

Thermal Cracking of Waste Engine Oil (Lubricant) at Different Temperatures



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Abstract

The recycling of used oil became the need of hour due to economic, environmental, public health and legal reasons. Using waste lubricating oil as a source of energy or feedstock for chemical industries is an intelligent option for any country, more so for India, as it would conserve both the valuable natural resources as well as foreign exchange.

Used automotive engine oil (Lubricant) is thermally cracked at 420°C, 430°C and 440°C using batch reactor, with the intention of assessing the suitability of the process in recovering valuable products from this otherwise difficult to dispose off waste engine oil. The yield and properties of the recovered products were determined.

The recovered cracked liquid and gaseous products can be used as valuable blending components for fuels and/or as feedstock for various chemical processes. The results indicate the effect of temperature on thermal cracking of waste engine oil when cracked in batch reactor. The recovery of commercially valuable products show advantage over traditional, more destructive disposal methods, and suggests excellent potential for scaling the process to the commercial level.

Keywords:

1. Waste engine oil- Lubricating oil used in IC engines
2. Cracking- Decomposition of hydrocarbons molecules in smaller molecules
3. Combustion products- Products formed during combustion of fuel in IC engine
4. Degradation products- Products formed due to deterioration of lubricating oil during combustion of fuel in combustion chamber due to heat and friction
5. Petrochemicals- Chemicals manufactured from petroleum
6. ASTM- American society for testings and materials
7. API- American petroleum institute

Introduction

The current lube oil demand in India is of the order of more than 10 lakh tonnes per year. Out of this, almost 60% accounts for automotive and the rest of 40% for industrial lubricants. Indian crude is predominantly waxy, hence, not suitable for lubricating base oil production. In fact, our entire lube oil production is based upon imported lube bearing crude. Huge amount is spent every year towards procurement of lube base stocks and importing the crude oil to meet increasing demand of fuels. More than 75% of total requirement of crude oil is imported from the other countries to meet the rising demand of fuels and petrochemicals. (14, 15)

Large and increasing volumes of lubricating oil are produced each year. After use, they are considered a hazardous waste. After a certain period of useful life, the lubricating oil loses its properties and cannot be used as such in machinery because of their high content of thermal degradation products from the base oil & additives and combustion products from the fuel and lubricant. (6,7,8,9,10,18)

In our country, re-refined oil is generally termed as spurious. This is the possible reason that is why; the very concept of re-refining could not become successful in our country. In developed countries, re-refined oil is not regarded as spurious. It is rather regarded as an important source of non-conventional energy. There exist well defined guidelines for its (used oil) collection, transportation, processing and re-use (16). Composition of Used Oil Lubricating oil does not wear out during use. It is only the additive part, which gets depleted (18). The oil molecules are not degraded, but because of presence of following contaminants, oil needs replacement:

1. Free and emulsified water.
2. Light hydrocarbons, i.e. gasoline and gas oil.

3. Dust, rust and soot.
4. Metals (iron, copper, zinc, lead, calcium, phosphorus, etc.) resulting from engine wear and corrosion.
5. Gasoline/diesel and lube additives.
6. Products of thermal degradation, i.e., carbon, unsaturated hydrocarbons, polymers and asphaltenes.
7. Complex compounds from additive packages. (10, 12, 17)

Facts about Hazardous Nature of Used Oils

Used oils themselves are not toxic, but the contaminants such as additives, degradation products, etc. make them so hazardous.

1. They have high potential to cause damage to the environment by virtue of their persistent nature and potential to spread over large surface areas on land and water.
2. Films of oil prevent light and air from reaching to life forms of all types on land and water.
3. As per the data, one liter of oil can render one million liters of fresh water unusable.

For these reasons, most of the countries in the world have classified used oil as hazardous waste and introduced regulations for its collection, handling and disposal. These regulations clearly recommend re-refining of used oils over burning or other means of disposal. (1,2,3,4,5)

Unlike virgin crude oils, re-refined oil is a renewable source of energy. Re-refining or recycling of used oils helps in the following ways:

1. Conservation of valuable oil reserves by using the oil again and again.
2. Saving of huge amount of foreign exchange.
3. Checks environmental degradation and saves ecology. (12)

When used oil is dumped on the ground, sewers or sent to landfills, it is capable of seeping into ground and surface water. Just one liter of used oil can render one million liters of water undrinkable. It is also a serious threat to plant and animal life (1,2,3,4,5,13). Marine species can be adversely affected by oil concentrations as low as one ppm. The oil film on water blocks sunlight, making it harder for plants to photosynthesize. Loss of plants and animal life, while tragic, also results in economic loss. When used oil is burnt, the presence of various contaminants and usually high water content, results in the incomplete combustion, thus increasing the air pollution. Hence, re-refining is a much better option. (6,7,8,9,10,11,12,)

S. N.	Property	Observations
1.	Redwood Viscosity	a) at 40°C--- 424 seconds. b) at 100°C---61 seconds.
2.	Specific Gravity at 29°C	0.8885
3.	API Gravity at 29°C	27.7589°API
4.	Pour Point	-24°C
5.	Flash Point By Cleveland Open Cup Method	193°C
6.	Fire Point By Cleveland Open Cup Method	252°C
7.	Conradson carbon Residue (wt%)	0.9088%

Sample

In this study, waste engine oil is used as a sample. The waste engine oil is collected from different private two wheeler service stations in Amravati city and mixed together to form a single homogeneous sample. This sample is a typical feedstock for the experiments.

Prior to the runs the oil was filtered to remove solid particle and then heated to 150°C with continuous stirring and maintained for one hour to eliminate water. This filtered and dehydrated waste oil (FDWO) was used as starting material in all experimental runs.

In the refinery conversion processes carry out chemical reactions that fracture ("crack") large, high-boiling hydrocarbon molecules (of low economic value) into smaller, lighter molecules suitable, after further processing, for blending to gasoline, jet fuel, diesel fuel, petrochemical feedstocks, and other high-value light products. The conversion processes of primary interest are fluid catalytic cracking (FCC), hydrocracking, and coking.

The basic principle is breaking large hydrocarbon molecules into smaller ones thus increasing the value of low value materials. Lighter liquid fuels are generally more valuable than heavier ones. Gasoline for example, is more valuable than diesel fuel.

Thermal cracking process can be successfully applied for manufacturing distillates from a feedstock of used oil. The significant aspect of this technology is its ability to adjust process operating conditions to tailor the desired products. This can be a tremendous advantage in used oil processing due to the variability of the used oil feedstock. It follows that the process parameters can be adjusted to vary the boiling range of product; the process can also be manipulated to maintain a target product quality with feed variability.

