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WASTE MANAGEMENT

PHYSICOCHEMICAL ANALYSIS OF LIQUID FUELS OBTAINED IN THE PYROLYSIS PROCESS OF VULCANIZED RUBBER

ANÁLISIS FISICOQUÍMICO DE COMBUSTIBLES LÍQUIDOS OBTENIDOS EN EL PROCESO DE PIROLISIS DE CAUCHO VULCANIZADO

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Abstract

Analysis of the pyrolytic fuel (liquid fuels obtained from the pyrolysis process of used tires), the physicochemical characteristics correspond to gasoline, kerosene and diesel fractions. To determine the environmental sustainability of its use, combustion tests were developed in a diesel test engine (800 and 2500 rpm), in order to establish the comparison with a commercial ecuadorian diesel. The obtained results proved based on the analysis of emissions and combustion failures that this fuel cannot be a direct replacement for diesel.
Keywords: Rubber, tire, pyrolysis, fuel, combustion, emission, pollutant.

Resumen

En el análisis del combustible pirolítico (combustibles líquidos obtenidos a partir del proceso de pirólisis de residuos de neumáticos usados), las características fisicoquímicas corresponden a fracciones de gasolina, queroseno y diésel, para determinar la sustentabilidad ambiental de su uso, se desarrollaron ensayos de combustión en un motor de pruebas a diésel (800 y 2500 rpm), para establecer la comparativa con un diésel ecuatoriano comercial, los resultados obtenidos comprueban en base a los análisis de emisiones y fallos de combustión, que dicho combustible no puede ser un reemplazante directo del diésel.
Palabras claves: Caucho, neumáticos, pirólisis, combustible, combustión, emisión, contaminantes.

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1 Introduction

The environmental impact of unused tires is that their waste cannot be easily recycled, making it an unusable waste. Inadequate final disposal negatively influences environmental quality and human health (Cano, Cerezo & Urbina, 2007).

In Ecuador, millions of tires are discarded annually, of which a minimum percentage is reused for retreading, but the vast majority is incinerated or deposited in open-air garbage dumps, which poses a threat for the environment (El Telégrafo, 2013).

In general, the national problem is very different in both large and small population centers. In response, the Ministry of the Environment (MAE), issued with Ministerial Agreement No. 20, the Integral Management Plan for Used Tires, establishing as an opportunity the energy valorisation to obtain fuels, applying technology such as pyrolysis, thereby reducing the amount of waste that would enter a landfill thus increasing its useful life.

The use of used tires in energy recovery processes represents a short-term opportunity to consume large quantities in a safe and environmentally friendly way. Fuels derived from tires have also gained merit to increase thermal efficiency, reduce emissions and provide fuel users with a significant reduction in costs (Astafan, 1995).

1.1 Energy utilization of waste of used tires

The energy use or energy assessment, aims to extract the calorific value of used tires, in order to replace part of the conventional fuels. The calorific value of the tires is 35 MJ/kg, reason why this is an excellent fuel that allows to save between 10-12 kg of coal, or 7.52 kg of oil for each tire used as fuel (Aguado Alonso, 2010).

Pyrolysis is based on thermal decomposition of organic matter or carbon-based compounds at oxygen concentrations low enough not to produce combustion (Aguado Alonso, 2010), the pyrolytic oil and gases obtained from this process can be used directly as fuels (Williams, Besler, & Taylor, 1990), while coal can be used as solid fuel, activated carbon or carbon black (Comstock, 1980).

The optimum process temperatures range from 250 to 350 ° C and the distribution of the products depends on the starting material and the operating conditions. At higher temperature and residence time, the higher the gas yield (Murillo, 1999).

1.2 Characterization of fuels

The characterization of a fuel consists of identifying its physical and chemical properties through various laboratory tests. It is important to know these parameters because the design of fuel tanks in cars, pumps and pipelines are based on the physical and chemical properties of the fuel to be used, to avoid wear, losses by evaporation, as well as pressure drops (Camarillo Montero, 2011).

1.3 Combustion test in diesel engines

Among the main combustion tests to which an engine is subjected are the emission of combustion gases and failures of the combustion process.

The analysis of combustion gases consists of measuring the amount of pollutant gases that the engine emits into the atmosphere. The following gases are usually analyzed: Carbon Dioxide (CO_2), Carbon MoNO_xide (CO), Nitrogen Oxides (NO_x), Sulfur Dioxide (SO2), and Non-Burned Hydrocarbons (HC) and Opacity of Fumes (Serrano & Carranza, 2005).

In Ecuador there are several standards, highlighting the Standard of Air Emissions from Fixed Sources of Combustion in book VI Annex III of the Unified Text of Secondary Environmental Legislation (TULSMA), which establishes the maximum permissible emission levels of Exhaust gases from internal combustion engines.

