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RESEARCH ARTICLE

QUALITY IMPROVEMENT OF REFORMAT GASOLINE BY USING ANILINE AND TOLUENE

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ABSTRACT

The main objective of this project was blending of aniline and toluene with local reformat gasoline, to enhance the octane number of reformat gasoline product from Khartoum refinery (Sudan). Reformat gasoline was tested according to American society for testing and materials(ASTM), Tests included distillation, sulfur content, density, vapor pressure, oxidation stability, cupper strip corrosion, gum existence, lead content, and octane number determination by Cooperative Fuels Research (CFR engine). Previously tetra ethyl lead (TEL) and tetra methyl lead (TML) have been used to improve the fuels octane number, but they have negative effects on the environment, and have been banned internationally. The motor octane number (MON) of reformat gasoline used in this research was recorded (88.5) before additives. In order to enhance the ON of reformat gasoline, aniline and toluene were added to the reformat gasoline with different concentration (5, 10,% v/v). Octane number of blends was measured by CFR engine. The properties of reformat gasoline after all additions were improved to the limits assigned by ASTM and Khartoum refinery. Motor octane number (MON) was increased into the range of 1 - 7. Addition of 10% v/v aniline showed high value of MON, but addition of 5% v/v toluene showed lower value of MON.

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INTRODUCTION

Gasoline, also called gas or petrol, mixture of volatile, flammable liquid hydrocarbons derived from petroleum and used as fuel for internal-combustion engines. It is also used as a solvent for oils and fats. Originally a by-product of the petroleum industry (kerosene being the principal product), gasoline became the preferred automobile fuel because of its high energy of combustion and capacity to mix readily with air in a carburetor (Nichols, 1997). Gasoline was at first produced by distillation, simply separating the volatile, more valuable fractions of crude petroleum. Later processes, designed to raise the yield of gasoline from crude oil, split large molecules into smaller ones by processes known as cracking. Thermal cracking, employing heat and high pressures, was introduced in 1913 but was replaced after 1937 by catalytic cracking, the application of catalysts that facilitate chemical reactions producing more gasoline (Mcnair, 2001). Other methods used to improve the quality of gasoline and increase its supply include polymerization, converting gaseous olefins, such as propylene and butylene, into larger molecules in the gasoline range; alkylation, a process combining an olefin and a paraffin such as isobutene; isomerization, the conversion of straight-chain hydrocarbons to branched-chain hydrocarbons; and reforming, using either heat or a catalyst to rearrange the molecular structure (Domask, 1984). Gasoline is a complex mixture of hundreds of different hydrocarbons. Most are saturated and contain 4 to 12 carbon atoms per molecule. Gasoline used in automobiles boils mainly between 30° and 200° C (85° and 390° F), the blend being adjusted to altitude and season. Aviation gasoline contains smaller proportions of both the less-volatile and more-volatile components than automobile gasoline, the antiknock characteristics of a gasoline—its ability to resist knocking, which indicates that the combustion of fuel vapour in the cylinder is taking place too rapidly for efficiency—is expressed in octane number. The addition of tetra ethyl lead to retard the combustion was initiated in the 1930s but was discontinued in the 1980s because of the toxicity of the lead compounds discharged in the combustion products. Other additives to gasoline often include detergents to reduce the buildup of engine deposits, anti-icing agents to prevent stalling caused by carburetor icing, and antioxidants (oxidation inhibitors) used to reduce "gum" formation, in the late 20th century the rising price of petroleum (and hence of gasoline) in many countries led to the increasing use of gasohol, which is a mixture of 90 percent un leaded gasoline and 10 percent ethanol (ethyl alcohol)

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(Zagentd, 2001). Gasohol burns well in gasoline engines and is a desirable alternative fuel for certain applications because of the renewability of ethanol, which can be produced from grains, potatoes, and certain other plant matter (Sax, 1989).