This thermal cracking technology is emerging as the technology of choice for progressive environmental services companies as they consider technology choices. Conversion of used oil to gasoil is desirable from an environmental point of view since the product displaces the need to consume a virgin distillate produced from crude oil. This could be a

significant benefit to the world environment in aiding to solve the used oil problem. Of all the methods of processing used oil to be consumed for its calorific value, the thermal (cracking) conversion to distillates technology is the highest form of processing method available, from an economical and environmental viewpoint.

Waste engine oil (two wheeler petrol engine) is used as a feedstock for cracking. This oil is filtered to remove the solid particles and removed moisture by maintaining 150°C temperature for one hour. This filtered and dehydrated waste oil (FDWO) is used as the feedstock for the experiments. Basic tests of this feed sample are done, e.g. Redwood viscosity, pour point, viscosity index and CCR. The results of these tests are shown in Table-1.

Table-1: Properties of Filtered and Dehydrated Waste Engine Oil

1200ml (1090.5gms) of feed is cracked in the batch reactor at 420°C, 430°C, 440°C. It is observed that as reactor temperature inches closer to 400°C vapors started coming out of the reactor indicating the start of cracking reactions. The temperature of the reactor is immediately raised and maintained at cracking temperature as close as possible throughout the process. As the reaction reached to completion, vapors stopped coming out of the reactor, heating stopped and allowed the reactor to cool. Liquid products as a distillate and residue is collected from the reactor. Total material balance on reactor is done to study the total conversion, gasification and residue formed when cracked at different temperature in batch reactor.

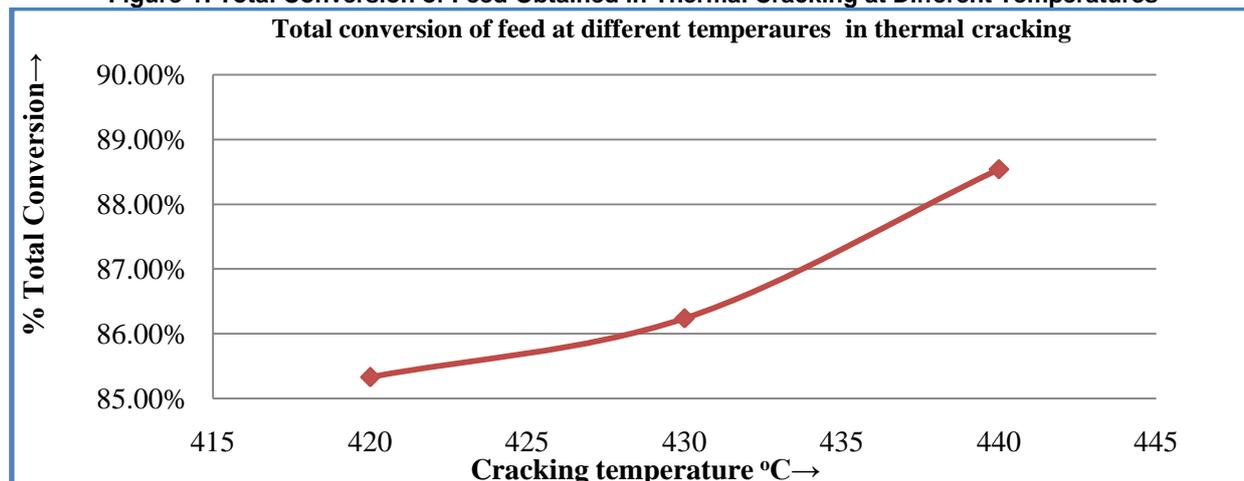
Material Balance of Feed When Thermally Cracked at Different Temperatures
Table-2: Important Observations of Experiments of Thermal Cracking

S.N.	Cracking Temp. °C →	420	430	440
1.	Liquid product obtained, (gms)	833.1	840.8	861.4
2.	% (wt) Liquid product obtained	76.39	77.10	79.00
3.	Residue formed, (gms)	160.0	150.0	125.0
4.	% (wt) Residue formed	14.67	13.75	11.46
5.	Feed gasified, (gms)	97.40	99.70	104.10
6.	% (wt) Amount of feed gasified	08.93	09.14	09.55
7.	Total Amt. of product formed, (liquid+gases) gms	930.50	940.50	965.50
8.	% (wt) Total conversion	85.33	86.24	88.54
9.	Time required to start liquid collection, (mins)	47	48	41
10.	Time required from 400°C to completion of cracking, (mins)	52.00	42.00	27.00
11.	Total time required for cracking, (mins)	100	90	70

When waste engine oil is cracked at different temperatures, it is observed that percent total conversion (liquid products + gaseous products) increased with the increase in cracking temperature (Figure-1). 85.33%, 86.24% and 88.54% (wt.) total conversion is obtained at 420°C, 430°C and 440°C

cracking temperatures respectively. It is observed that with increase in temperature yield of liquid product is also increasing (Figure-2). 76.39%, 77.10% and 79.00% (wt.) liquid products are obtained when waste engine oil is cracked at 420°C, 430°C and 440°C respectively.

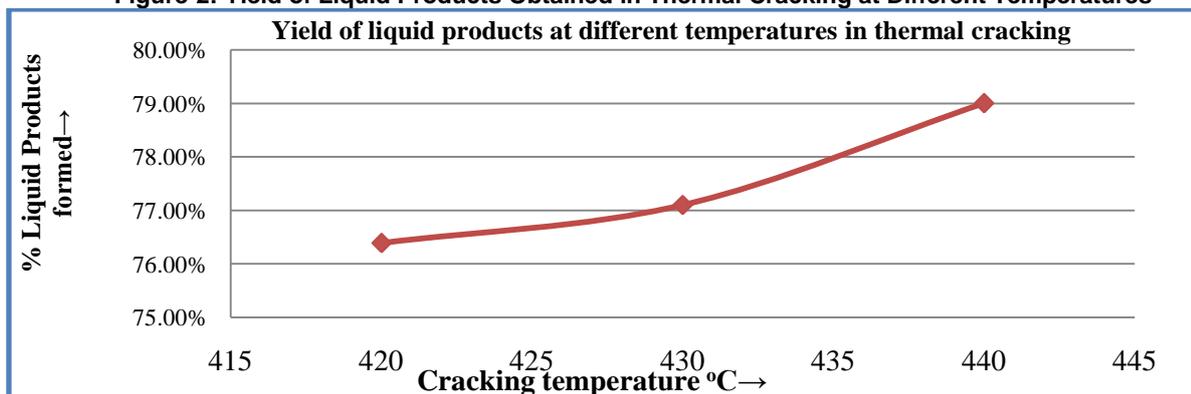
Figure-1: Total Conversion of Feed Obtained in Thermal Cracking at Different Temperatures



As there is an increase in total conversion of feed with increase in cracking temperature, formation of residue is declining consistently with rise in temperature (Figure-3). This may be due to high temperatures and less contact time, since the reaction is carried out in batch reactor the heavy hydrocarbon

molecules are decomposed to smaller ones and immediately pushed out of the reactor, providing very little time to undergo further cracking. This observation is well supported by the time required for cracking.

