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Research Article

BIOFUEL PRODUCTION FROM PALM OIL (ELAEIS GUINEENSIS JACQ.) BY THERMAL CRACKING IN PLUG FLOW REACTOR

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A B S T R A C T

Given the need to develop and implement alternative renewable energy sources, this research was focused on using palm oil (*Elaeis guineensis* Jacq.) as a raw material for biofuel production. Abench-scale plug flow reactor was designed and built and it was then used to carry out the thermal cracking experiments aimed at bio-oil production. For each experiment, the bio-oil products were characterized according to the acid value, refraction index, viscosity, density and distillation curve. The results obtained from each experiment were compared with those for crude oil in order to identify the operation conditions that provide the best quality bio-oil. The bio-oil from each experiment was then fractionated using a distillation column, to produce bio-gasoline, bio-kerosene and greendiesel. The distillation products were also characterized, based on the same properties evaluated for the bio-oil, and the results were compared with those for gasoline and diesel fuels. The results of this study show that it is possible to produce a bio-fuel based on bio-oil obtained from the thermal cracking of palm oil using a plug flow reactor, and the product is similar to crude oil, with the exception of the acid index value. With regard to the distillation curve, when compared with those for crude oil (Hungo and Cabinda blends) and its derivatives, good approximations are observed. The thermal cracking of palm oil can therefore be used as a technological strategy to obtain bio-oil and its derivatives and thereby reduce the green house gas emissions from fossil fuels.

INTRODUCTION

The accelerated consumption of fossil fuel is associated with population increases around the world, stimulating the extraction of mineral and non-renewable resources to attend the energy needs. Thus, studies have been carried out to development alternative fuels, derived from renewable sources, such as biomass and waste from food processing (Chiarelo *et al*., 2020; Pitt *et al*., 2019; EIA, 2009; Rosa, 2017).

The literature reports scientific and technological research seeking alternative strategies using renewable sources, with environmental sustainability, to replacement of fossil fuels. Environmental sustainability involves increasing the energy yield while reducing the costs and environment impact, using renewable, efficient and sustainable technologies. The strategies described above offer a new perspective to solve current energy problems, not only through the use of renewable sources and but also minimizing energy needs.

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Thus, the sustainable use of biomass can reduce both environmental and economic problems. According to the International Energy Agency (EIA), biomass accounts for14% of the total primary energy used globally. The use of this type of energy reduces gas emission rates by 56%, when compared to emissions from fossil fuels (EIA, 2009; Rosa, 2017).

In this context, scientific studies have explored alternative approaches to fuel production, based on the use of biomass obtained through thermal cracking or thermal catalytic cracking. The processes cited above involve kinetic studies, based on understanding the relation between all parameters associated with production of bio-oil as well as fossil oil. The bio-oil produced was fractionated to obtain the bio-fuel, as bio-gasoline, bio-kerosene or green diesel, with the same characteristics as fuel derived from fractionated fossil oil. The fractionation process produces compounds that can be used as lubricant oil and bituminous asphaltic waste, which have the same applicability as the fossil oil product obtained in refineries (Wiggers *et al*., 2008; Botton *et al*., 2012), as shown in Figure 1.