Octane number

Octane number (ON) is one of the most important properties of gasoline streams and is a measure of its antiknock property. It is defined as the volume percentage of iso - octane in a blend of n - heptane and iso - octane, which produces the same knock intensity as the test fuel under standard test conditions in an ASTM internal combustion engine. ASTM defines two different types of ONs, the research octane number (RON) and the motor octane number (MON), which are evaluated using the ASTM D2699 and the ASTM D2700 tests, respectively (Vanleenawen, 1994). Both methods use the same standard test engine but differ in the operating conditions. RON is measured in an engine running at 600 rpm and a fuel/air mixture at a temperature of 60 ^{o}F, while MON is measured with the engine running at 900 rpm and a fuel/air mixture at a temperature of 300 ^{o}F. The slower engine speed and the lower fuel/air temperature as required in the RON test are representative of the fuel performance for city driving, while the faster engine speeds and higher fuel/air temperature represent the fuel performance for highway driving Knock results from the premature combustion of the gasoline, due to compression in the engine (Lago *et al.*, 1999). As the fuel/air mixture is compressed in the internal combustion engine, certain molecules in gasoline tend to self-ignite even before they reach the ignition spark, thereby creating a resistive expansive motion in the compression stroke of the engine and hence the knock. Depending on the thermal stability of the molecule (which depends on its molecular structure) and the ensuing radicals, certain molecules tend to combust sooner (and knock more) than others. consequently, ON is a direct function of the molecular composition of the gasoline fuel, and any modeling effort should explicitly acknowledge it (Lago *et al.*, 1999).

MATERIALS AND METHODS

Materials

All chemicals used were of analytical reagent grade (AR) and of highest purity degree available. They included hydrochloric acid, iso octane, n-heptane, methyl isoketone, standard lead (II), reformat gasoline product from Khartoum refinery.

Procedures

Gasoline specification

Gasoline are usually defined by government regulation, where properties and test methods are clearly defined. In the US, several government and state bodies can specify gasoline properties, and they may choose to use or modify consensus minimum quality standards, such as American Society for Testing Materials (ASTM). The US gasoline specifications and test methods are listed in several readily available publications, including the Society of Automotive Engineers (SAE), and the Annual Book of ASTM Standards (ASTM, 2005).

Standard test method for distillation of petroleum products at atmospheric pressure

- Based on its composition, vapor pressure, expected IBP or expected EP, or combination there, of the sample is placed in one of five groups. Apparatus arrangement, condenser temperature, and other operational variables are defined by the group in which the sample falls.
- A 100 mL sample is placed in a round bottom flask and heated at a rate specified for samples with its vapor pressure characteristics. Temperatures are recorded when the first drop is collected(initial boiling point), at recorded volumes of 5 mL, 10 mL, every subsequent 10mL interval to 90 mL, 95 mL and at the end of the test(end point). For gasoline samples, the temperatures associated with each incremental volume percentage recovered are converted to temperatures for each incremental volume percentage evaporated by correcting for any sample loss during the test.
- At the conclusion of the distillation, the observed vapor temperatures can be corrected for barometric pressure and the data are examined for conformance to procedural requirements, such as distillation rates. The test is repeated if any specified condition has not been met.
- Test results are commonly expressed as percent evaporated or percent recovered versus corresponding temperature, either in a table or graphically, as a plot of the distillation curve (ASTM, 2005).

Fig. 2.1. Distillation apparatus assembly using electric burner

Standard test method for density of petroleum products

A small volume (approximately 0.7 mL) of liquid sample is introduced into an oscillating sample tube and the change in oscillating frequency caused by the change in the mass of the tube is used in conjunction with calibration data to determine the density of the sample(ASTM., 2005).

Standard test method for vapor pressure of petroleum products

The chilled sample cup of the automatic vapor pressure instrument is filled with chilled sample and is coupled to the instrument inlet fitting.