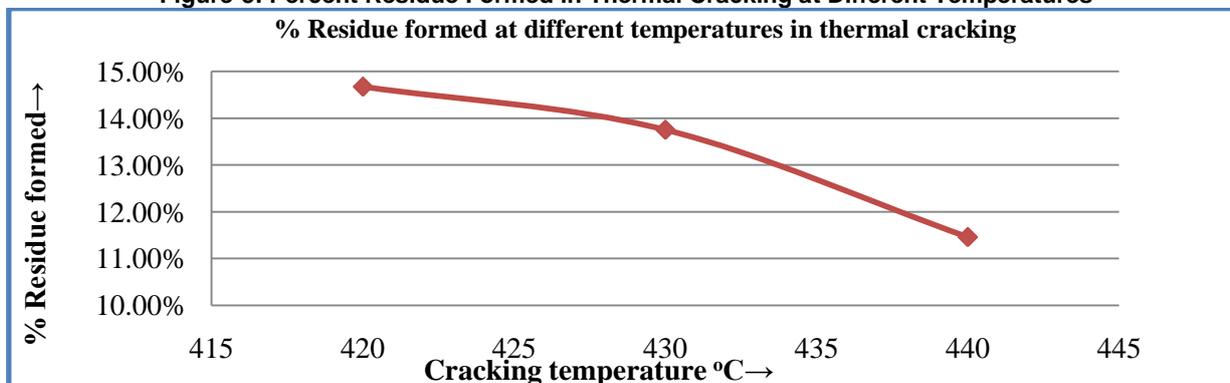
Figure-2: Yield of Liquid Products Obtained in Thermal Cracking at Different Temperatures



14.67%, 13.75% and 11.46% (wt.) residue formed when the feed is cracked at 420°C, 430°C and 440°C temperatures respectively (Figure-3). Residue formation is decreasing with increase in cracking temperature. And this is due to the less contact time

available; which is decreasing the extent of cracking minimizing further cracking of cracked products/molecules reducing the possibility of ultimate cracking leading to the residue formation during cracking operation.

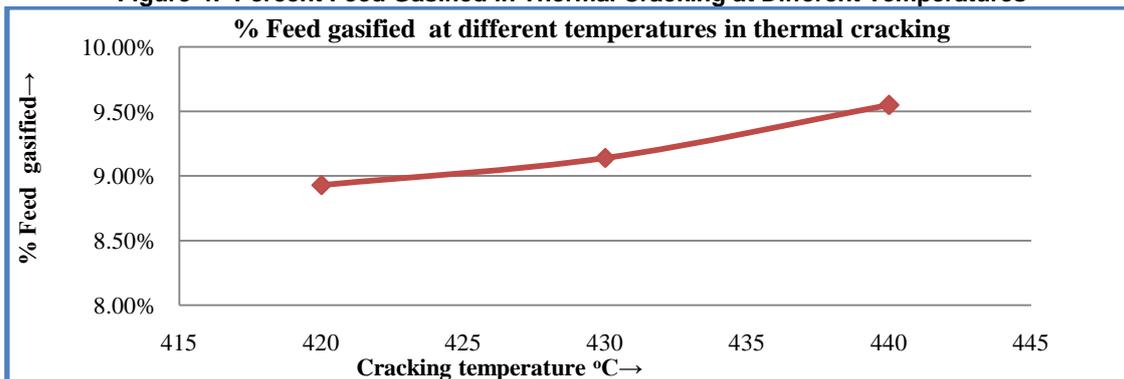
Figure-3: Percent Residue Formed in Thermal Cracking at Different Temperatures



As the ultimate cracking of feed is minimized due to less contact time, the formation of gaseous product increased slightly with increase in cracking temperature (Figure-4). 8.93%, 9.14% and 9.55% of

feed is converted to gaseous hydrocarbons that left the system without condensing when feed is cracked at 420°C, 430°C and 440°C respectively.

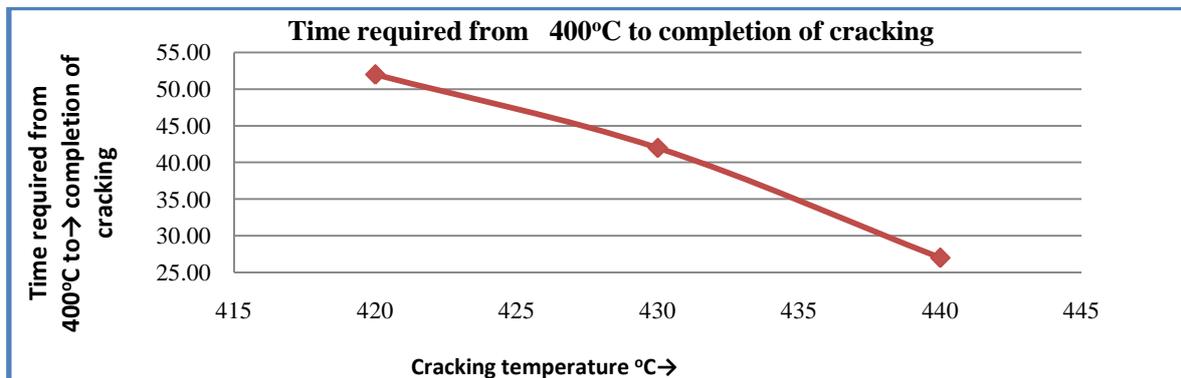
Figure-4: Percent Feed Gasified in Thermal Cracking at Different Temperatures



Time required for all these thermal cracking reactions is shown in Table-2. It is observed that time required for thermal cracking reaction is decreasing with increasing cracking temperature (Figure-5). As the contact time decreased, cracked products/molecules are not subjected to higher

temperatures for longer time to undergo further cracking, that caused the maximum conversion of feed to liquid products and fair amount gaseous products and at the same time possibility of ultimate decomposition to form asphaltic residue also decreased.

