis in five percent different from zero to 60% of catalyst compared to heavy polyethylene at 450°C and stirring speed RPM50

Stay time (minute)	Coke%	Percentage of incompressible products	Percentage of compressible products	Catalyst percentage
50	0.3	6.5	93.8	**0**
40	0.4	6.4	94.18	**15**
30	0.5	6.3	98.28	**25**
25	0.6	7.5	94.38	**35**
20	0.7	8.5	43.48	**45**

The results show that without the presence of catalyst, although the product is semi-solid, the most liquid product is obtained.

However, a liquid product with a large amount of wax is not suitable for consumption as a hydrocarbon fuel. With 20% of catalyst, the highest amount of hydrocarbon fuel (about 90%) is obtained [31]. And further increase of catalyst will have a downward trend in liquid product production. With the increase of catalyst due to the increase of catalyst level as

well as its porosity in the reactor and intensification of Dils-Alder reactions and dehydrogenation, the percentage of coke production increased and on the other hand with the increase of catalyst from zero to sixty percent, coke from 7 increases to 0.6 to 2.6 percent [32].

FCC Catalyst

Table (2) shows the specifications of the FCC catalyst.

Table 2: Specifications of the FCC catalyst used

The amount of	Properties
333 square meters per gram	Level rate
50%	SiO$_2$ level
15%	Al$_2$O$_3$ content
0.8%	Level
1.2%	Level
7	Si / Al ratio

H-Mordenite catalyst

H-MOR40 catalyst with Mordenite structure made by German company Süd-Chemie AG is used.

HZSM-5 catalyst

H-MFI90 catalyst with H-ZSM-5 structure made by German company Süd-Chemie AG is used.

Heavy polyethylene

Heavy polyethylene used is HD 6050 grade. Which is a heavy-duty polyethylene injection grade with a narrow molecular weight distribution. This polymer is used in cases where high strength, easy process ability and good impact strength are required [33].

Investigation of heavy polyethylene catalytic pyrolysis system with FCC catalyst

Heavy polyethylene catalytic pyrolysis with 18% FCC catalyst was performed at 450°C in a semi-continuous spiral stirrer reactor under nitrogen gas [34-36].

Table 3: Percentage of thermal and catalytic pyrolysis products of heavy polyethylene in different experiments

Specific density	Percent of coke	Percentage of gas product	Percentage of liquid product	Product percentage the experiment
-	2.5	4.2	93.5	HD/1
-	8.5	29.2	65.4	HD/F/15/1
-	9.5	24.2	70	HD/F/15/2
0.672	2.55	3.7	91.1	HD/F/15/3
-	2.45	44.8	46.7	HD/Z/15/1
0.715	2.65	37.1	54.6	HD/Z/15/2
-	2.85	37	58.5	HD/M/15/1
-	2.15	29.7	68	HD/M/15/2

Effect of different catalysts on pyrolysis of polybutadiene rubber

By pyrolysis of polybutadiene rubber in the best conditions with 15% of FCC catalyst, about 85.2 g of liquid product and 2.2 g of coke were produced. Pyrolysis of polybutadiene rubber using H-Mordenite catalyst also produces about 86.4% of the liquid product. The liquid product produced with the FCC catalyst had the highest amount compared to other catalysts. On the other hand, the amount of coke left by pyrolysis of polybutadiene rubber with FCC catalyst is less than H-Mordenite catalyst. In general, compared to HZSM-5 and H-Mordenite, FCC catalysts have a better effect on the degradation of polybutadiene rubber in terms of fluid production [37-39].

As the results show, in pyrolysis of polybutadiene rubber with H-Mordenite catalyst, aromatics with more than 36% and in pyrolysis of polybutadiene rubber with FCC catalyst, olefins with 40% Contains a significant amount of hydrocarbon fluid.

Effect of FCC catalyst on pyrolysis of polybutadiene rubber

The effect of FCC catalyst value on pyrolysis of polybutadiene rubber in five catalyst concentrations between zero and sixty percent compared to polymer is evaluated in table (4).

Table 4: Effect of FCC catalyst value on pyrolysis of polybutadiene rubber

Specific density	Percent of coke	Percentage of gas product	Percentage of liquid product	Product% the experiment
0.76	1.3	8.1	90.6	PBR /0
0.665	2.2	12.6	85.2	PBR/F/15/6
0.718	6.7	4	89.3	PBR/F/30/1
0.717	7	0.5	92.5	PBR/F/45/1
0.721	8.1	5.2	86.7	PBR/F/60/1

The results show that pyrolysis of polybutadiene rubber with 45% FCC catalyst with 92.5% produces the most liquid product. However, in comparison with the non-catalyst state and other percentages of catalyst, the thermal degradation produces more than the catalytic degradation, i.e. with 90.6% the most liquid product. As the amount of catalyst increases from zero to sixty percent, the amount of coke increases from 2.2 to 8.1. The results of the analysis of the obtained fleas show that they are polyaromatics with high molecular mass and are resistant to heat at high temperatures and do not break down into compounds with lower molecular mass [40-42].