Failures of the combustion process are necessary for reliable and safe operation of the machinery, the risk of failure and the time an engine is out of service can be reduced only if potential problems are anticipated and avoided. Therefore, one of the tools used to analyze the flaws in the combustion process is the measurement and analysis of noise and vibration in internal combustion engines (Tapia, 2006).

The most widely used vibration and noise amplitude descriptors are:

RMS value: The average value represents an estimate of the energy content in the vibration or noise of a machine, in the vibration analysis this value is used to quantify the severity of the vibration (Díaz, 2011).

Crest Factor: Equals the amplitude of the peak of the waveform divided by the RMS Value, in the analysis of vibrations allows a quick idea of what type of impact is occurring in the shape of the wave and is associated with the wear of the bearing of the rollers, cavitation and wear of gear teeth (Friedman, 2004).

Kurtosis: The kurtosis and crest factor represent high value statistical parameters in the detection of faults, since for the normal case the signal that is observed by means of an accelerometer has a normal distribution and when some of its elements begins to fail, it tends to move away from it, in these cases the crest factor has to increase taking higher values (Bendat, 1986).

2 Methodology

2.1 Experimental conditions of the pyrolysis process

The experimental pyrolysis work was carried out in a stainless steel batch reactor and in the complete absence of oxygen. The variables studied were pressure, temperature and retention time; with the purpose of determining the ideal conditions for the greater production of liquid fuels.

These results were analyzed with the MINITAB statistical package.

2.2 Physicochemical characterization of liquid fuel

For the physicochemical characterization the following methods were used:

a) Venezuelan regulations COVENIN 2052-93, "Products Derived from Petroleum. Determination of Density and Relative Density of Liquids by the Binghman Picnometer ", to measure density.

b) The Ecuadorian Technical Standard INEN 808: 1986 "Petroleum Products. Determination of Inflammation and Combustion Points in Cleveland Open Cup ", to determine the Flash Point

c) The Ecuadorian Technical Standard INEN 810: 1986 "Petroleum Products. Determination of Kinematic and Dynamic Viscosity in Transparent and Opaque Liquids ", in the determination of the viscosity.

d) Ecuadorian Technical Standard INEN 0926. "Petroleum Products. Distillation tests

e) Ecuadorian Technical Standard INEN 1489: 99 "Products Derived from Petroleum - Requirements. Diesel".

f) The SHATOX oil quality meter model SX-300 which complies with ASTM D 4737-03, ASTM D 613 and ISO 5165, for measuring the number of Cetane, solidification temperature and kerosene content.

g) A THERMO SCIENTIFIC gas chromatograph, model Trace GC ULTRA, with FID detector (Flame Ionization Detector), belonging to the Life Sciences Laboratory, to determine HAP's (Polycyclic Aromatic Hydrocarbons).

2.3 Experimental conditions of the combustion test

For the tests based on the Ecuadorian Technical Standard INEN 2 202: 2000 (Environmental Management. Air. Motor Vehicles, Determination of Opacity of Diesel Engine Exhaust Emissions by Static Test, Free Acceleration Method), an Internal combustion engine, Diesel ENGINE Diesel Model G-130301 of 2500 CC was used.

To measure the concentrations of combustion gases, the EUROTRON meter GREENLINE 4000 was used, which allows the reading of concentrations of the gases: Carbon MoNO$_x$ide (CO), Nitrogen Oxide (NO), and Nitrogen Oxides (NO_x); In real time during the combustion process.

Concentrations of opacity and particulates emitted by the fuels at the time of combustion were used to test diesel exhaust gases MAHA model MET 6.2.

Finally, for the noise and vibration analysis, a unidirectional piezoelectric microphone of the brand PCB, model HT378B02, and a submersible accelerometer model 8042 were used, the obtained signals were taken to an A3716 vibration analyzer of the brand ADASH, which has the function to condition the signals and allow the visualization of the magnitudes of sound pressure and vibrations emitted by the engine.

3 Results and discussion

3.1 Optimization of the pyrolysis process

In order to establish the factors influencing the performance of the liquid fuel production process, it is verified if the data shown in Table 1 present a normal behavior, necessary condition to apply the design completely at random.

For the use of this model, it is considered as an input factor (IF) the Temperature and as the output factor (OS) the Net Volume, the resulting p-value is 0.010 being this a value less than 0.05 with a 95% confidence level, it is shown that the temperature variable significantly influences the experiment.

Considering with IF, the Pressure variable yields a p-value of 0.688 and with the IF Residency Time, the p-value = 0.607, as evidenced, the values are greater than 0.05 which shows that they are not statistically significant by which they were discarded in the experiment.

In addition, a polynomial regression is performed with the data in Table 2, in order to identify the maximum point of the curve (see figure 2) and determine the optimal temperature (x-axis), to obtain the largest liquid volume (Y-axis).

Based on a fourth-degree polynomial regression, the following equation is obtained:

$$y = -1E - 07x^4 + 0,0001x^3 - 0,0678x^2 + 14,815x - 1206,4 \quad (1)$$

Considering this equation the maximum critical point of temperature is calculated that is 376 °C, which produces the largest volume of liquid fuel.

