CHARACTERISATION OF PYRO OIL FROM TYRE PYROLYSIS FOR ENERGY CONTENT

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Abstract

Disused tyres present formidable problems in terms of disposal and reprocessing, as they are almost immune to biological degradation. Also, the indiscriminate discarding and stockpiling of disused tyres is a challenge to safety and environment. Pyrolysis of scrap tyre to obtain pyrolysis by-products such as pyro-gas, pyrolytic oil and solid residue for useful purpose is a way of ameliorating these problems. In this report, characterisation of liquid product (pyro oil) from the pyrolysis of disused tyres was carried out. Shredded samples of the tyre (in 1 kg batches) were pyrolysed in a refurbished 17.4 litre capacity fixed-bed batch thermochemical reactor at varying temperatures of 250^{o}C, 350^{o}C, 450^{o}C and 600^{o}C with four batched per temperature respectively, and the percentage yield of oil was calculated. Proximate and ultimate analyses of the raw tyre sample and the oil resulting from the pyrolysis were carried out for the determination of the volatile composition, fixed carbon, moisture content and ash content. Fuel properties (heating value, density, kinematic viscosity and flash point) of the oil resulting from pyrolysis were determined, and compared with those of conventional diesel. Finding of the study showed that the pyro oil has a higher heating value than the raw sample, and close to that of conventional diesel, suggesting it as a viable choice of alternative fuel for energy production.

Keywords: *Disused tyre, heating value, liquid fuel, pyrolysis, thermochemical reactor*

1. INTRODUCTION

The increasing industrialization and the attendant usage of automobile cars and trucks globally have led to a steep rise in the number of waste tyres generated around the World. This coupled with the negative environmental impact caused by disposal of used automobile tyres in landfills or by incineration is of great concern (Akinola and Adewole, 2015). Waste tyres can be used directly as fuels in the incinerators (Caballero *et al.*, 2004). Due to their high heating value, scrap tyres are excellent materials for energy recovery. The use of tyres directly as fuel in incinerators has the following advantages: Reduced power-production costs, maximum heat recovery, and environmentally acceptable process. The disadvantages are: no material recovery, large capital investment, need for flue gas cleaning, CO_2 emission, and high operating costs. Scrap tyres are used also as fuel in cement kilns (Juan *et al*., 2001).

Pyrolysis is an endothermic process that induces the thermal decomposition of feed materials without the addition of any reactive gases, such as air or oxygen (Ejim *et al*, 2007). It is the thermochemical conversion process of biomass or other organic matters into primarily liquid (oils) and solid (char products) and some gaseous (methane, carbon monoxide, carbon dioxide, and other organic compound) products in the absence of oxygen (Akinola, 2012). The thermal efficiency of this process is approximately 70% and can increase to 90% with the use of pyrolysis products as fuel (Islam, 2010). The use of tyre chips instead of whole tyres may also increase the efficiency of the process by 20-30% (Burger, 2012). The thermal energy used to drive the pyrolysis reaction is applied indirectly by thermal conduction through the walls of the containment reactor. Pyrolysis generally occurs at temperatures between 400 and 800°C (Kruger and Hings, 2004). As the temperature changes, the product distribution (or the phase of the products) are also altered. Lower pyrolysis temperatures usually produce more liquid products while higher temperatures favour the production of gases. The speed of the process and rate of heat transfer also influences the product distribution (Encinar *et. al*., 2009). There is need for greener fuel alternatives due to fossil fuel depletion (Ramirez-Canon, et. al. 2018; Neto *et. al*., 2019), increasing oil prices and emission challenges (Giugliano *et. al*., 2006). The waste tyre pyrolytic liquid is an oily organic compound, dark brown in colour and with a strong acrid smell. The aliphatic compounds mainly consist of alkanes and alkenes with alkenes being the predominant group, 43.23% (Kegl, 2006). The aromatic compounds are mainly single ring alkyl aromatics. The aromatic nature of the waste tyre pyrolytic oils is due to aryl chain fragments from aromatic rings splitting and circulation of olefin structures through dehydrogenation reaction, (Islam *et al.,* 2008).

Pyrolysis offers an environmentally and economically attractive method of waste tyres transformation into useful products and energy. Utilization of pyrolysis as a medium of recycling disused tyres depend on its by-products reception in the market hence, the importance of pyrolysis products characterization that aids the determination of the area of their usefulness. The characterization of the liquid product (pyro-oil) in tyre pyrolysis will help determine the energy properties for useful optimum applications.