Figure-5: Time Required for Complete Thermal Cracking at Different Temperatures



ASTM Distillation Characteristics of Distillates Obtained By Thermal Cracking of Feed

The liquid distillates obtained from thermal cracking experiments are subjected to ASTM

distillations. The ASTM distillation characteristics of these distillates are shown in Figure-6, Figure-7 and Figure-8, and summarized distillation characteristics are shown in Table-3.

Table-3: ASTM Distillation Characteristics of Liquid Distillates Obtained by Thermal Cracking of Feed

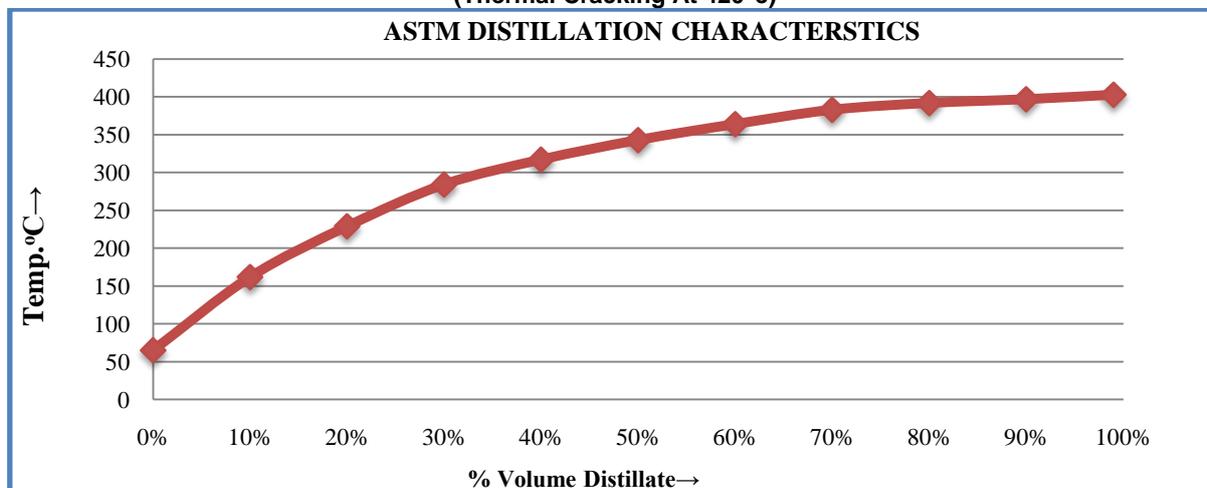
S.N.	Cracking Temp. °C →	420	430	440
1.	IBP, °C	65	80	81
2.	FBP, °C	403	409	410
3.	Vol. % of liquid product in thenaphtha range (<200°C)	15.5	12.0	11.5
4.	Vol. % of liquid product in the gas oil range (200-390°C)	59.5	63.0	73.5
5.	Vol. % of liquid product boiling above 390°C till FBP	24.0	24.0	13.5
6.	Total distillate, %(vol.)	99.0	99.0	98.5
7.	Residue, %(vol.)	0.5	0.5	0.75
8.	Losses, %(vol.)	0.5	0.5	0.75

In thermal cracking experiment that carried out at 420°C, 8.93% (wt.) material is converted to non-condensable hydrocarbons. These liquid products were subjected to various tests such as ASTM distillation (IP123/93), Redwood viscosity (IP70/62 25th edition), Sp. Gravity, Aniline Point (IP2/91, ASTM D611-87, ISO 2977:1989(E)), Conradson carbon residue (CCR) (IP13/82, ASTM D189-88, BS:2000 Part 13:1993), Pour Point (IP15/67, ASTM D97-87, BS:2000:Part 15:1993), Flash and Fire Point by Cleveland Open Cup Method (IP 36/84(1989), ASTM

D92-90)), Total Acidity (IP 1/74 (1990), BS 2000: Part 1:1993)) Bromine Number (IP129/93, BS2000 :Part 129:1993) etc. (19,20)

When cracked liquid product (Thermal cracking at 420°C) is subjected to ASTM distillation characteristics (Figure-6), it is observed that approximately 15.5% (vol.) naphtha range (<200°C) liquid product is formed, while 59.5 % (vol.) gas oil range (200-390°C) product and 24% (vol.) material boils in the range of 390- 403°C are formed.

Figure-6: ASTM Distillation Characteristics of Distillate Obtained from Experiment No.01 (Thermal Cracking At 420°C)



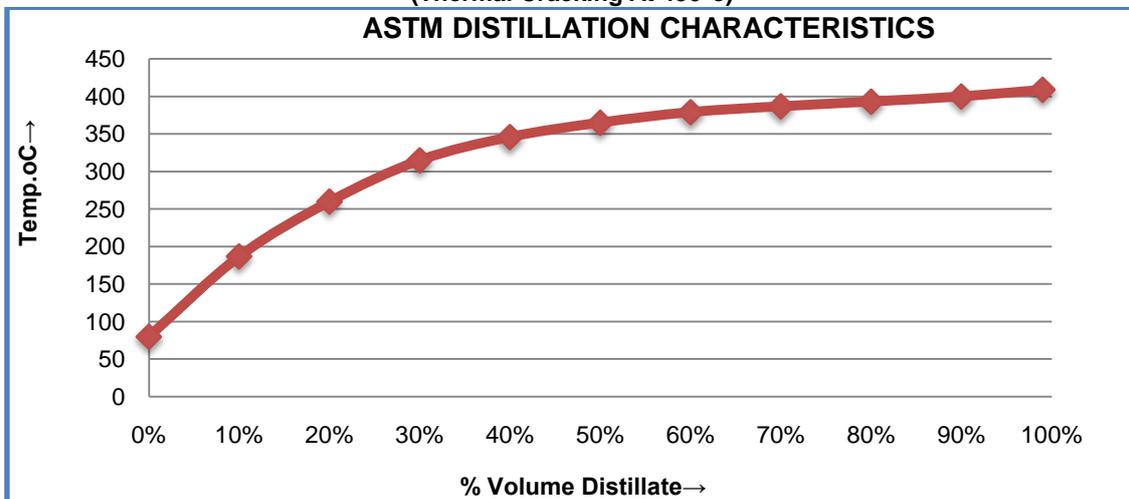
Second thermal cracking experiment is performed at 430°C. ASTM distillation characteristics

(Figure-7) of the product obtained from this experiment shows that around 12% (vol.) material falls

in the naphtha range (<200°C), 63%(vol.) material falls in the gas oil range (200-390°C) and around 24% (vol.) material boils in the range of 390°C-

409°C. Details of the ASTM distillation characteristics are shown in Table-3.