3.2 Physicochemical characterization of fuel

The obtained physicochemical analyzes of the liquid fuel determine that it possesses specific characteristics of fuels derived from petroleum (gasoline, diesel and kerosene); The physicochemical properties of the pyrolytic fuel are described in Table 3.

The pyrolytic fuel, possesses color and odor characteristic of petroleum products; the density and the gravity API correspond to a medium crude, according to the Venezuelan standard COVENIN 2052-93; Its viscosity corresponds to a diesel type I, based on the Ecuadorian Technical Standards (NTE) INEN 810: 1986 and INEN 1489: 99; The flash point is in the range of gasoline according to NTE - INEN 808: 1986; The solidification point, the Cetane number and the kerosene content are determined by the SHATOX model SX-300 which complies with ASTM D 4737-03, ASTM D 613 and ISO 5165 and INEN 1489: 99, corresponding results to a type II diesel; for the distillation process is based on the methodology of INEN 0926; The 90% distillation temperature obtained corresponds to a type II diesel, according to INEN 1489: 99.

3.3 Characterization of Polycyclic Aromatic Hydrocarbons (HAP's)

From the analyzes in the gas chromatograph it is established that of the six HAP's analyzed whose concentration results are observed in Table 4, Fluorantene and Benzo (a) pyrene, are the ones that are in greater proportion within the chemical composition of the pyrolytic fuel, the volume percentage of the six analyzed HAP's corresponds to 1.49% of the liquid fuel.

3.4 Comparison of the combustion process

Emissions of Nitrogen Oxides (NO_x, NO), Carbon MoNO$_x$ide (CO), Particulate Material, Opacity. And to establish the failures in the combustion process are analyzed: noise and vibrations; This comparative test is set at 800 rpm and 2500 rpm of the engine.

3.4.1 NO_x, NO and CO emissions at 800 rpm

The information on the test at 800 rpm shows us that commercial diesel emits 96% more NO_x than pyrolytic fuel and 109% more NO. The pyrolytic fuel emits 261% more CO than the commercial diesel, as shown in Figure 2.

With the data obtained in the measurements detailed in figure 2, the units of the reference standard of Annex 3 (TULSMA) are processed, the value obtained for NO_x is 119.03 mg m3 lower than the value established in the standard (2300 mg/m3) and in terms of CO and NO emissions, there is no applicable Ecuadorian regulation see Table 5.

3.4.2 NO_x, NO and CO emissions at 2500 rpm

In the 2500 rpm test (See Figure 3) the process fuel emits 98% more Nitrogen Oxides (NO_x), 98% more Nitrogen MoNO$_x$ide (NO), and 276% more Carbon MoNO$_x$ide (CO) than commercial diesel.

Table 1. Table 1. Performed Pyrolysis tests

Temperature (°C)	Pressure (Psi)	Tiempo de Retention (min)	Volumen Liquid (cm^3)
340	55	10	4,51
340	15	10	4,72
340	55	20	4,5
340	15	20	4,82
440	55	10	4,26
440	15	10	4,16
440	55	20	4,42
440	15	20	4,28

Table 2. Tests to determine higher fuel efficiency

Temperature (°C)	Liquid Volume (cm^3)
280	5,03
310	5,73
340	6,26
370	6,93
400	6,02