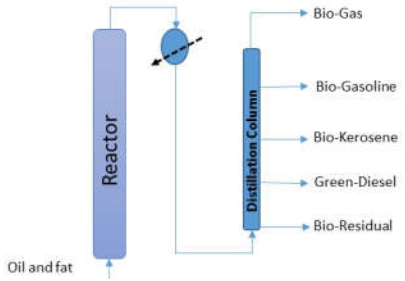


Figure 1 Characteristic of reactor used by Wiggers for cracking thermal

Wiggers *et al*. (2008) carried out catalytic cracking experiments applying arange of temperatures from 450 to 500ºC, using a glass vessel (250 mL). The temperature was measured at two positions using calibrated thermocouples. When the temperature in the reactor had reached 450–500ºC, the woody oil was introduced into the cracking reactor, with a constant pumped flow, and it was then pyrolysis and vaporized. The vapor leaves the reactor through a rectification column, with conventional metal packing at temperatures ranging from 330 to 340ºC. The vapor feed then enters a water-cooled heat exchanger. As a result, two liquid fractions are obtained in the collector, an aqueous fraction and an organic fraction. The organic phase was weighed to give the yield of organic liquid product (OLP) and analyzed by gas chromatography, size exclusion chromatography (SEC) and FTIR spectrometry. The residue in the reactor was weighed to give the coke yield.

Botton *et al*. (2012) performed cracking experiments for the thermal conversion of a frying oil and textile stamping sludge mixture in a continuous reactor (Figure 2). The textile stamping sludge was used to catalyze the thermal cracking reaction, to produce oil and the physical and chemical properties of the oil obtained were determined. A notable result was that the level of acidity index was around 12 mg KOH/g. The oil product of this process was fractionated providing a fuel fraction with the same characteristic as fuel obtained from fossil oil.

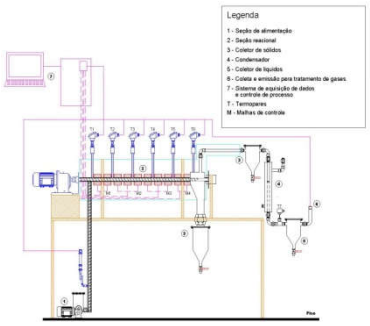


Figure 2 Continuous reactor used for the crackingoffrying oil and textile stamping sludge mixture.

Bottom *et al*. (2012) subjected the liquid products of thermal cracking, characterized as bio-oil, to fractionation to produce light and heavy phases that were analyzed and compared with afossil fuel, considering the distillation curves, based on the ABNT NBR 9619 norm. Chromatographic analysis was also carried out and the acid index value was determined. The distillation curve relates the volume recovered from distillation and the bubble point temperature of a mixture to determine the composition of a fuel. The parameters described above useful for characterizing fuels and determining the quality of bio-oils.

Bio-oil from thermal cracking can be obtained from different raw materials, as highlighted in a study by Beims *et al*. (2010), who performed the thermal cracking of soybean oil and blends of soybean oil with hydrogenated fat. Araújo *et al*. (2017) performed the thermal cracking of sunflower seeds. Wiggers *et al*. (2009) studied the thermal cracking of waste oils, including waste fish oil. Bottom *et al*. (2016) carried out thermal cracking experiments using the methyl esters in castor oil to produce heptaldehyde and methyl undecenoate and they observed a high acid index value. Trabelsi *et al*. (2014) pyrolyzed, under nitrogen, several animal-derived (lamb, poultry and swine) fatty wastes, in a laboratory-scale fixed-bed reactor and the liquid bio-oil, solid bio-char and syngas products were obtained. Chromatographic (GC–MS) and spectroscopic (FTIR) analysis of the bio-oil showed that it is a complex mixture consisting of different classes of organic compounds. Based on their properties, bio-oils are suitable for use as engine fuel or as a potential source of synthetic fuels and chemical feedstock. For all cases cited, the bio-oil produced had a high acid index value and the quality was dependent on the raw material and operating conditions used, notably the temperature and residence time (t_R).

In this study, a bench-scale plug flow reactor (PFR) was designed and built. It was then used to carry out the thermal cracking of palm oil. In the heating system, the combustion (non-condensable) gas was used as a heat source, flowing through piping in the equipment, contributing to the sustainability of the reactor process.

MATERIAL AND METHODS

Materials and Equipment

For this study, a PFR was designed, built and installed, in bench scale, with heating using combustion gas flowing internally in a jacketed system. This apparatus was used to carry out the experiments on the cracking of palm oil (*Elaeis Guineensis* Jacq.) for the production of bio-oil with same characteristic of petroleum oil. The bio-oil was then fractionated to produce bio-gasoline, bio-kerosene and green diesel and these fuels were characterized, showing the same physico-chemical characteristics as fossil fuels.