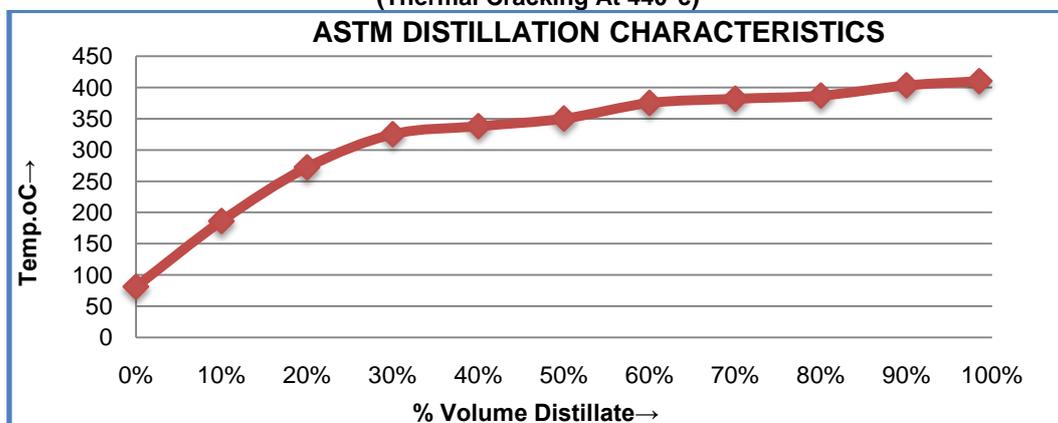
Figure-7: ASTM distillation Characteristics of Distillate Obtained from Experiment No.02 (Thermal Cracking At 430°C)



ASTM distillation characteristics of products obtained from third thermal cracking experiment carried out at 440°C and the details are shown in Table-3. In this test 98.5% distillate is collected during the test. According to the ASTM distillation characteristics approximately 11.5 %(vol.) of cracked

product falls in the boiling range of naphtha (<200°C), while 73.5 %(vol.) in the gas oil range(200-390°C) and almost 13.5% material boils in the range of 390°C to 410°C. 0.5% residue obtained, while 0.5% vapor losses occurred during this test.

Figure-8: ASTM Distillation Characteristics of Distillate obtained from Experiment No. 03 (Thermal Cracking At 440°C)



Properties of Distillates Obtained by Thermal Cracking of Feed

Redwood viscosity, specific gravity, API gravity, aniline point, flash and fire point, Conradson

carbon residue, pour point, bromine number and acid value are determined for the distillates obtained in thermal cracking experiments.

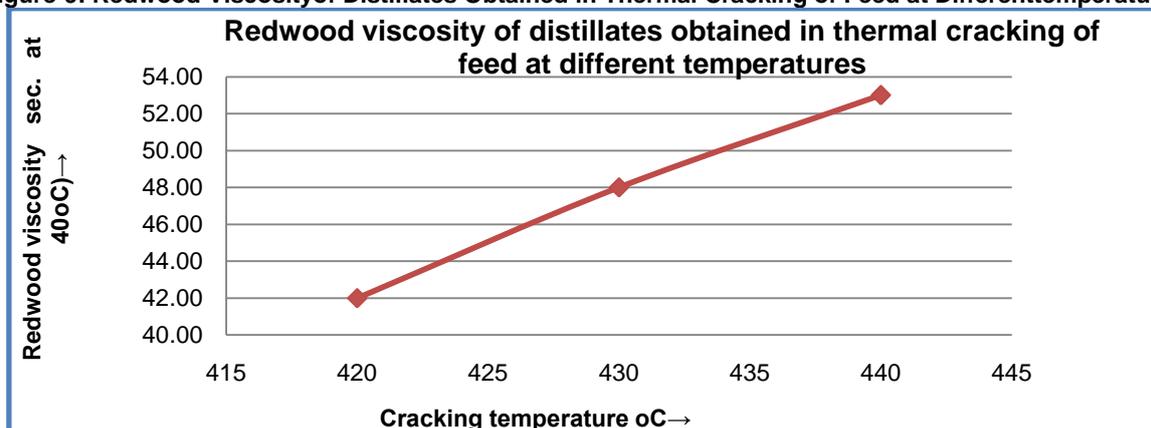
Table-4: Properties of distillates obtained in thermal cracking of feed

S.N.	Cracking Temp. °C → Properties ↓	420	430	440
1.	Redwood viscosity, Seconds(at 40°C)	42.00	48.00	53.00
2.	Specific gravity, (at 29°C)	0.8396	0.8474	0.8519
3.	API Gravity, °API(at 29°C)	37.0326	35.4813	34.5993
4.	Aniline point, (°C)	77.0	84.0	87.0
5.	Flash point by Clevelandopen cup method, (°C)	41	55	76
6.	Fire Point by Clevelandopen Cup Method, (°C)	52	65	88
7.	Conradson Carbon Residue, (wt.%)	0.0230	0.0050	0.0250
8.	Pour point, (°C)	-11	-07	-05
9.	Bromine number	14.46	13.11	15.61
10.	Acid value, mg KOH/gm	0.8427	0.7517	0.7517

Since the contact time decreased with increase in cracking temperature, the cracked material is not subjected to higher temperatures for longer time so further cracking of cracked products is minimized and some of the lighter molecules of feedstock come out alongwith vapors of cracked lighter molecules, therefore at higher temperatures formation and presence of relatively high

boilingmolecules is more in cracked product, that is why Redwood viscosity of liquid products is increasing significantly with increasing cracking temperatures(see Figure-9). The viscosity of the cracked products obtained at 420°C, 430°C and 440°C is 42.00 Redwood seconds, 48.00 Redwood seconds, and 53.00 Redwood seconds (at 40°C) respectively.

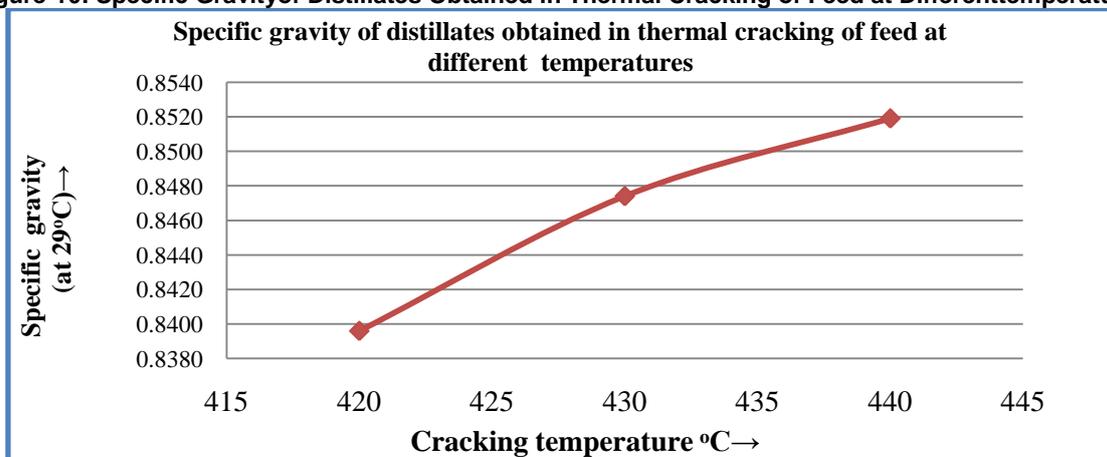
Figure-9: Redwood Viscosity of Distillates Obtained in Thermal Cracking of Feed at Different temperatures.