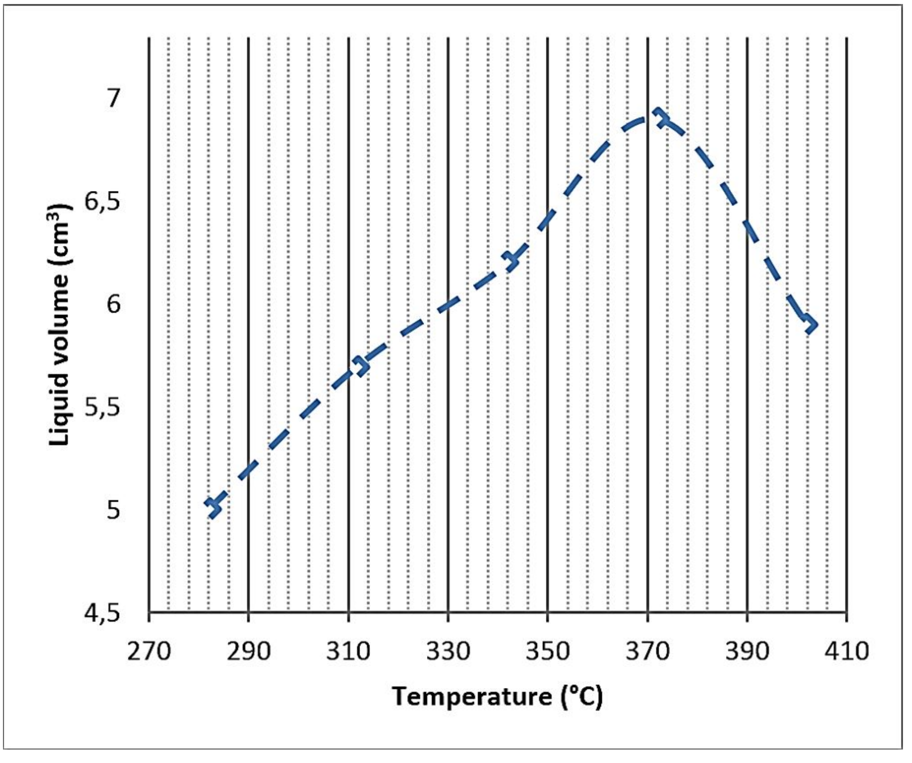

Figure 1. Optimum Temperature Calibration Curve

Table 3. Physicochemical characteristics of the obtained fuel

Parameter	Unit	Value
Density	g/cm^3	0,88
API gravity	-	23,87
Viscosity	Centipoise	1,11
Flashpoint	°C	20,5
Freezing point	°C	-39,3
Cetane Number	-	46,7
Destillation temperatura of 90 %	°C	320
Kerosene percentaje	%	45,4

Table 4. Concentration of HAP's in fuel

HAP's	Concentration	
	ppm	% v/v
Fluorantene	6089,48	0,61
Benzo (b) fluorantene	441,04	0,05
Benzo (k) fluorantene	0	0
Benzo (a) pyrene	4892,56	0,49
Indeno (1,2,3,-c,d) pyrene	2100,6	0,21
Benzo (ghi) perylene	1376,31	0,14
TOTAL	**14900**	**1,5**