Experimental Apparatus

The apparatus was installed in a professional laboratory of ISPTEC and used to carry out palm oil cracking in thermal experiments. The unit in pilot scale is composed of the following: a) feed section; b) evaporation section; c) reactive section; and d) condensation and product recovery section. As shown in Figure 3, all sections are interlinked to form the experimental apparatus used in this study.

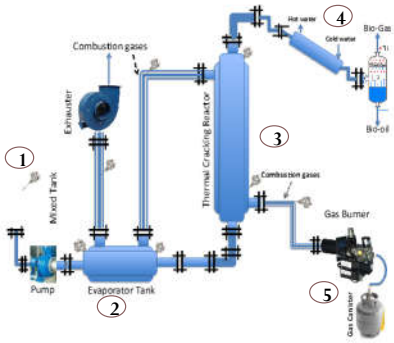


Figure 3 Reactor used to carry out the experiments.

The feed section of the apparatus is comprised of a tank installed at a height of 2 m in relation to the evaporator, to ensure the flow of the raw material through the action of gravity. This section also incorporates the liquid level control with a global valve for feed control.

The evaporation section is composed of 2 concentric cylindrical pipes, installed in the horizontal position, where the raw material flows in an internal pipe and the combustion gas flows in a jacketed system, located between the internal surface of the external pipe and external surface of the internal pipe. This section allows the evaporate draw material to vertically ascend to the reactive section.

The reactive section is formed by 2 vertical concentric cylindrical pipes, where the raw material from the evaporation section flows through an internal pipe where the cracking reactionoccurs. The combustion gas, flowing in a jacketed system, is used to heat the reactor. At both ends of reactor a PTK thermocouple was installed to measure the vapor and product temperatures.

The condensation section is comprised of 2 concentric cylindrical pipes, in series, separated by two flash tanks used to recover the reaction products.

The above-described apparatus was used to carry out the experiments, controlling the operational parameters to obtain the reaction products, in this case seeking a bio-oil similar to crude oil. The products from this process were characterized and the results compared with those for crude oil. The bio-oil was then fractionated to obtain products with the same characteristics as those obtained from crude oil. In the characterization, specific reagents were used and the physical and chemical parameters measured were correlated with the operation conditions applied during the experiments.

Bio-oil Fractionation

The bio-oil was fractionated, according to temperature, using a simple distillation column set up in laboratory scale, composed of a round bottom flask (500 mL), heating plate (model Nema-05, 1500 watts power), distillation column (Vigreux), 2 borosilicate Liebig condensers, beakers (100 mL) used to collect the products and a thermostatic bath with recirculation (model CORIO CD-200F, 220V and 1 kW).

The temperature was controlled using a PT100 thermocouple, placed inside in the top of distillation column, and the reading was carried out using a digital multimeter (model MM-2090). The round bottom flask and the distillation column were insulated with aluminum paper to minimize the thermal dissipation from the system to the external environment.

Material and reagents

Palm oil was mechanically extracted by pressing cooked palm fruits. The oil obtained in this operation was washed with hot water for approximately 3 h. After the phase separation, the lighter phase containing the oil was dried before characterization. The production of palm oil was carried out "*in loco*" at Esperança Farm located in the Kwanza Sul province of Angola (Gabriel *et al*. 2015).

The physical-chemical characterization of the palm oil was based on the acid index value obtained by titration with KOH solution, the viscosity determined using an Ubbelohd viscosimeter, the density obtained using an automatic densimeter and the refractive index determined with an Abbe refractometer. The FTIR spectra were acquired using a spectrophotometer (Thermo Scientific: Nicolet iS10). The spectra were recorded in duplicate for each sample, at room temperature (24-26ºC), with the aid of the Galactic Grams software package (Galactic Industries, Salem, NH, USA), in the wave number range of 12,000- 4,000 cm^{-1}, with a spectral resolution of 16 cm^{-1}.