2. LITERATURE REVIEW

Pyrolysis offers ecologically attractive method of decomposition and estimation of a wide scale of waste, including used tyres. Processing of used tyres by pyrolysis means their thermal degradation occurring without the presence of oxygen, in order to volatize and decomposes it to produce oil, gas and char. In the pyrolysis process organic volatiles from the tyres (approximately 60 %) are decomposed into low molecular weight products, liquids or gases that could be subsequently used as fuels or chemicals (Natalia et al., 2013). Non-volatile soot and inorganic components (approximately 40 %) remain in the solid residue and can be recycled in different technological applications.

Cypress and Bettens (1989) checked the influence of tyre type on product yield pyrolysis. For different brands of scrap tyre in two-stage reactor, they reported that, with pyrolysis in the first stage and post-pyrolysis cracking of the gases in a second reactor at higher temperatures there is small but significant differences in product yield depending on the difference brand of tyre used. While according to Kyari et al., (2005) tyre pyrolysis process yield were not significantly influenced by the tyre's brand and its origin but concluded that there are difference in composition of the derived gases and oil from pyrolysis of different brand and types of tyre from different country.

A number of studies have been reported in literature related to tyre pyrolysis for its conversion into valuable compounds. William and Brindle, (2003) used fixed bed and fluidized bed reactors to maximize the selective determination of single ring aromatic hydrocarbons. Ucar *et al*, (2005) pyrolyzed car tyre and truck tyre in a fixed bed reactor and reported that tyre pyrolysis liquids were lighter than diesel but heavier than naphtha.

Berrueco *et al*., (2005) reported that yield of the gas fraction obtained in different experimental systems showed important variations, using an autoclave in a nitrogen atmosphere at temperatures between 400 and 700 ^oC, it was observed that the pyrolyzed gases consisted of CO, CO_2, H_2S and hydrocarbons such as CH_4, C_2H_4, C_3H_6 and C_4H_8, and their unsaturated derivatives. On analysing the pyrolysis gases by gas chromatography, it was noticed that the main gases produced by the pyrolysis process are H_2, CO, CO_2 and hydrocarbons: CH_4, C_2H_4, C_3H_6 and C_4H_8. It was also reported that the produce gas is flammable (Akinola and Adewole, 2015).

Laresgoiti *et al*., (2004) presented a detailed characterization of the liquid products obtained in tyre pyrolysis under nitrogen at 300, 400, 500, 600, and 700 ^oC. The GC/MS analysis, elemental analysis, gross calorific values and distillation data were studied. They report that tyre derived liquids are a complex mixture of C_6-C_{24} organic compounds, containing a lot of aromatics (53.4–74.8%), some nitrogenized (2.47–3.5%) and some oxygenated compounds (2.29–4.85%). Their GCV (42 MJ kg^{-1}) is even higher than that specified for commercial heating oils, but the sulphur content (1–1.4%) is close to or slightly over the limit value. Significant quantities of valuable light hydrocarbons such as benzene, toluene, xylene and limonene were obtained.

Rahim *et al*., (2013) worked on Possibility Study of Pyrolysis Oil Produced from Pine Seeds as an Alternative of Fossil Oil and its Comparison with Pyrolysis Oil Produced from other Sources. A fixed bed pyrolysis system was designed and fabricated for obtaining liquid fuel from biomass solid wastes. The major components of the system were: fixed bed reactor, liquid condenser and liquid collectors. The reactor was heated by means of a cylindrical biomass source heater. Rice husk, cow dung and charcoal are used as the energy source. The products are oil, char and gas. By the experiment it was found that the pyrolysis oil can be used as fossil fuel after some treatments

Natália *et al*., (2013) examined the effects of pyrolysis operational conditions on gas products, solid residues and liquid fractions. It was concluded that the most significant parameter that influenced the final products of pyrolysis is temperature (above all, chosen thermal interval of pyrolytic process duration) and heating speed of the liquidated sample.

Suhanya *et al*., (2013) reported the recovery of oil from waste tyres using pyrolysis method. Thermo-gravimetric analysis revealed that the pyrolysis of tyre rubber at atmospheric pressure starts at a temperature around 250^oC and finishes at a temperature of about 550^oC. In general, by pyrolysing waste tyre, three fractions are obtained: solid residue (around 40 wt. %), liquid fraction (around 50 wt. %) and gas fraction (around 10 wt. %). The general trend is an increase in yields of liquid and gas fractions as the temperature increases.