Fig. 2.2. Density apparatus at Khartoum refinery laboratory

The sample is then automatically forced from the sample chamber to the expansion chamber where it is held until thermal equilibrium at 37.8°C (100°F) is reached. In this process the sample is expanded to five times its volume (4:1 vapour – to - liquid ratio). The vapour pressure is measured by a pressure transducer. The measured vapor pressure is automatically converted to a DVPE value by the instrument. A correction to this value is necessary to account for the observed bias between the test result and that obtained by Test Method D 4953.

Recording to the following:

Calculate a DVPE, using the following equation. This corrects the instrument reading for the relative bias found in the 1991 inter laboratory cooperative test program (see Note 10) between the dry vapor pressure measured in accordance with test Method D 4953, Procedure A and this test method:

DVPE = (0.954 X)+ A

Where:

X = measured total vapor pressure, in units consistent

with A, and A = 1.94 kPa (0.281 psi), (ASTM, 2005).

Standard test method for gum content in fuels by jet evaporation

A measured quantity of fuel is evaporated under controlled conditions of temperature and flow of air or steam. For aviation gasoline and aviation turbine fuel, the resulting residue is weighed and reported as milligrams per 100 mL. For motor gasoline, the residue is weighed before and after extracting with heptane and the results reported as milligrams per 100 mL. Recording to the following:

Fig. 2.3. Vapour pressure apparatus at Khartoum refinery laboratory

Wight of Gum = weight of (peaker gum) - (peaker plank) ÷2000 The weight is so Alblanc worth its weight in pre-heating and weighed the purpose of heating, but if there was a difference between these two weights raises the value of the sample (glue) and then recording the output is obtained. That's where 2000 = factor, (ASTM, 2005).

Fig.2.4. Gum content apparatus at Khartoum refinery laboratory

Standard test method for copper strip corrosion

A polished copper strip is immersed in a given quantity of sample and heated at a temperature (200^{o}C) and for a time (2 hours) characteristic of the material being tested. At the end of this period the copper strip is removed, washed, and compared with the ASTM copper strip corrosion standards shown in Fig 3.5 (ASTM, 2005).

1a	1b	2a	2b	2c	2d	2e	3a	3b	4a	4b	4c
Slight tarnish		Moderate tarnish					Dark tarnish		Corrosion		
🟨		🟧					🟫		⬛		

Fig. 2.5. The ASTM copper strip corrosion standard

Standard test method for motor Octane Number

The Motor ON of a spark-ignition engine fuel is determined using a standard test engine and operating conditions to compare its knock characteristic with those of PRF blends of known octane number. Compression ratio and fuel-air ratio are adjusted to produce standard knock intensity for the sample fuel, as measured by a specific electronic detonation meter instrument system. A standard knock intensity guide table relates engine compression ratio to octane number level for this specific method. The fuel-air ratio for the sample fuel and each of the primary reference fuel blends is adjusted to maximize knock intensity for each fuel.

The fuel-air ratio for maximum knock intensity may be obtained (1) by making incremental step changes in mixture strength, observing the equilibrium K.I. value for each step, and then selecting the condition which maximizes the reading or (2) by picking the maximum knock intensity as the mixture strength is changed from either rich-to-lean or lean-to-rich at a constant rate. Bracketing Procedures—the engine is calibrated to operate at standard knock intensity in accordance with the guide table. The fuel-air ratio of the sample fuel is adjusted to maximize the knock intensity, and then the cylinder height is adjusted so that standard knock intensity is achieved. Without changing cylinder height, two primary reference fuels are selected such that, at their fuel-air ratio for maximum knock intensity, one knocks harder (higher K.I.) and the other softer (lower K.I.) than the sample fuel.