Rise in viscosity is also supported by increase in specific gravity of cracked liquid products. With the increase in cracking temperatures specific gravity of cracked liquid products is increasing (Figure-10). Higher the specific gravity of the cracked

products lower is the API gravity of these products (Figure-11). The specific gravity is found to be 0.8396, 0.8474 and 0.8519 when feed is cracked at 420°C, 430°C and 440°C (at 29°C) respectively.

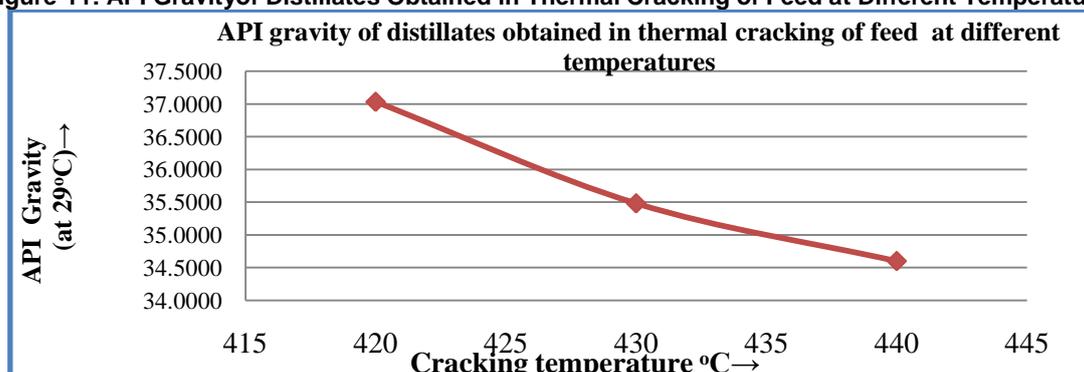
Figure-10: Specific Gravity of Distillates Obtained in Thermal Cracking of Feed at Different temperatures.



It is observed that the API gravity of the cracked liquid products is decreasing with increasing cracking temperature as shown in Figure-11. API

gravity of the cracked products is 37.0326, 35.4813 and 34.5993°API (at 29°C) when waste engine oil is cracked at 420°C, 430°C and 440°C respectively.

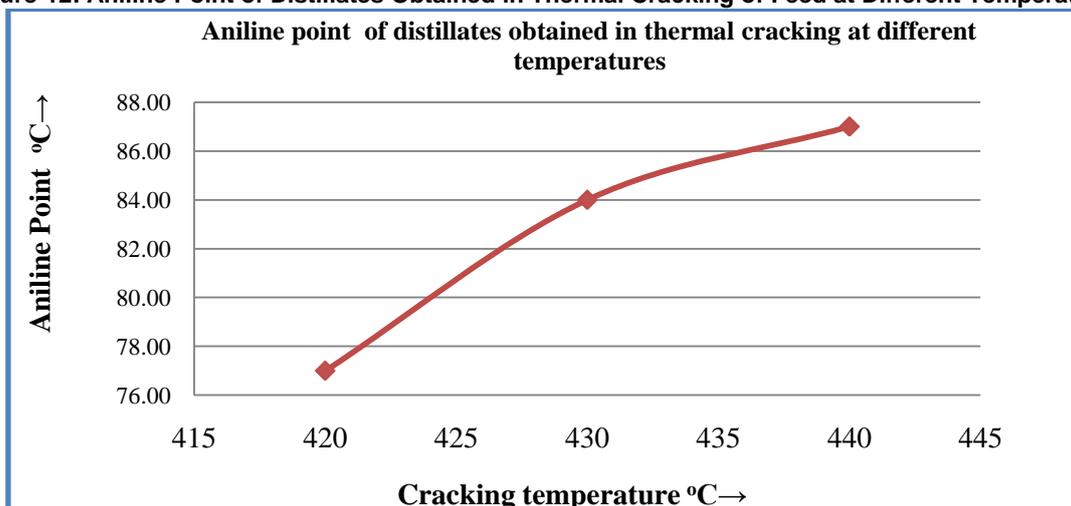
Figure-11: API Gravity of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures



With increase in cracking temperatures cracking time is decreasing as it is a batch reactor that caused the reduction in further more cracking of cracked products and that caused increase in yield of liquid product and little increase in gaseous product formation and lesser residue formation during the cracking. That means paraffinic side chains detached from ring structures are more in the cracked product

which could not undergo further cracking because of less contact time. That caused the increased presence of paraffinic hydrocarbons in cracked products that obtained at high cracking temperatures, so the aniline point of cracked products is increasing with increasing cracking temperatures as shown in Figure-12. The aniline point obtained for these products are as shown in Table-4.

Figure-12: Aniline Point of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures



It is observed that with increase in cracking temperature, the viscosity and specific gravity of the cracked liquid products is increasing. These observations indicate that with increase in cracking temperatures there is decrease in volatility of the products obtained. This observation is well supported by the flash and fire point of these products (see

Figure-13 and Figure-14). Significant increase in flash point and fire point of these products is observed when tested by Cleveland open cup method. The flash point of the products obtained when feed stock is cracked at 420, 430 and 440°C are 41, 55 and 76°C respectively; and fire point for these products are 52, 65 and 88°C respectively.