Ningbo, *et al*., (2009) researched into fine powder and large particle tyre pyrolysis. The work investigated the kinetics of the pyrolysis of scrap tyre using thermo gravimetric and a small batch laboratory-scale externally heated fixed-bed. Five particle sizes (20–40, 40–60, 60–80, 80–100, 100–200 mesh) and five heating rates (5, 10, 15, 20, 40 K/min) were investigated. The results show that there is no obvious distinction on weight loss for different sizes. With increasing heating rate, the weight loss regions shift to a higher temperature range and the weight loss rate increases. The activation energy (E) and pre-exponential factor constant (A) were calculated using the Arrhenius type kinetic model. The heating value of pyrolysis gases is in the range of 12 to 22 MJ. The yields of solid, liquid and gas of pyrolysis scrap tyre and tyre cube at 800 °C were investigated, the ratio of pyrolysis production composition of scrap tyre and tyre cube for gas, oil and char is found to be 35:23: 42 and 10:31:59, respectively

González, (2001) studied the kinetics of the thermal decomposition of the feedstock using isothermal and non-isothermal thermo-gravimetric methods at different temperatures (400–600°C) and heating rates (5 – 50 K min−1). The results indicated that the isothermal pyrolysis process consisted of one stage and the non-isothermal three stages.

Tyre pyrolysis is currently receiving renewed attention. Recycling of tyres by pyrolysis offers an environmentally attractive method. The products of the tyre pyrolysis process are: Solid char (30 - 40 wt. %), liquid residue (40-60 wt. %), and gases (5-20 wt. %) (Cumali, 2011; Shah, 2006**)**. The solid residue contains carbon black and the mineral matter initially present in the tyre. This solid char may be used as reinforcement in the rubber industry, as activated carbon or as smokeless fuel. The liquid product consists of a very complex mixture of organic components. Thus, the derived oils may be used directly as fuels, petroleum refinery feedstock or a source of chemicals. The gaseous fraction is composed of non-condensable organics as, H_2, H_2S, CO, CO_2, CH_4, C_2H_4 and C_3H_6. The gas fraction can be used as fuel in the pyrolysis process (Juan *et al*, 2001).

The Socio – economic development, standard of living, as well as the quality of life of any country largely and primarily depend on the availability and supply of energy. The availability and supply of energy came under great pressure because of increasing demands of the ever-increasing global population. This eventually leads to the problem of energy crisis which is especially true for developing countries like Nigeria (Akinola and Fapetu (2015). The aim of this paper therefore is to perform a characteristics study of the liquid product (pyro-oil) of waste tyres that include calorific value and fixed carbon for its energy content.

3. MATERIALS AND METHOD

3.1 Materials and Equipment

The materials used for the experiment include: disused tyres retrieved from vehicle users, vulcanizing centres and dump hills in Akure and its environment in South West Nigeria. The equipment is a 17.4 m^3 refurbished reactor that consists of a furnace, a thermal reactor, condensing unit, digital electronic control unit, water reservoir, water pump, flow meter, control valve, interconnected pipes and condensed gas collection point. The plant can withstand a pressure of 2.3 MN/m^2 and a temperature of 1200^{o}C (Akinola, 2012; Akinola *et al.,* 2018).

3.2 Methods

i. Sample Preparation

Disused borex tyres were shredded into average sizes of about 20 x 30 x 6.8 mm. The shredded products were grouped into four, labelled A, B, C and D, each having four samples of 1 kg each, totalling 16 samples. The respective feed stocks were weighted using a digital top loading weighing balance as shown in Plate 1. The four samples in each group were used for one temperature each; i.e.; samples A(1 – 4) for 250^{o}C, B(1 – 4) for 350^{o}C, C(1 – 4) for 450^{o}C and D(1 – 4) for 600^{o}C respectively.

Plate 1: Weighing of Feedstock sizes

ii Pyrolysis of the Shredded Tyre

All 16 samples were pyrolysed in the refurbished fixed bed batch reactor of inner diameter 240 mm and depth 385 mm, one sample at a time. Two K-type thermocouples were located at the inner chamber to measure the pyrolytic temperature and the outer one beside the chamber to measure the temperature around the reactor and the bed.