Experimental Procedure

Experimental Cracking Reactor

The reactor operation was started by heating the apparatus using the combustion gas flow from the heating section, with the evaluation of the reactor dynamics, registering the temperature in relation to time. The data were collected using a PT100 thermocouple at three points of the reactor. The raw material was previously heated to reduce the viscosity and surface tension in the wall of the pipe.

To reduce the residence time, gas exhaust equipment was installed in the top of last flash tank, which promoted agradient pressure to ensure the product flow in the upward direction. Once the reactor was thermally stability, the feeding was started to ensure the experimental flow rate required. In this phase, the triglycerides of the raw material are broken through chemical reaction to form new components characterized as reaction products. The products formed pass through an outlet at the top of reactor to a condensation system and are recovered in one of the two flash tanks. The products were then collected and the volume registered prior to characterization.

Characterization of Cracking Products

The physico-chemical characteristics of the bio-oil and its derivatives obtained in the distillation were determined using the procedures described in technical norms, mainly the Brazilian Norms (ABNT) and those of the American Society for Testing and Materials (ASTM). The specifications established by regulations Nº 37/2009, 57/2011 and 65/2011 of the Brazilian National Petroleum Agency (ANP, 2011) were used to qualify the fuel proprieties. In this study, the density, viscosity (viscometer Schott-Garate), acid index value, degree of saponification and refraction indexes were considered in the evaluation.

Fractionation by cuts

The bio-oil cut points in the simple distillation column in laboratory scale to obtain the fraction bio-fuel in the temperature ranges of bio-gasoline, bio-kerosene and green diesel were determined, respectively, as $40ºC \leq T \leq 175ºC$, $175ºC \leq T \leq 235ºC$ and $235ºC \leq T \leq 305ºC$. The fractions obtained from the distillation were later characterized to determine their chemical and physical proprieties.

RESULTS AND DISCUSSION

Plug Flow Reactor Operation

In the reactor operation, the combustion gas flow was used as a heat source, with the support of an exhaustion system and contained in a jacketed reactor system to ensure the cracking reaction temperature. For the best operational system, the reactor was insulated with a glass lining to minimize the thermal dissipation on the rector surface and reach the

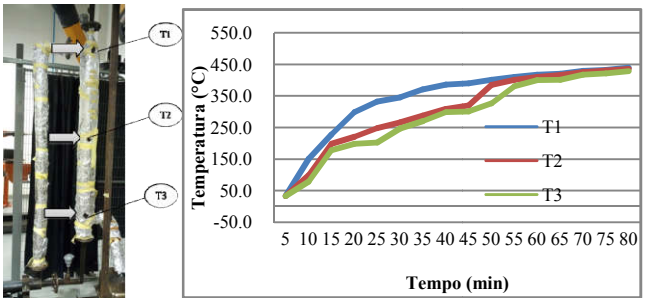
stationary conditions, and the temperature and time were rigorously monitored and registered.
Once the reaction temperature had been reached, the reactor was fed with palm oil, initially held in the feed tank. The transformation of raw material was started, followed by the vaporization, rising of the vapor to the reactor and product recovery in the condensation system.

During the conversion process, the palm oil was fed to the reactor without water, and under these conditions it was observed that the residence time was long and product recovery was low. To address this, when the raw material was in the feed tank, water was added in the same proportions as the biomass, leading to the formation of superheated vapor (water stream), which drags with it the products retained on the internal surface of the reactor, thus recovering the remaining bio-oil.

When the palm oil was mixed with water, providing the feed as a mixture, the product recovery rates were high. This verifies the effect of water on the yield in the thermal cracking process. This finding was also reported by Wiggers *et al*. (2009a and 2009b), who carried out the experiments in a continuum reactor and observed that the proportion of water in the mixture is a determinant factor, in terms of the yield obtained in the thermal cracking process.