Figure-13: Flashpoint of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures (By Cleveland open Cup Method)

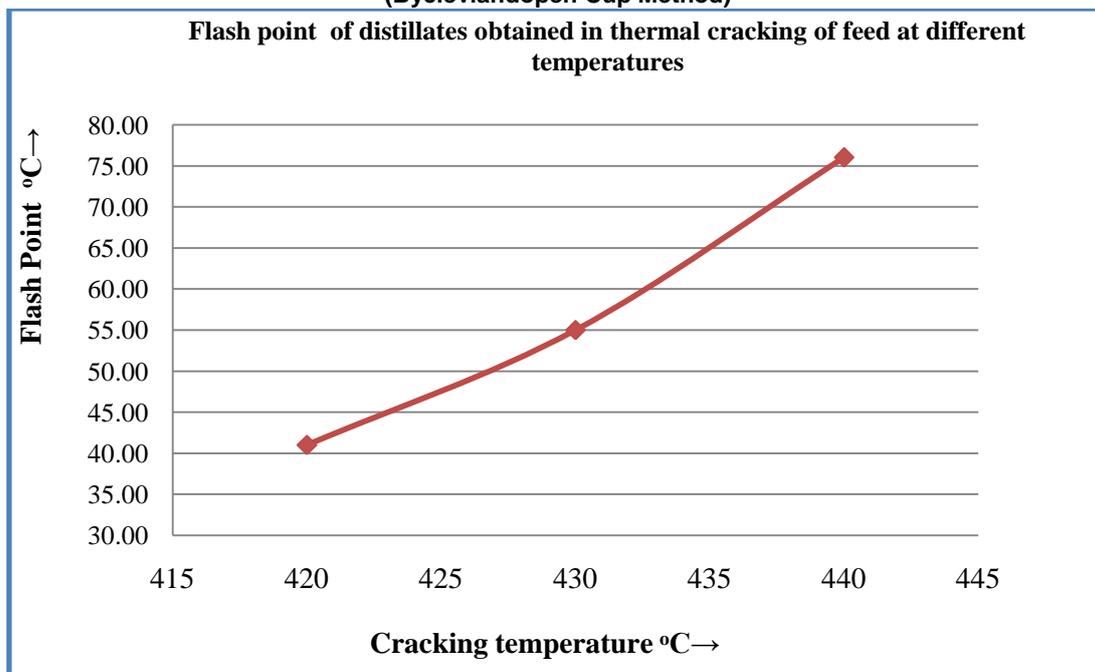
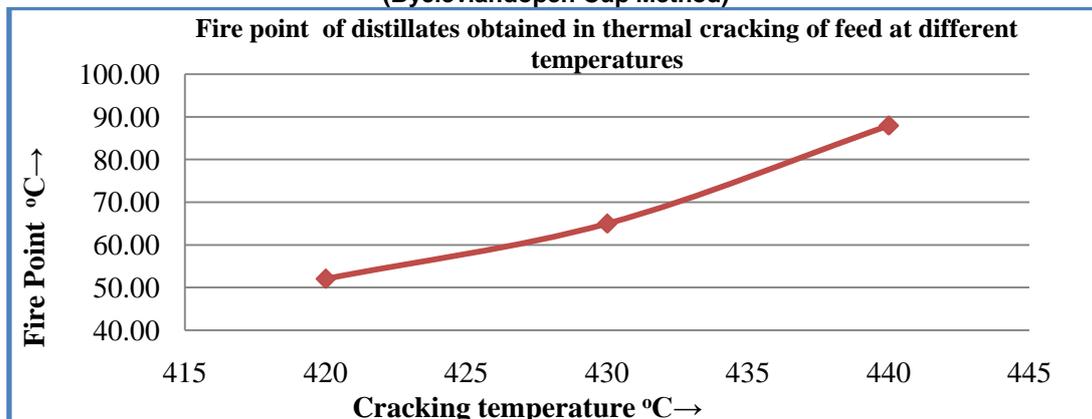
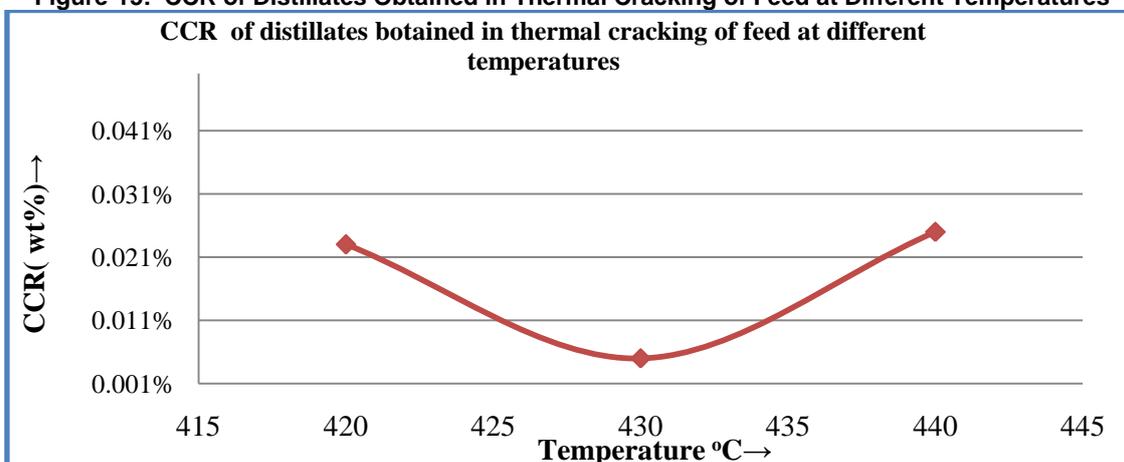


Figure-14: Fire Point of Distillates Obtained In Thermal Cracking of Feed at Different Temperatures (By Clevelandopen Cup Method)



The Conradson carbon residue (CCR) of these products is found to be very low. It is not showing any significant variations with increasing cracking temperatures (Table-4).

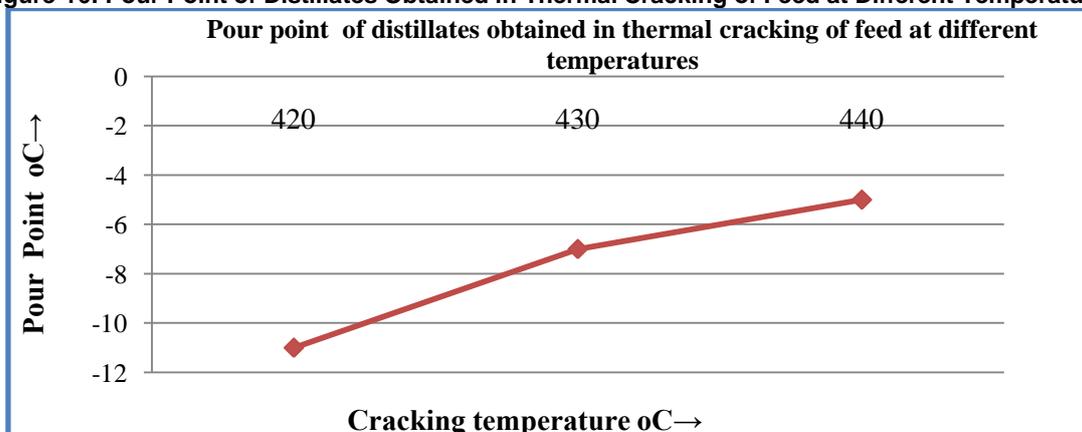
Figure-15: CCR of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures



The presence of more paraffinic hydrocarbons is indicated by the aniline point of these cracked products. With increase in cracking temperature aniline point of the cracked product is increasing, indicating more presence of paraffinic hydrocarbons in the products when feed is cracked at higher temperatures. This observation is well

supported by the pour point of these products. With increase in cracking temperature pour point of cracked product is decreasing as shown in Figure-16. The pour points of the cracked products are -11, -7 and -5°C respectively for the products obtained at 420, 430 and 440°C cracking temperatures respectively.