One sample from category A(1) was loaded into the reactor and heated up to a temperature of 250^{o}C. Once the temperature had stabilized, the system was held at this temperature for 4 hours. The temperatures throughout the reaction were taken at intervals of 30 minutes. Then the liquid condensate was collected in a vacuumed gas cylinder immersed in an iced bath. The other three samples in group A(2, 3, 4) were then heated one after the other, to 250^{o}C. The experimental setup is shown in Figure 1

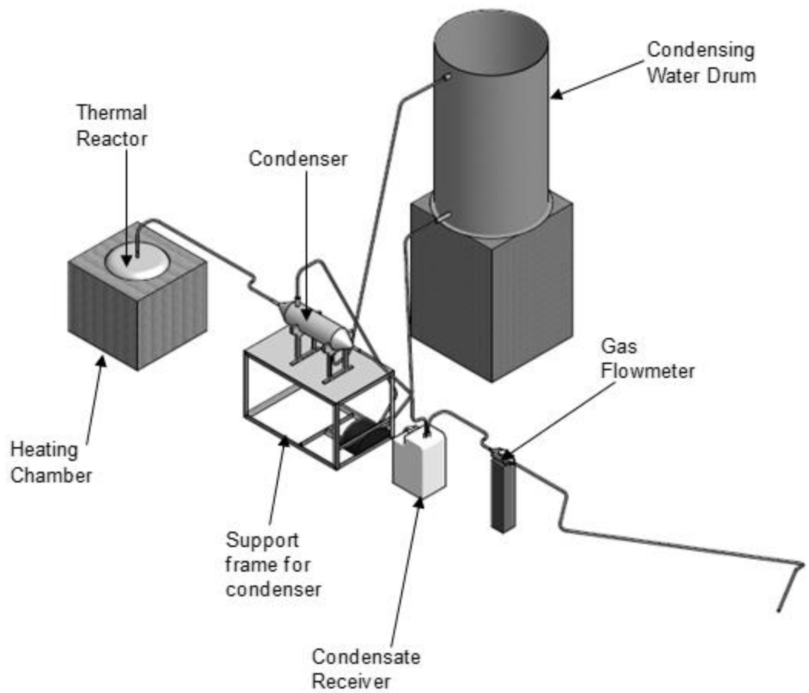

Fig. 1: Experimental set up for fixed batch thermochemical reactor
Source: Akinola, 2012

Similarly, the procedure used for group A was followed in carrying out the experiment for the remaining tyre samples in groups B(1 – 4), C(1 – 4), and D(1 - 4) at 350°C, 450°C, 600°C respectively. The average liquid yield per time for each samples A, B, C and D were taken for each of the specified temperatures to measure the effect of temperature on the liquid yield from the pyrolysis.

The oil yield was determined by its mass and expressed as a percentage by weight as given in equations (1) and (2)

$$Y_{oil} = \frac{M_{oil}}{M_{rt}} \times 100 \quad (1)$$

But M_{rt} = 1 kg

Therefore,

$$Y_{oil} = M_{oil} \times 100 \quad (2)$$

where

Y_{oil} is the oil yield (%)
M_{oil} is the mass of oil (kg)
M_{rt} is the mass of raw tyre (kg)

4. RESULT AND DISCUSSION

4.1 Oil Yield

The pyrolysis of the raw shredded borex tyre in the reactor was carried out at four different temperature ranges of 250°C, 350°C, 450°C, and 600°C. The average results of the percentage yield of the products collected at each of the temperature ranges and various time intervals are shown in Table 1. The percentage by weight yield analysis of each constituent (solid, liquid and gas) with respect to temperature of the reactor is shown in Fig. 2.

Table 1. Percentage yield of products at different temperatures and time intervals.