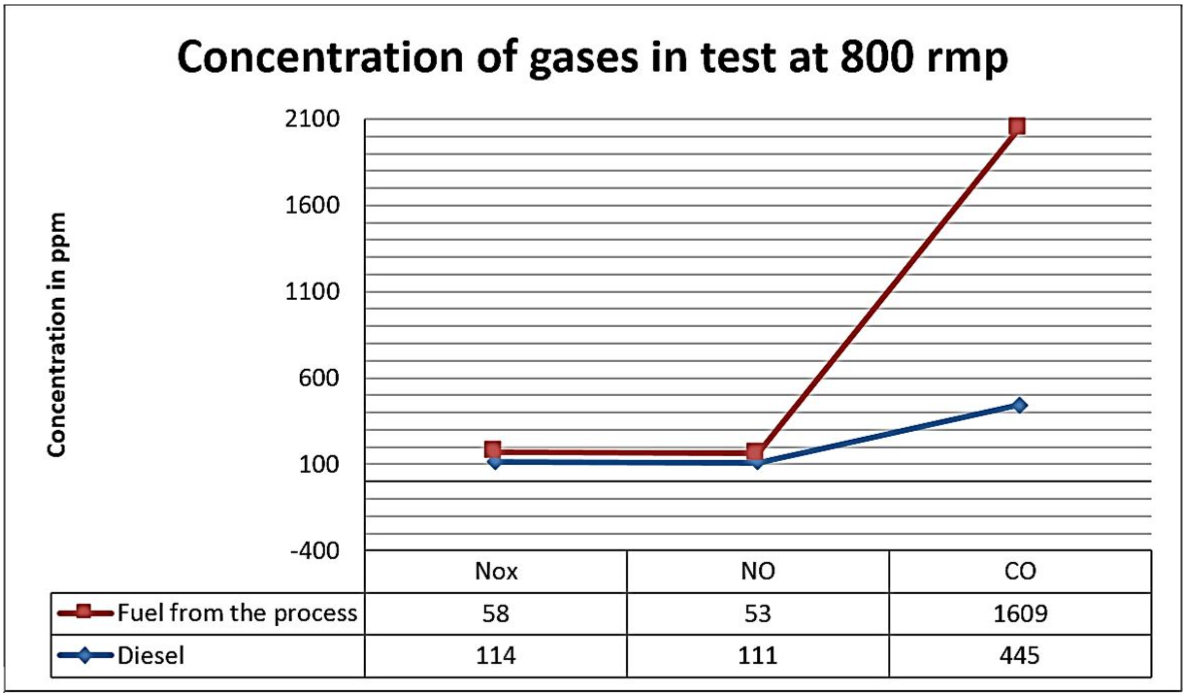

Figure 2. Concentration of gases at 800 rpm

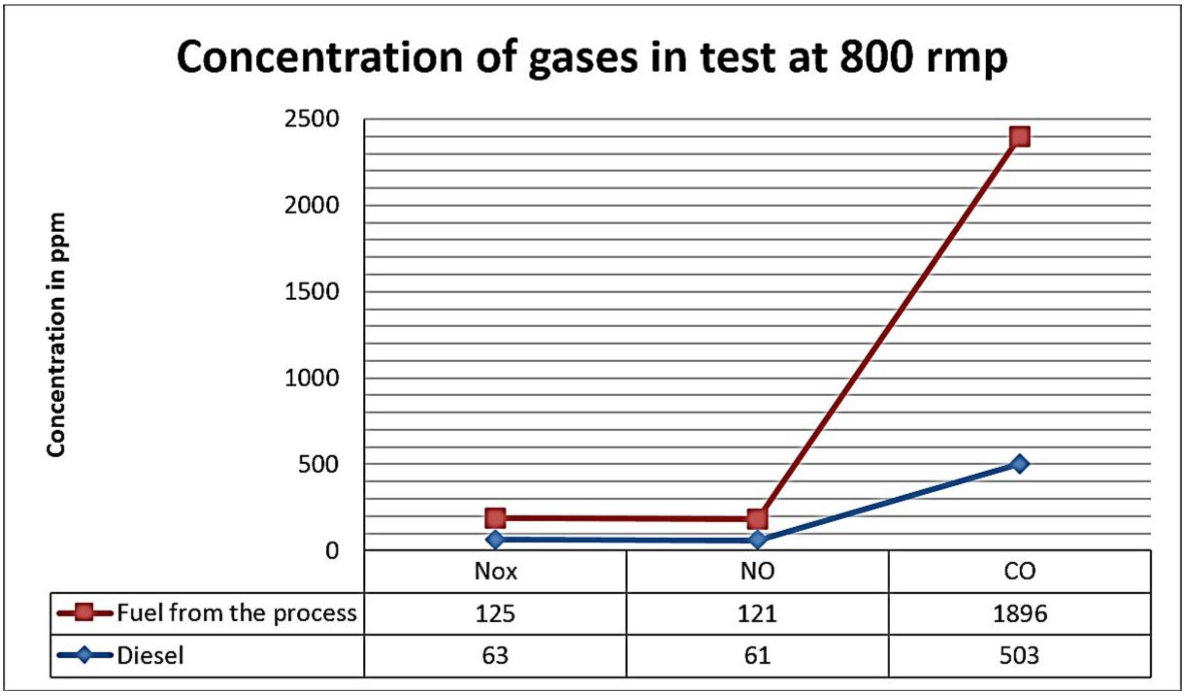

Figure 3. Concentration of gases at 2500 rpm.

With the data obtained in the measurements detailed in figure 4, the units of the reference standard of Annex 3 (TULSMA) are transformed, the value obtained for NO_x is 256.54 mg/m3 lower than the value established in the standard (2300 mg/m3) and in terms of CO and NO emissions, there is no applicable Ecuadorian regulation see Table 5.

3.4.3 Emission of particles at 800 rpm and 2500 rpm

For the particulate emission test the comparison is made with the commercial diesel at 800 rpm and 2500 rpm, taking measurements with the MAHA diesel model 6.2 diesel exhaust gas analyzer.

Figure 4 shows the particle concentration at 800 rpm, and it is observed that the emission of particles from the pyrolytic fuel is 588% higher than the diesel.

The pyrolytic fuel compared to diesel at 2500 rpm, as shown in figure 5 has a 4629% greater percentage than the commercial diesel one.

According to the analysis of the NTE INEN for Fixed Sources of Combustion in the maximum permissible emission limits for internal combustion engines, the fuel of the pyrolysis process at 800 rpm is within the limits established for new sources, and at 2500 Rpm exceeds the set value by 913%, as observed in Table 6.

3.4.4 Opacity at 800 rpm and 2500 rpm

In the results for opacity, in which diesel and pyrolytic fuel are compared, the two are within the limits established by the Ecuadorian Technical Standard INEN 2 202: 2000.

In the tests of 800 rpm the opacity of the obtained fuel is greater 538% more than the opacity of the diesel, as indicated in Figure 6.

For the tests of 2500 rpm the opacity of the obtained fuel is 2589% greater than the opacity of the diesel, as shown in Figure 7.

For the opacity analysis, a diesel exhaust gas analyzer MAHA model MET 6.2 was used, which determines that the k factor of the pyrolytic fuel is 0.134, equivalent to 1.3% of opacity, which represents a higher index in Comparison of the diesel factor k which is 0.021 equivalent to 0.2% opacity, for the data obtained at 800 rpm, see Table 7.