Dynamic thermal reactor

The experimental tests revealed the temperature evolution with time, that is, the heating dynamics of the reactor. As shown in Figure 4, the reactor dynamics were determined at three points, after 55 min, after the start of thermal stability in the reactor observed from the ending curves, and during the stationary state step. In the last step, was appropriate to reactor feeding.



a) **b)**

Figure 4 Thermal dynamics of the reactor at time points 1, 2 and 3 (Experiments 1,2 and 3): a) localization point and (b) dynamic profile

Palm Oil Characterization

The characteristics of the palm oil, that is, density, kinematic viscosity, acidity, refraction index and oxidative stability, parameters also used to evaluate crude oil, are shown in Table 1,

Table 1 Characterization of palm oil

Oil	Density [kg/m^3]	Kinematic viscosity [mm^2/s] at 40°C	Acidity [mg KOH/g]	Refraction Index	Oxidative stability [h]
Crude palm oil (reddish color)	906.9	30.1	15 – 21*	1.461	15.4 ± 1.6

Source: Gabriel *et al*., 2015

The spectra in Figure 6 show bands typical of vegetable oils. The band at 720 cm^{-1}is attributed to –(CH_2)n- and -HC=CH- *cis* bending while those in the region of 1100 - 1170 cm^{-1} correspond to the vibrations of the C-CH_2-O group, the asymmetric stretching of C-O-C and C-C bond stretching. The intense peak located at 1745 cm^{-1}is associated with the carbonyl radical and is characteristic of esters. The band at 2852 cm^{-1} is related to the symmetric stretching of CH(-CH_2-) and at 2921 cm^{-1},which corresponds to the asymmetrical stretching of CH(-CH_2-) saturated bonds that are abundant in palm oil (Gabriel *et al*., 2017).

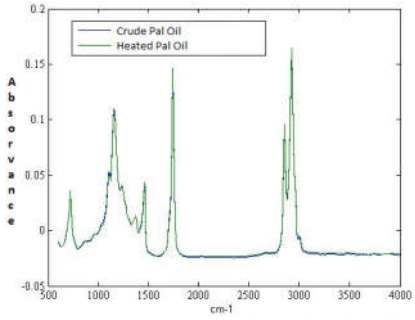


Figure 5 FTIR spectra for the crude palm oil.

Bio-oil Characterization

The bio-oil and its derivatives were characterized using the procedures described in this paper and the results are shown in Table 2. The viscosity and density values are in the same range as those for crude oil and its derivatives but the acid index value lies outside the range for these fossil products. Correia *et al*. (2014) carried out the thermal cracking of palm oil for bio-oil production and the product had an acid index value of 30.45 mg KOH/g, which was attributed by the authors to a lack of process stability. To minimize the acid index value, they proposed the implementation of complementary operations, mainly related to neutralization with a base solution or the use of vacuum distillation or thermal catalysis cracking.

Table 2 Characteristics of bio-oil obtained from palm oil and crude petroleum oil.

Parameters	Unity	Crude Oil (ANP)	Bio-oil
Acid value	mg KOH/g	0.5– 1.0	20.2
Viscosity at 40 ºC	mm²/s	2.0– 4.5	2.01
Refraction Index	-----	------	1.46
Density at 26ºC	g/cm3	0.86–0.94	0.81
Color and physical state	-----	dark brown	dark brown

Source: Jesus (2017)

Bio-Oil Distillation Curve

The distillation curve was used in the bio-oil characterization, and the results were compared with two national crude blends, Cabinda 32.2^{0}API and Hungo 30.3^{0}API, as shown in Figure 6.