Temperature (°C)	% Solid	% Liquid	% Gas	Time (Minutes)
250	97.20	-	2.80	28
350	63.70	31.80	4.50	35
450	58.15	30.80	11.10	105
600	51.90	29.40	18.70	245

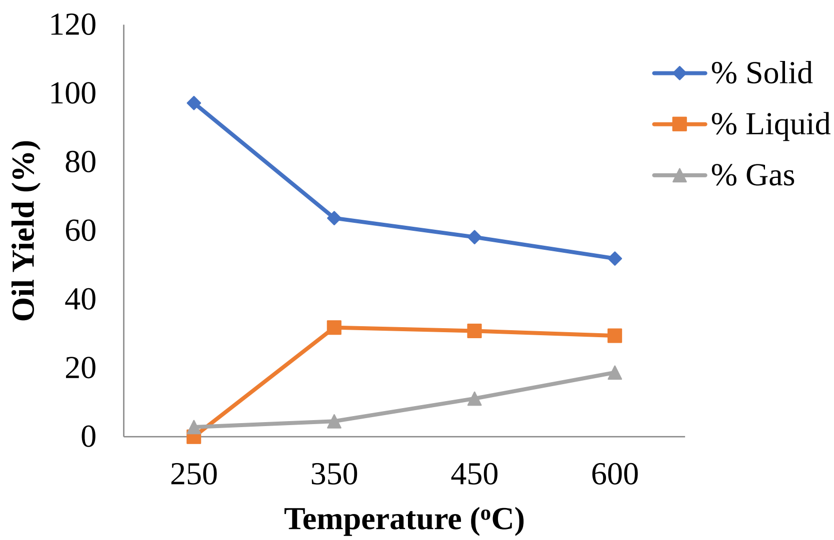

Fig. 2. Percentage yield of Solid, Liquid and Gas against Temperature

This result shows that the higher the temperature the higher the gas yield and the lower the solid and liquid yield from the pyrolysis because the system temperature is approaching the boiling point of the tyre material.

3.2 Proximate and Ultimate Analyses

The liquid samples were taken for analysis. The results of the ultimate and proximate analysis of the raw tyre and pyro-liquid are shown in Tables 2 and 3.

Table 2. Proximate and Ultimate analysis for raw Borex tyre

Proximate Analysis			**Ultimate Analysis**		
Constituents	**% Weight**	**Heating Value (MJ kg–1)**	**Constituents**	**% Weight**	**Specific Gravity (kg/m^3)**
Volatile Matter	71.88	37.18	Carbon	79.86	343.00
Fixed Carbon	28.75		Hydrogen	7.35	
Moisture Content	2.07		Nitrogen	1.08	
Ash	5.36		Sulphur	1.57	
			Oxygen	6.50	

Table 3. Proximate and Ultimate analysis for pyro-liquid from Borex tyre

Proximate Analysis			**Ultimate Analysis**		
Constituents	**% Weight**	**Heating Value (MJ kg–1)**	**Constituents**	**% Weight**	**Specific Gravity (kg/m^3)**
Volatile Matter	0.78	40.58 MJ/kg	Carbon	84.52	811.50
Fixed Carbon	96.81		Hydrogen	6.33	
Moisture Content	2.04		Nitrogen	0.09	
Ash	0.37		Sulphur	0.05	
			Oxygen	0.02	

Proximate and ultimate analyses of the raw tyre and the oil resulting from the pyrolysis were carried out. The proximate analysis shows a volatile composition of 0.78%, 96.81% fixed carbon, 2.04% moisture content and 0.37 ash content. The pyrolysis process produced oil with an average of 84.52% carbon composition 6.33% hydrogen, 0.09% nitrogen, 0.05% sulphur and 0.02% oxygen composition. The analysis of percentage composition of the raw scrap tyre samples gave 79.86% for carbon, 7.35% hydrogen, 1.08% nitrogen, 1.57% for sulphur and 6.50% for oxygen. The energy content of the oil resulting from pyrolysis gave an average value of 40.58 MJ/kg and 811.50 kg/m^3 for specific gravity. The average fixed carbon content was 96.81% while the volatile matters gave an average of 78 %. The ultimate analysis of the raw tyre samples gave values of 37.18 MJ/kg for the heating value and 343.00 kg/m^3 for specific gravity.

The fuel properties of the pyro oil are presented in Table 4. The fuel properties obtained were compared with those of raw waste tyre and conventional diesel. Results showed that the produced pyro oil from waste tyre compares favourably well with conventional diesel in terms of heating value, density, kinematic viscosity and flash point.

The liquid phase is the most important product of tyre pyrolysis process because of its wide range of potential applications such as being an alternative to fossil fuel. Flash point as defined by word web dictionary is the lowest temperature at which the vapour of a combustible liquid can be ignited in air (Nhlanhla and Edison, 2014). A flash point value of 44.3ºC agrees with the work of Williams (2013) who reported that the flash point is low when compared to that of conventional diesel fuel. This was due to its being unrefined oil within the range of boiling point fraction and volatile hydrocarbons. The viscosity of tyre pyrolysis oil is also close to that of diesel oil.