Also, for 2500 rpm the k factor is 3,765 which is equivalent to 37.6% opacity, therefore it is a higher index compared to the diesel factor k of 0.14 corresponding to 1.4% of opacity, fulfilling with the NTE

Table 5. Concentrations of pollutants in the pyrolytic fuel

Contaminant	TULSMA	Comb. Pirolytic (800 rpm	Pyrolytic fuel 2500 rpm	Units	Obs.
Nitrogen oxides	2300 mg/m^3	81,72	175,99	mg/m^3	complies
Nitrogen moNO$_x$ide	-	53	121	ppm	There is no limitation
Carbon moNO$_x$ide	-	1609	1896	ppm	There is no limitation

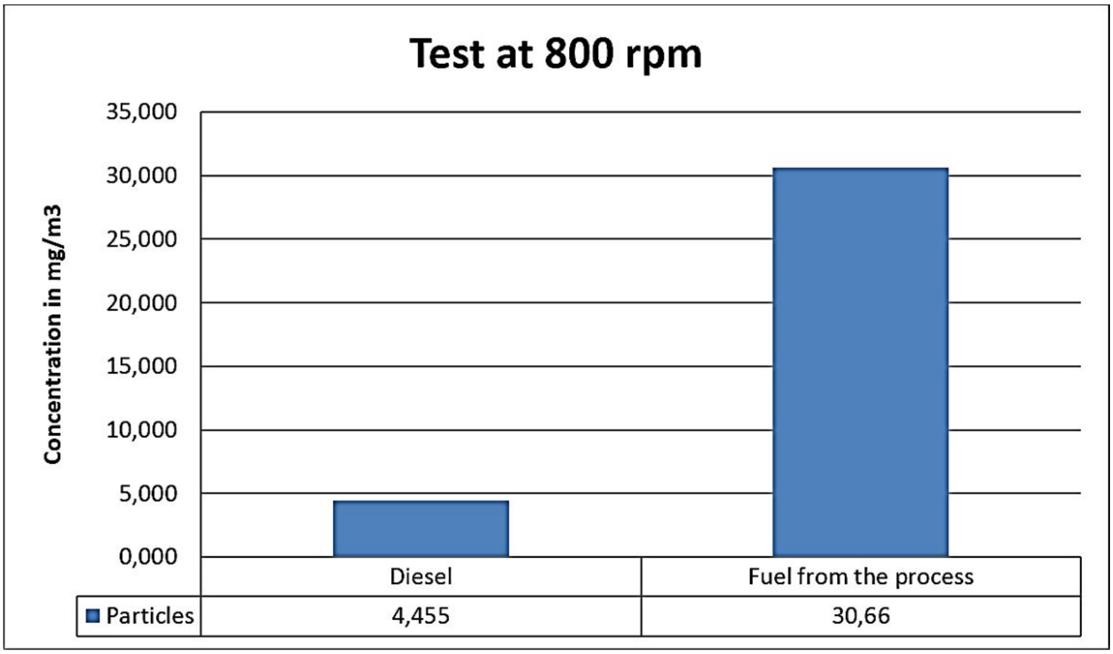

Figure 4. Particle concentration at 800 rpm

Table 6. Concentrations of pollutants in the pyrolytic fuel

Pollutant	INEN standard	Pyrolytic fuel	Unidad	Observacition
Total particles (800 rpm)	150	30,66	mg/m^3	Cumply
Total particles (2500 rpm)	150	1520	mg/m^3	Does not cumply

Table 7. Concentrations of pollutants in the pyrolytic fuel

Pollutant	INEN standard	Diesel	Comb. Pyrolyitic	Unit	Observation
Opacity (800 rpm)	50	0,2	1,3	%	Cumplies
Opacity (2500 rpm)	50	1,4	37,6	%	Cumplies

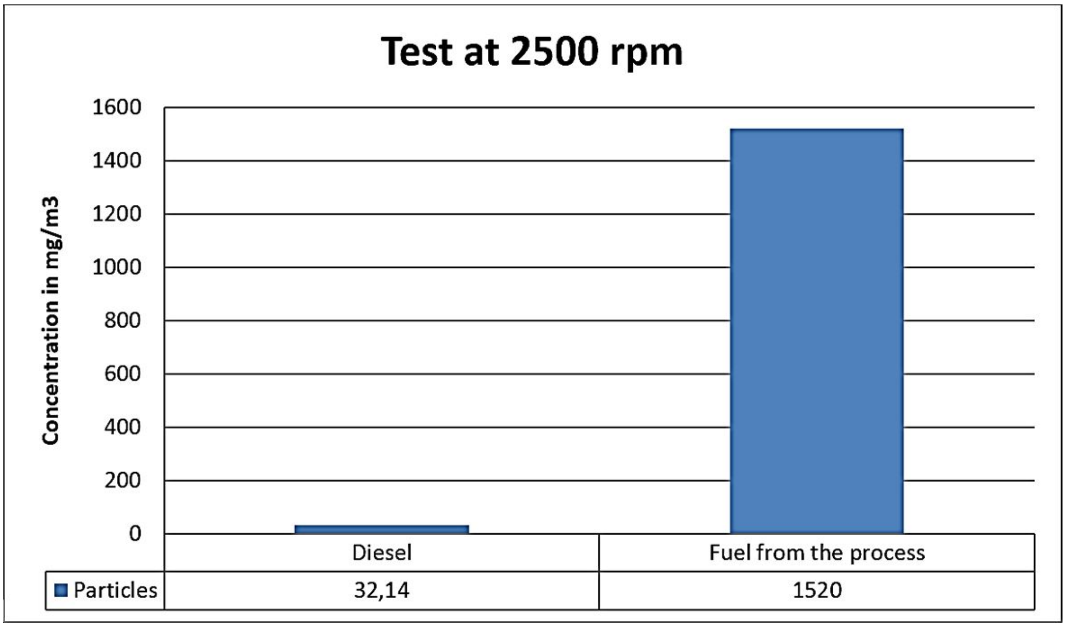

Figure 5. Particle concentration at 2500 rpm.