In general, some fleas form in most cases, and they are often resistant to heat even at high temperatures. As mentioned before, coke production has a negative effect on the phenomenon of heat transfer and mass and practically increases the production cost. As the amount of catalyst in the pyrolysis system increases and the fracture energy decreases, the reaction time is significantly reduced, but this does not mean that increasing the amount of catalyst causes a dramatic change in the pyrolysis reaction. The work done in this regard shows that active and high-level catalysts at the same low levels also affect the reaction [43-45].

The coke formed in the pyrolysis of tires appears to be three-dimensional and closed due to the many double bonds present in the rubber. This coke sits on the catalyst, closing its cavities and not allowing the rubber chains to pass into the catalyst. Coke production during the pyrolysis process is not desirable at all. Prolonged stay or high temperatures exacerbate these reactions [46-48].

The coke produced in pyrolysis rubbers is different from plastics and in a way the three-dimensional crosslinks produced in pyrolysis rubbers act like a cage and prevent the movement of the catalyst in the mass of the

rubber and therefore the effect of the catalyst on the pyrolysis of the rubbers. Far less than plastics. Table (5) shows the distribution of different compounds in the liquid product resulting from the pyrolysis of polybutadiene rubber with different percentages of FCC catalyst [49].

Table 5: Distribution of different compounds in the liquid product resulting from pyrolysis of polybutadiene rubber with different percentages of FCC catalyst

PBR/F/60/1	PBR/F/45/1	PBR/F/30/1	PBR/F/15/6	PBR/0	Sample Product components (Mole percentage)
43.2	38.9	35.5	22.3	20.5	Aromatic
25.0	22.3	21.7	19.8	20.4	Oil
16.3	17.4	22.0	40.1	39.0	Alfin
15.5	21.4	20.8	17.8	20.1	Paraffin

Table 8: Distribution of liquid product in terms of carbon number and percentage of products in the gasoline range resulting from pyrolysis of polybutadiene rubber with different percentages of FCC catalyst

PBR/F/60/1	PBR/F/45/1	PBR/F/30/1	PBR/F/15/6	PBR/0	Sample Product groups (Weight percentage)
9.3	5.3	2.8	11.6	0.1	Bhutan
5.6	5.2	5.1	6.2	1.3	Pantan
13.2	16.3	19.5	31.6	9.7	Hexane
13.6	17.2	14.2	12.9	7.8	Heptane
17.5	18.1	17.9	15.4	17.0	Octane
17.8	15.2	15.2	14.8	36.7	Noonan
9.1	10.2	9.1	1.8	9.8	store
13.9	12.5	16.2	5.7	17.6	+ Dokan

The results show that at zero and fifteen percent of FCC catalysts in pyrolysis of polybutadiene rubber, olefins are the main share of products [50-52]. However, by increasing the catalyst from 30% upwards, the aromatic percentage in the product increases with a gentle slope and thus the amount of olefin decreases. However, the percentage of paraffin's and naphthenic in the liquid product do not show a specific trend with the amount of catalyst [53-55]. As mentioned earlier, the FCC catalyst has a twin pore distribution [56-58]. During the pyrolysis process, although the tendency of chains to pass through larger cavities is much greater than that of smaller cavities, groups of chains become aromatic rings as they pass through smaller cavities. As the percentage of catalyst increases and also the number of smaller pores increases, the percentage of aromatics in the liquid product increases [58].

Conclusion

According to the data obtained from various tests, it can be concluded that:

➤ By performing catalytic pyrolysis of heavy polyethylene with FCC, H-Mordenite and HZSM-5 catalysts, it was concluded that the amount of liquid product with FCC catalyst is higher than other catalysts.

➤ The distribution of different compounds in the product of heavy polyethylene pyrolysis liquid with FCC catalyst shows that the major part of the product is olefins. While with the ZSM-5 catalyst, naphthenic are the main component of the liquid product. According to the results, using FCC catalyst, six carbon compounds have the largest share among the liquid product and with HZSM-5 catalyst, five carbon

compounds with more than 22% constitute the major part of the liquid product. Compared to HZSM-5 and H-Mordenite, FCC catalysts have a better effect on the degradation of polypropylene in terms of liquid product distribution.

➢ The results also show that in the polypropylene pyrolysis product, olefins have the main share with more than 55% of the products. On the other hand, the number of olefins in the final product in polypropylene pyrolysis is less than polyethylene's. This may be related to the structure of polypropylene and the presence of methyl groups.

➢ In the process of pyrolysis of rubbers, due to the presence of many double bonds in the structure of the polymer and the formation of many radicals during pyrolysis, huge polymer networks are produced and the effect of the cage is strengthened.

➢ In this way, as the temperature increases, the polymer networks are re-destroyed and the resulting radicals can subsequently become new polymer networks.

➢ In the same way, the process of destroying this network and producing radicals continues. Therefore, in the process of degrading tires, we will have a multi-stage pyrolysis process, and unlike pyrolysis of plastics, the hydrocarbon liquid product is extracted separately in several stages.

➢ The process of temperature changes with grams of liquid product during pyrolysis is increasing and decreasing. The FCC catalyst is a catalyst with a pore size distribution of two peaks, so it is suitable for passing both small and large chains into its cavities. 6-12 carbon polymer chains are able to easily pass through its pores and do not allow the chain to turn into gas with further failure.

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