A second set of K.I. measurements for sample fuel and reference fuels is required, and the sample fuel octane number is calculated by interpolation in proportion to the differences in average knock intensity readings, a final condition requires that the cylinder height used shall be within prescribed limits around the guide table value for the calculated octane number. Bracketing procedure ratings may be determined using either the equilibrium fuel level or dynamic fuel level approach. Compression Ratio Procedure—A calibration is performed to establish standard knock intensity using the cylinder height specified by the guide table for the octane number of the selected primary reference fuel. The fuel-air ratio of the sample fuel is adjusted to maximize the knock intensity under equilibrium conditions; the cylinder height is adjusted so that standard knock intensity is achieved. The calibration is reconfirmed and the sample fuel rating is repeated to establish the proper conditions a second time. The average cylinder height reading for the sample fuel, compensated for barometric pressure, is converted directly to octane number using the guide table. A final condition for the rating requires that the sample fuel octane number be within prescribed limits around that of the octane number of the single primary reference blend used to calibrate the engine to the guide table standard knock intensity condition (ASTM, 2005).

Fig. 2.6. CFR engine

Method for Determination of lead concentration in gasoline by atomic absorption spectrophotometer (A.A.S)

Preparation of sample

about 30 mL of solvent methyl Isoketone ware taken , a 5 mL of buffer solution ware added, then 0.1g of iodine was weighted and transferred to the solution, then 5 mL of gasoline ware added to the solution, finely the solution was completed to the mark in a 100 mL volumetric flask with iso butyl ketone (ASTM, 2005).

Preparation of the standard lead (II) solution

Preparation of standard lead (II) solutions (0.5, 1.0 and 2.0 ppm).

Blank of instruments

Deionized water, is zero the device use and the injected into the sample where a process of atomization, so atoms of lead in gasoline (in the path of the light from the bulb short) and the device read the focus directly (Kerr and Sarede, 1997).

Fig. 2.7. Atomic absorption spectrophotometer at Khartoum refinery laboratory

Standard test method for oxidation stability of fuels

The sample is oxidized in a pressure vessel initially filled at 15 to 25°C with oxygen pressure at 690 to 705 KPa and heated at a temperature between 98 and 102°C. The pressure is recorded continuously or read at stated intervals until the breakpoint is reached. The time required for the sample to reach this point is the observed induction period at the temperature of test, from which the induction period at 100°C can be calculated. Precaution: In addition to other precautions, to provide protection against the possibility of explosive rupture of the pressure vessel, the pressure vessel should be operated behind an appropriate safety shield. Recording to the following:

The number of minutes from the time the pressure vessel is placed in the bath until the breakpoint has been reached is the observed induction period at the temperature of the test. Method of Calculation—Calculate the induction period at 100°C from one of the following equations:

- When the test temperature is above 100°C:
  Induction period at 100°C = (IPt) (1 + 0.10) (t a – 100)
- When the test temperature is below 100°C:
  Induction period at 100°C, min = (IP t)/(1 + 0.101)(100 – tb)

where:

IPt = induction period, min, at the temperature of the test,
t_a = test temperature when above 100°C, °C, and
t_b = test temperature when below 100°C (ASTM, 2005).

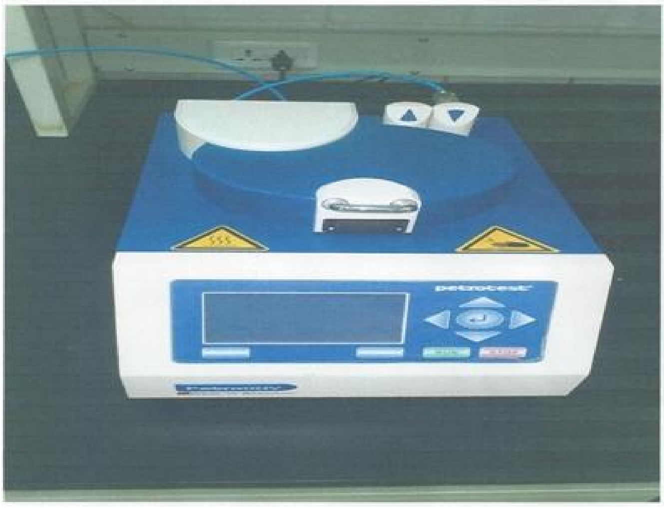

Fig. 2.8. Stability apparatus at Khartoum refinery laboratory

Standard test method for sulfur content in gasoline by energy dispersive X-ray fluorescence spectrometry