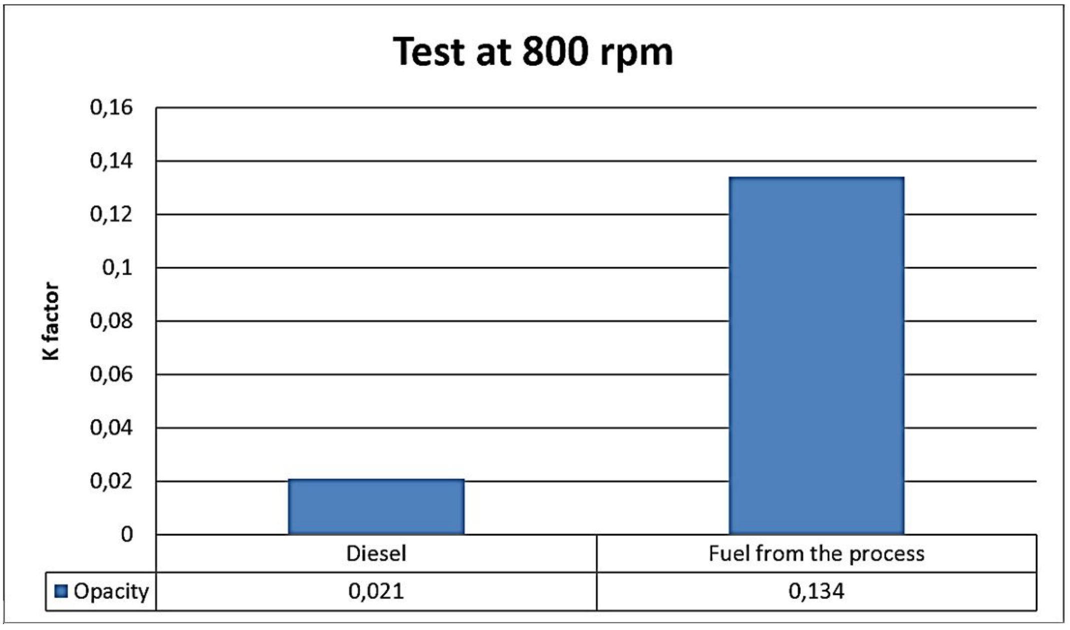

Figure 6. Opacity at 800 rpm.

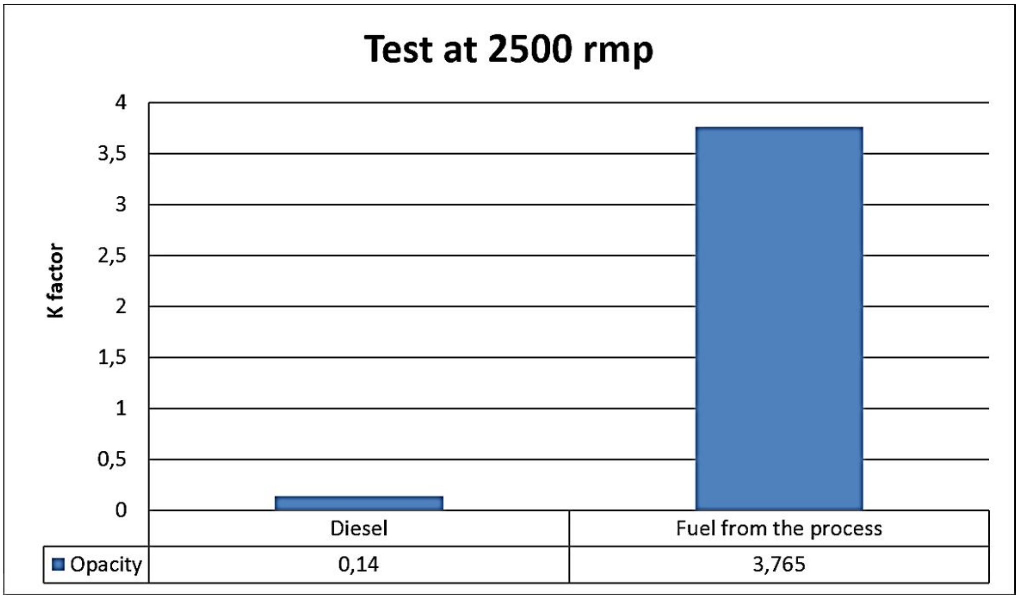

Figure 7. Opacity at 2500 rpm.