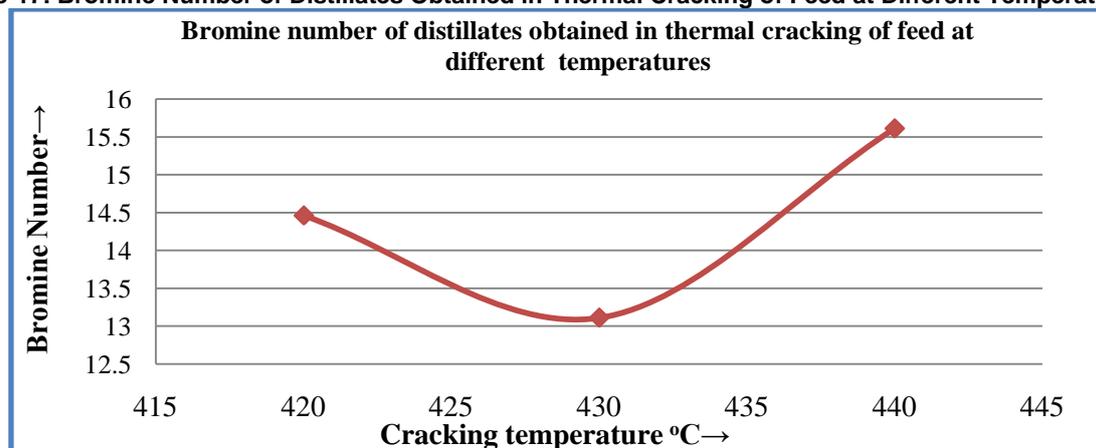
Figure-16: Pour Point of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures



There is not significant variation in bromine number of cracked products. But higher bromine number of thermally cracked product indicates the

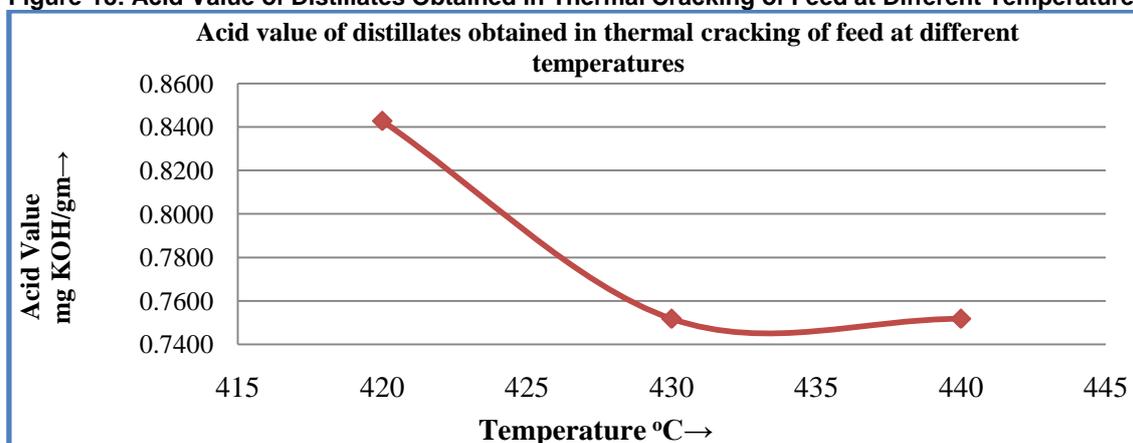
higher presence of unsaturates in these products (Table-4).

Figure-17: Bromine Number of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures.



No significant variation in acid value of thermally cracked product is observed, still with increase in temperature drop in acid value is observed (Table-4).

Figure-18: Acid Value of Distillates Obtained in Thermal Cracking of Feed at Different Temperatures



Conclusions

The thermal cracking of waste engine oil at 420°C, 430°C and 440°C yields good amount of cracked products. The viscosity, specific gravity/API gravity, flashpoint and fire point of products indicates the extent of cracking occurred.

As all the contaminants accumulate in residue, the liquid and gaseous products obtained are free from all type of contaminants. So these products forms high value refinery streams which can be further processed with suitable refinery streams or can be used as the feedstock for various refining and/or petrochemical processes. It should be noted that the problems related to used oil treatments by vacuum distillation, such as fouling of heating and distillation equipment can be avoided by thermal cracking of these oils.

This can be one of the ways to conserve the valuable oil and reducing the rate of depletion of crude oil. So used oil may again be source of fuels and/or feedstock for various petrochemical processes.

The study of material balance and properties of cracked products suggests that the cracking waste engine oil at 440°C temperature yields better results than cracking at 420°C and 430°C. Comparison of these three experiments indicates that product yield i.e. conversion of feed to high value products (liquid and gaseous product together) is good when the feed

is cracked at 440°C. Even though the yield of lighter product (boiling below 200°C) is less, the presence of heavier hydrocarbons (boiling above 390°C) in product is also less and another important aspect is, compared to other experiments carried out at 420°C and 430°C residue formation in this case is less. And the major product obtained is the product boiling in the range of 200°C to 390°C; and yield of this fraction is more when feed is cracked at 440°C.

These observations suggest that cracking of waste engine oil at 440°C is more effective and beneficial than that of cracking at 420°C and 430°C. And when cracking is carried out at 440°C, time required for the cracking process is very much less than that required for the cracking at 420°C and 430°C.

Almost all types of waste oil have the potential to be recycled safely, saving a precious non-renewable source and at the same time minimizing environmental pollution. Besides its great adverse impact on the environment, if used oil is properly recycled and/or reused, it could have significant savings on fresh crude oil. Disposal of used lubricating oil into the eco-system creates environmental hazards. Tough laws are being enacted throughout the world for the disposal of waste petroleum products and every genuine effort should be made for its re-use.

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