The sample is placed in the beam emitted from an X-ray source. The resultant excited characteristic X radiation is measured, and the accumulated count is compared with counts from previously prepared calibration standards to obtain the sulfur concentration in mg/kg. One group of calibration standards is required to span the concentration 5 to 1000 mg/kg sulfur (ASTM, 2005).

Fig. 2.9. Sulfur Content Apparatus in Al Khartoum Refinery

Method for Addition of aniline and toluene to reformate gasoline at different concentration

Aniline and toluene additives are gasoline soluble chemicals mixed with reformate gasoline to enhance octane number of reformat gasoline.

Typically, they are derived from petroleum based raw materials and their fractions, chemistry are highly specialized. Antiknock compounds increase the antiknock quality of gasoline, because the amount of additive needed is small, they are the lowest cost method for increasing octane number compared with changing gasoline chemistry. Aniline and toluene were added to the reformate gasoline at different vol% as follow:

- 1000 ml of reformat gasoline was prepared at refrigerator temp. In glass container had fitting cover.
- Octane number of gasoline was measured by CFR engine, and all properties of gasoline detected before additives.
- two glass container were filled with 1000ml of reformate gasoline and added aniline with shaking by using pipette in different concentrations (5,10%).
- 4-Octane number of these blend were measured by CFR engine, and all properties was detected after adding.
- step 3 and 4 were Repeated with toluene (Ezeldin and Massad, 2015)

RESULTS AND DISCUSSION

In this project reformat gasoline used was produced from Khartoum refinery, aniline and toluene were added to improve octane number of local reformat gasoline. Quality control of Sudanese gasoline is checked by applying many tests according to American Society for Testing and Materials (ASTM). These tests included: distillation, density, vapour pressure, stability, Copper corrosion, lead content, gum content, sulfur content and octane number. The experimental work was conducted at central laboratories of Khartoum refinery (Algily, north Khartoum) and central petroleum laboratories (CPL), alamarat, str. 61, Khartoum.

Table 3.1. Reformat gasoline before and after adding aniline (5, 10% v/v)

Test name	R. G. before additions		1000 mL R. G+ 50 mL analine		1000 mL R.G + 100 mL analine	
Distillation	IBP	37.9°C	IBP	32 °C	IBP	25 °C
	10 %	58.5°C	10%	54.1 °C	10%	36 °C
	50 %	97.2 °C	50%	86.4 °C	50%	76 °C
	90 %	159.0°C	90%	149 °C	90%	133 °C
	FBP	190.5°C	FBP	182 °C	FBP	170 °C
Density	736.4 Kg/m^3(150°C)		690 Kg/m^3(150°C)		681 Kg/m^3(150°C)	
Vapor pressure	52.5 KPa(37.8°C)		55 KPa(37.8°C)		64.9 KPa(37.8°C)	
Gum content	0.8 mg/100ml		0.8 mg/100ml		0.95 mg/100ml	
Copper corrosion	1a		1a		1a	
Lead percentage	0.001mg/L		0.001 mg/L		0.001mg/L	
Oxidation stability	504 min		304 min		Min	
Sulfur percentage	58.49 mg/L		49.3 mg/L		40 mg/L	
Pressure reading in CFR engine	0.583KPa		0.506KPa		0.498KPa	
Motor octane number	88.5		93.8		95.5	

Key: R.G ≡ Reformat Gasoline, IBP ≡ Initial Boiling Point, FBP ≡ Final Boiling point

Table 4.2. Reformat gasoline before and after adding toluene (5, 10% v/v)