- INEN 2 202: 2000, as can be seen in Table 7.

3.4.5 Failure analysis of the combustion process

The analysis of the failures in the combustion process is performed to determine the effects of the fuel in the operation of the engine, using a piezoelectric microphone of the brand PCB model HT378B02 and a submersible accelerometer model 8042. The obtained signals were taken to an ADIX brand A3716 vibration analyzer.

Combustion noise at 800 rpm Table 8 shows the values of kurtosis and the crest factor of the pyrolytic fuel, which are higher than those of the diesel, so that variations in the amplitude of the noise occur, whereas the RMS value that shows us that the concentration of the energy of sound are appreciably equal.

Combustion noise at 2500 rpm In this case the kurtosis factor and the crest factor of the pyrolytic fuel are lower than the values of the diesel, which means that there are no significant variations in the amplitude of the noise, while the RMS value is higher than the fuel obtained, which means higher energy contained in the noise and therefore higher noise, this can be seen in Table 9.

Vibrations in the engine at 800 rpm In Table 10, it is shown that the kurtosis factor and the crest factor of the pyrolytic fuel are higher than those of the diesel, which means that the engine with the use of the fuel will show serious wear of the rollers; the crest factor is relatively high due to the number of impacts occurring inside the bearing, which will lead to wear of the roller bearings, cavitation and wear of the gear teeth.

In addition, the RMS value of the pyrolytic fuel is higher than the diesel, which means that the energy content in the vibration is greater with greater severity of the vibration.

Vibrations in the engine at 2500 rpm As shown in Table 11, the kurtosis factor and the crest factor of the pyrolytic fuel are higher than those of diesel, which means that when used, it will exhibit serious wear on the rollers, the crest factor is relatively high which will result in wear of the roller bearing, cavitation and wear of the gear teeth. The RMS value of the pyrolytic fuel is higher than that of the diesel, therefore, the energy content of the vibrations is higher and its severity increases.

Table 8. Noise signals at 800 rpm

Characterization of noise signals at 800 rpm		
Signal characteristics	**Diesel**	**Pyrolytic fuel**
Kurtosis factor	2,3078	2,6506
RMS value	0,002	0,0021
Crest factor	3,1681	3,4113

Table 9. Noise signals at 2500 rpm

Characterization of noise signals at 2500 rpm		
Signal characteristics	**Diesel**	**Pyrolytic fuel**
Kurtosis factor	2,4	2,3806
RMS value	0,01	0,0111
Crest factor	3,131	3,0382

Table 10. Vibraciones a 800 rpm

Characterization of vibration signals at 800 rpm		
Signal characteristics	**Diesel**	**Pyrolytic fuel**
Kurtosis factor	21,97	39,1095
RMS value	0,006	0,0081
Crest factor	15,58	20,2924

Table 11. Vibraciones a 2500 rpm

Characterization of vibration signals at 2500 rpm		
Signal characteristics	**Diesel**	**Pyrolytic fuel**
Kurtosis factor	11,28	16,1305
RMS value	0,034	0,0348
Crest factor	9,778	15,2128

4 Conclusions

After the tests of emissions of gases and particles is determined that diesel presents better results in combustion, showing values well below the emissions of the Pyrolytic Fuel, in which also it is observed an excessive generation of white smoke, symptom of anticipated combustion in the chamber.

When comparing the combustion of pyrolytic fuel and commercial diesel, it is established that under idle conditions at 800 rpm of the engine, the two fuels are within the maximum permissible limits of the Ecuadorian regulations for fixed sources of combustion in the emission of NO_x and particles.

In the process of combustion at an acceleration of 2500 rpm of the engine, it is determined that the emission of NO_x is below the maximum permissible limits.

The percentage of Opacity of the two fuels complies with the NTE INEN 2 202: 2000 standard.

From the results of failures of the combustion process through the analysis of noise and vibrations, in tests at 800 rpm and 2500 rpm, it is determined that the engine presents more difficulties in the combustion process of the pyrolytic fuel in comparison to the commercial diesel, finding greater noise and vibrations that generate a high wear in the engine parts.

Based on the analysis of emissions of the pyrolytic fuel with the Ecuadorian Standard for Fixed Sources of Combustion, it is observed that for the emission of NO_x it complies with the established permissible limits, while for CO and NO emissions, there is no limit established in the normative.

After analyzing the results, it is demonstrated that the sustainability of the pyrolysis process does not constitute a methodology for the management of residues of vulcanized rubber, by the analysis of the combustion test in the diesel engine. This because the fuel obtained from the process has higher emissions of pollutants (CO, NO, NO_x, Particles and Opacity) and combustion failures that affect the engine.

It is determined that the fuel obtained in the pyrolysis process has characteristics similar to those of diesel. But by combustion tests it is established that this cannot be a direct replacement of the commercial diesel.

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