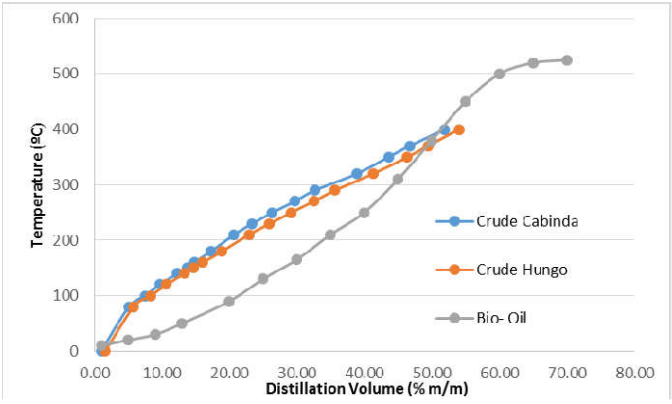


Figure 6 Bio-oil and crude oil distillation curves.

The maximum operation conditions were limited by the capacity of the instruments used in this study, but distillation curves of similar quality were observed for the three types of oil evaluated herein, due to the similarity of the molecular structure of the hydrocarbon compounds present in the oils.

In Figure 6, a good approximation between the distillation curve for the bio-oil and crude oil can be observed, as in the case of the results obtained by Wiggers *et al*. (2008). This performance is due the same structure of the hydrocarbons present in fuels evaluated in this study. Within the operational limits of the bio-oil experiments, as shown in Figure 6, this bio-fuel has components with a low bubble point than crude oil, as can be observed in the curve from the initial point up to50%. These results are associated with the operational conditions used to carry out the experiment on bio-oil production and the type of raw material used in this study, which shows a loss of conversion to bio-oil, and the limitations of the instruments employed in this research.

Bio-Oil characterization

In Table 3 the parameters used to evaluate the characteristics of the bio-oil are compared with those for petroleum from Angola (Hungo and Cabinda blends), used here to evaluate the distillation curve. It can be observed that the density and viscosity are in same range as the values for the two petroleum blends. However, the bio-oil does not contain sulfur compounds and therefore when it is used in the combustion process it does not produce sulfur gases, such as SOx. In relation to the acid index value, the behavior of this parameter is more complex in the thermal cracking process, as cited by many authors. For all cases found in the literature, this problem is considered to be the most complex to solve. Therefore, many authors suggest alternatives to minimize the acid index value, such as the use of catalysis during the thermal cracking process or neutralization of the thermal cracking product after the process.

Table 3 Physico-chemical characterization of the crude oil and green diesel.

Proprieties	Unity	Norm	Cabinda Blend	Hugo Blend	Bio-oil
Density at 15ºC	g/cm^3	EN ISO 12185	0.8637	0.8739	0.8589
Viscosity at 40 ºC	mm^2/s	EN ISO 3104	12.318	8.615	8.345
Acid Index value	mg KOH/g	ASTM D664	0.17	0.42	10.2
API Gravity 60/60ºF	----	EN ISO 12185	32.2	30.3	-----
Sulphur (Total)	%m/m	ASTM D4294	0.16	0.54	0.0

As seen in Table 3, the bio-oil parameters were compared with the Cabinda and Hugo crude oil and there was good approximation in terms of the density and viscosity values. The acid index values for the bio-oil are higher compared with the fossil fuels and this trend has also been reported by Wiggers *et al*. (2008, 2009) and Bottom *et al*. (2012).

Bio-fuel characterization

Table 4 shows the density of the bio-gasoline and greendiesel, for which the results are within the limits established by the Brazilian Petroleum National Agency (ANP) for fossil fuels, while the viscosity values are close to the limit. On the other hand, the acid values for these two fuels are over the limits defined by the ANP, and this parameter thus requires further study. Reports in the literature show that the acid index value can be reduced when catalysis is used in the thermal cracking process. In this regard, Santos (2015) and Correia *et al*. (2014) carried out experiments on the cracking of palm oil to obtain bio-oil for use in fractionation to produce bio-fuel. The authors reported that the acid values for the bio-fuels produced were higher than those obtained for fossil fuels.