Compared to other fuels' heating values namely Diesel fuel (45.50 MJ/kg), Kerosene (43.10 MJ/kg) and Petrol (45.8 MJ/kg), Pyro-oil which has 40.58 MJ/kg will compare favourably as alternative fuel. The compared values are shown in Figure 3. Also since the raw materials are waste from used tyres that littered the environment; and with readily available technology the conversion from waste to wealth will be readily achievable.

Table 4: Fuel properties of pure diesel fuel as reported compared with present study

Fuel properties	**Raw waste tyre**	**Conventional (Pure) diesel**	**Borex tyre (Present study)**
HV (MJ/kg)	37.18	45.50	40.58
C (wt %)	86.19	86.14	81.26
H (wt %)	10.33	13.20	5.67
O (wt%)	0	0.66	4.23
N (wt %)	0.79	0.00	1.03
S (wt %)	0.83	0.00	0.68
FAME (Vol %)	0	5.8	-
H/C atomic ratio	1.44	1.84	-
Stoichiometric air/fuel ratio (kg/kg)	13.46	14.38	-
molecular weight (kg/kmol)	142.5	203.7	-
Density (kg/m^3)	917	845	811.5
kinematic viscosity at 40°C	2.39	2.79	3.8
flash point (°C) EN 3679	23.0	60.5	44.3
TAN (mg KOH/g)	5.0	0.30	0.42
Cetane number	-	-	55

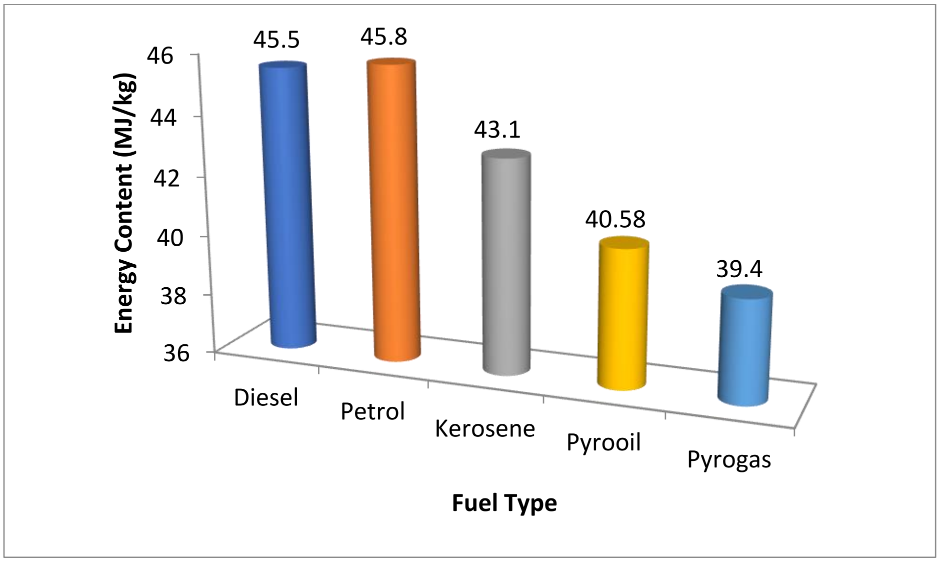

Fig. 3: Energy content of Borex Pyro-liquid with other fuels

4. CONCLUSION

The Variation in temperature affects the yield of the pyro-liquid. An increase in temperature reduces the yield of the char while simultaneously increasing the yield of tar and gas. In this report the pyro liquid high carbon percentage and low ash content shows good combustible property as shown in the heating value.

Also, there was an increase in carbon content as compared to that of the raw tyre samples. All the remaining compositions reduced with pyrolysis except nitrogen which also experienced an increased percentage composition. The nitrogen itself also has a direct cleaning effect, breaking down carbon deposits that can harden on an engine's moving parts. This make this pyro liquid unique compared with others.

The major content of the solid product is however carbon as it has a very high percentage composition in the product after analysis. This implies a more production of carbon dioxide during complete combustion.

Comparing the heating value of oil to the raw tyre sample showed that oil has a higher heating value and thus is considered a more viable choice for energy production. The ultimate analysis indicated low sulphur content of 0.68% which means the fuel has a low harmful emission from combustion.

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