Test name	R. G. before additions		1000 mL R. G+ 50 mL toluene		1000 mL R. G+100mL toluene	
Distillation	IBP	37.9°C	IBP	34.2°C	IBP	31 °C
	10%	58.5°C	10%	53.3°C	10%	48 °C
	5%	97.2 °C	50%	93.6°C	50%	85.5 °C
	90%	159.0°C	90%	156.1 °C	90%	152.3°C
	FBP	190.5°C	FBP	186.4 °C	FBP	180 °C
Density	736.4 Kg/m^3(150°C)		700.3 Kg/m^3(150°C)		682.3 Kg/m^3(150°C)	
Vapor pressure	52.5 KPa(37.8°C)		49 KPa(37.8°C)		48.2KPa(37.8°C)	
Gum content	0.8 mg/100ml		0.8 mg/100ml		0.95 mg/100ml	
Copper corrosion	1a		1a		1a	
Lead percentage	0.01mg/L		0.001mg/L		0.001mg/L	
Oxidation stability	504 min		302 min		300 min	
Sulfur percentage	58.49 mg/L		58.49 mg/L		58.49 mg/L	
Pressure result in number engine	0.583KPa		0.549KPa		0.500KPa	
Motor octane number	88.5		91		94.2	

Specific volume of local reformat gasoline (1000 mL) was treated by mixing different volumes (50 & 100 mL) with different concentrations (5 &10 % v/v) of aniline and toluene, respectively. Tests of quality control of reformate gasoline were carried out before and after additions. The obtained results of these tests according to ASTM are shown in Tables 3.1, 3.2 respectively. The obtained results in the above tables revealed that, distillation test of reformat gasoline before and after treatment showed decrease in final boiling points (FBP).

Therefore, these results give a strong evidence, that the quality of the reformat gasoline has improved after two additions of (5, 10% v/v) of aniline and toluene. On the other hand, the initial boiling points (IBP) of the same reformat gasoline sample after treatment were decreased. This may attributed to the conversion of some liquid petroleum gas (LPG) into fuel (Gibbs and Bonazza, 2008). Also, decrease in initial boiling points (IBP) after all additions indicated that, the quality of gasoline was improved. But ASTM assigned that, the value of FBP could not exceed 250°C. This refers to probability of separation of kerosene at this temperature value.

Importance of distillation test of reformat gasoline after additions, is to enhance the octane number (ON). The additions of these selected materials with different concentrations showed that, the octane number was increased after the distillation and refining processes. Therefore, the distillation test gives an interpretation about the raising or lowering of the octane number only (Ezeldin and Massad, 2015). No limits had been assigned for the density and vapor pressure. Because they depend to a greater extent on the temperature of the country, but the refineries has assigned limits to these physical parameters as shown in Table 3.3.

Table 3.3. Limits of physical parameters gasoline assigned by Khartoum refinery

Test	Summer	Winter
Density	>740 kg|m^3	>740 kg|m^3
Vapour pressure	40- 67 KPa	40 – 85KPa

According to the assigned results of density and vapour pressure, the obtained results in Tables 3.1 & 3.2, appears to be in permissible range. The percent of gum in gasoline affect to the stability of gasoline, which can be a (+ve) or (–ve) effect on the octane number and the quality of the gasoline. This explain why ASTM has assigned boundaries to the percentage of the gum refer to Table (3.4). We find the gum existence after all additions stable(0.8 mg/100ml). The test of copper corrosion is very important regard to the gasoline, because this causes corrosion and fatigue in gasoline tank, the engine and also the exhaust (Twu *et al*., 1999).

ASTM has put a colour table which to determine the corrosion of a copper plate immersed in a gasoline sample, and the comparison with ASTM copper strip corrosion standard. The results obtained for bath addition is within permissible limits. The result (1a) of the copper corrosion indicated the good quality of the gasoline after the additions. We find the lead content of gasoline not altered after the additions and the lead content of reformate gasoline before additions(0.001) within permissible assigned by ASTM .