The refraction index values for the bio-fuels obtained in this study are close to the limits established by the ANP for the assessment of fossil fuel quality. Ferreira *et al*. (2017) evaluated the density, viscosity and refraction index of bio-fuels obtained from bio-oil distillation, and the values for the parameters mentioned above were below the limits established by ANP, probably due the operational conditions applied in the study.

Table 4 Physico-chemical characterization of bio-gasoline and greendiesel.

Proprieties	Unity	Gasoline (ANP N° 57)	Bio-gasoline 40ºC ≤ T ≤ 175 ºC	Diesel (ANP N° 50)	Green Diesel 235 ºC ≤ T ≤ 305 ºC
Densityat 26^{0}C	g/cm^3	0.710- 0.780	0.775	0.820 – 0.860	0.827
Viscosity at, 40 ºC	mm^2/s	0.6	0.57	2.0 – 5.0	1.98
Acid Index Value	mg KOH/g	Write down	10.3	0.5 – 1	10.2
Refraction Index		1.42	1.44	1.46	1.43

Green Diesel Distillation Curve

The quality of the green diesel distillation curve was evaluated and compared with those for two fossil diesel samples, obtained from two national crude oil (Cabinda 32.2^{0}API and Hungo 30.3^{0}API). The similarity of the profiles of the three curves can be verified in Figure 7. In general, as the temperature increases, the liquid volume also increases, up to around 250-270ºC. After this point, thermal stabilization occurs in the volume range between 25 and 62 mL. The temperature then increases until the end of the process, reaching 380 ºC and 345 ºC for the two diesel simples and 340ºC for the green diesel.

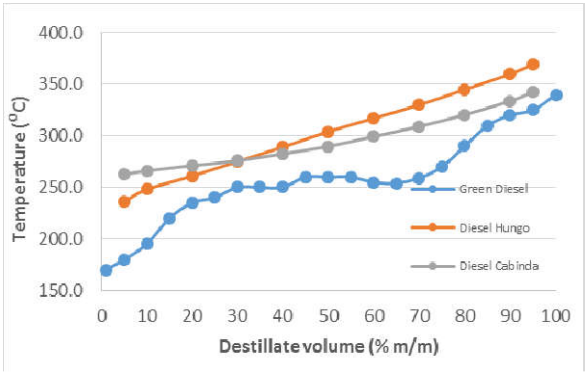


Figure 7 Distillation curves for greendiesel and two types of diesel obtained from crude petroleum.

The green diesel distillation curve shows a greater concentration of light hydrocarbons in the temperature range of 150-335ºC. Therefore, this bio-fuel with have a better performance in terms of combustion capacity than the fossil diesels. The instability observed for the green diesel can be attributed to instability in the experiments related to the heat dissipation by convection, mainly due to a deficiency in the insulation of the apparatus used to build the distillation curve.

CONCLUSIONS

Based on the results presented in this paper we can conclude that:

- The products resulting from the thermal cracking of palm oil have the same physico-chemical characteristics as crude oil, established by the ANP and data obtained

for Hungo and Cabinda crude oil, with the exception of the acid index value;

- The bio-gasoline and green diesel produced from the bio-oil distillation have physico-chemical characteristics in the ranges for gasoline and diesel established by the ANP and observed for fuels originating from Hungo and Cabinda blends, except for the acid index value;
- The bio-oil distillation curve, in the temperature range evaluated, showed a quality similarity to crude oil, but the hydrocarbons present in the bio-oil were lighter;
- The bio-oil profile is more similar to that of green diesel, when compared with fossil oil and fossil fuel. The distillation curves for bio-fuel and green diesel show lower temperatures than those for fossil oil and fossil fuel and could be improved by optimizing the process;
- The reactor developed in this study for the thermal cracking of palm oil was heated by combustion gases (coking gas) and the combustion was carried out using a Bunsen burner with a good support. This is one of the best alternatives for this type of study.

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