One of the most important properties which should be measured in order to assess the quality of gasoline, is the oxidation stability which give clue on the circumstances of the product storing, and also the heat required to start the combustion of gasoline inside the engine (Kerr and Richard, 2003). The results obtained from the oxidation stability test are within the permissible range of ASTM as shown in Table (3.4). The sulfur concentration after all additions was stable.

Table 3.4. The permissible range by ASTM

Test name	Permissible range	ASTM
Distillation	FBP ≤250◦c	D86-99a
Gum content	0.5-2mg/100ml	D381-99
Cupper corrosion	1a or 1b	D183-91
Oxidation stability	>240mints	D525-99a
Sulfur content	□250 ppm	D4294-89
Lead content	□ 0.001 ppm	D3341-91

The raises of ON 5.2 &1.5 with adding of aniline and toluene respectively (5%) to the reformat gasoline can be considered a clear indicator that the oil can raise the octane number and consequently improve the quality of reformat gasoline. As this addition is not accompanied with any rejected bad measurable properties, in contrast the addition of 10% aniline and toluene to the reformate gasoline raises the ON to 7.2 & 5.7.

The raising of ON and additions were attributed to the ability of the additives to convert the hydrocarbon chains into cyclic compounds or increasing the branched chains in gasoline, The effects of aniline and toluene concentrations on (MON) were shown in Fig 3.1 & 3.2 respectively.

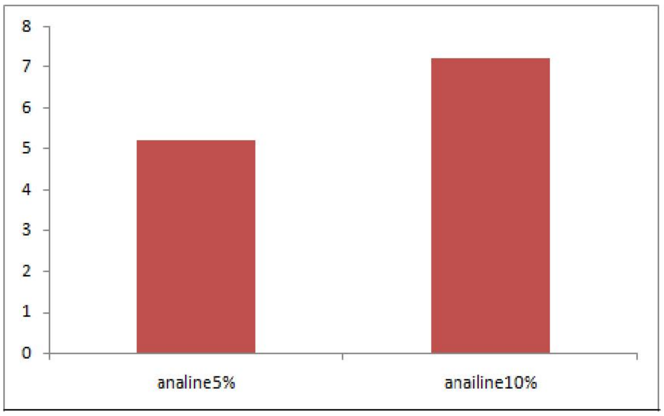

Fig. 3.1. Effects of aniline concentrations on MON

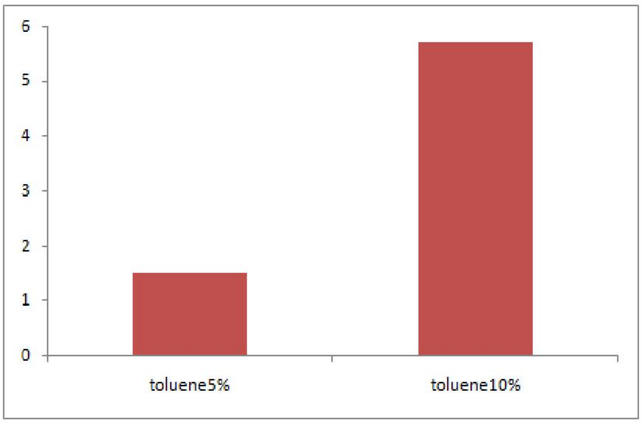

Fig. 3.2. Effects of toluene concentrations on MON

Conclusion

Based on the previously discussed analyses, results the following conclusions can be summarized as.

- The octane number of reformat gasoline used in this project was found to be 88.5.
- All additive materials used showed different abilities in improving ON of reformat gasoline produced from Khartoum Refinery.
- Aniline showed highest value of octane number. On the other hand, toluene showed least value of ON.
- The density and vapour pressure properties for reformat gasoline after all additions with different concentration were found within the range assigned by Khartoum refinery.
- Other properties for reformat gasoline after all additions with different concentration were found within the limits assigned by ASTM.

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