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Synthesis and Characterization of Fuel Oil Produced by the Catalytic Pyrolysis of Used Tyres

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Abstract— The effective solid waste management and transformation of wastes to useful products have been utilized. In this study, the catalytic pyrolysis of disposed tyres using NaOH Catalyst, in a pilot scale continuous stirred tank reactor was carried out. The experiment was carried out at varying operating conditions, from which the optimum yield of 52.075 % of fuel oil was achieved at the decomposition temperature, heating rate and the catalytic concentration of 700 oC, 40 oC/min and 12.5 % respectively. The characterization of the produced fuel oil properties compared favourably with that of conventional diesel fuel with heating value of 42.407 MJ/kg, within the standard range of 42 – 44.5 MJ/kg and the Cetane number of 48.511 falls within the recommended range. Furthermore, the FTIR analysis showed that the pr0duced 0il c0ntained c0mplex mixture 0f comp0unds with C6 – C18 carb0n 0f paraffins, naphthenes, 0lefins, and ar0matics c0mp0und that are f0und in standard conventional diesel fuel. The GC-MS analysis showed 84.4325 area-percentage of 41 hydrocarbon compounds comprising of 30.24 % Aromatics (Benzene, Tuolene and Xylene Compounds), 32.43 % Paraffin, 30.52 % non-saturated Alkenes and Alkynes compounds, 6.68 % sulphuric compound and 0.13 % of Nitrogen compound. Catalytic pyrolysis with NaOH is capable of converting disposed tyres to a valuable product that has the potential to be used as an alternative source of energy, could also be used as reformate or even blended with conventional fuel.

Keywords— Biodiesel, Catalytic Pyrolysis, Energy, Pyrolyzed oil Characterization, Solid Waste Management, Waste Tyres.

I. INTRODUCTION

In developing countries, solid waste disposal is mostly in an organized dumping sites and/or landfills (Hoornweg and Bhada-Tata, 2012). Used tyres are among such wastes, they are bulky, non-degradation, non-decomposable and non-recyclable. The idea of waste to energy (WtE) is taking the centre stage globally (Barua and Hossain, 2021). In addition, the over 90% dependency on fossil fuels which of course is non renewable, rising petroleum prices, increasing threat to the environment from exhaust emissions and global warming have generated intense international interest in developing alternative non-petroleum fuels for engines and power generation (Senthil and Prabu, 2014).

In recent years, most nations are faced with major problems of environmental hazards caused by used tyres due to the alarming use of automobiles, rapid industrialization and disposal. On a global view, approximate estimate of about 1.5 billion tyres is labelled as End of Life Tyres (ELTs) (Williams, 2013). About 85 % ELTs are derived from regular cars driven by individuals while the remaining 15 % are from truck and other categories. Disposed tyres or ELTs are no doubt very significant solid wastes and have increasingly influenced negatively to the environment. This increase will continue to grow proportionally as long as vehicle use keeps increasing all over the globe. (Dogan et al., 2012).

Oba et al. (2015) disclosed that, in Africa, Nigeria and South Africa are the highest producers of ETLs, roughly estimated at 259 million annually. Dogan et al. (2012), affirms that due to improper disposal methods, these waste tyres are capable of causing harm to both human and the environment. This is because the disposed tyres accumulate and fill up the landfills since they are not degradable.

In Weng and Chang (2001) views, these disposed tyres are not only non-degradable but also cannot easily undergo compression or folding hence, they are bulky and can easy fill up an entire landfill. Of all the methods of solid waste disposals, like landfills, incineration, compression, folding, etc, the realistic method of disposal for used tyres is the conversion of the waste tyres to useful products (Hinchliffe et al., 2017). Numerous methods of conversion have been carried out, however, one of the most successful methods of converting polymeric material to liquid oil is pyrolysis (Quek and Balasubramanian. 2013).

Pyrolysis is the thermal decomposition of organic material in an inert atmosphere or vacuum (Dogan et al., 2012). This process becomes an option of waste-to-energy technology to deliver bio-fuel to substitute fossil fuel and to curb the problems of environmental degradation caused by scrap tyres disposal. Pyrolysis is one of the conventional methods for the conversion of used tyres to energy (Quek and Balasubramanian. 2013). The pyrolysis of used tyres is an alternative environment-friendly process of disposing the waste while producing valuable product (Wongkhorsub and Chindaprasert, 2013). The process is also environmentally friendly unlike the use of incineration. In addition, the oil product from tyre pyrolysis has a higher shelf life (Dogan et al., 2012). The production of tyre-pyrolyzed oil and its ability to run on diesel engine with fuel properties that are comparable to diesel oil has been investigated by several researchers (Wongkhorsub & Chindaprasert, 2013). The conversion of used tyre to fuel oil through pyrolysis is affected by several factors. The effects of operating conditions such as heating rate, particle size, feed stock composition, pyrolysis residence time, catalyst ratio, pressure and temperature have been explored (Dogan et al., 2012; Quek and Balasubramanian. 2013; Pilusa and Muzenda, 2013; Wongkhorsub and Chindaprasert, 2013; Nazim et al., 2014; Osayi et al., 2014; Alkhatib, 2015; Wrzesińska et al., 2016).

Researchers over the years have affirmed that waste tyres can effectively be converted to fuel oil through catalytic pyrolysis. The effects of various advanced catalysts on the pyrolysis of used tyres to fuel was examined and the results showed that these catalysts influence the oil yield and characteristics of the pyrolyzed oil (Miandad, et al 2018). The oil yield for activated alumina (Al2O3), activated calcium hydroxide (Ca(OH))2, natural zeolite and zeolite (H-SDUSY) were 32 wt%, 26 wt%, 22 wt% and 14 wt% respectively (Miandad, et al 2018). The use of zeolite ZSM-5 in the catalytic pyrolysis of scrap tyres has a lower liquid yield with better aromatic compound contents (Abedeen, et al, 2021). This implies that zeolite catalysts are capable of producing pyrolyzed oil with higher aromatic properties but have lower oil yield (Arabiourrutia, 2020).

II. MATERIALS AND METHODS

Pyrolyzed Oil Production

A pilot size continuous stirred tank reactor (CSTR) was used for this studies. The feed is passed through a furnace linked to a CSTR were the temperature was maintained and the desired heating rate set. A condenser was attached to the reactor to condense the vapour coming from the reactor and cooling was provided through cooling water passed through the tubing of the condenser. The tubing ends were fitted with tapers and a stainless steel mesh at the joints to prevent large particulates from escaping with the carrier gas as depicted in figure 1. 100g of the prepared waste tyre sample was used in the pyrolysis reactor for each run. The produced tyre pyrolyzed oil yield was determined by weighing the oil produced. Yield (%) was then determined using expression in Equation 1.

$$Percentage\ Fuel\ Oil\ Yield = \frac{W_o}{W_s} \times 100 \qquad (1)$$

Where; W_o = weight of pyrolyzed oil per run (g) and W_s = Weight of tyre sample used (g)

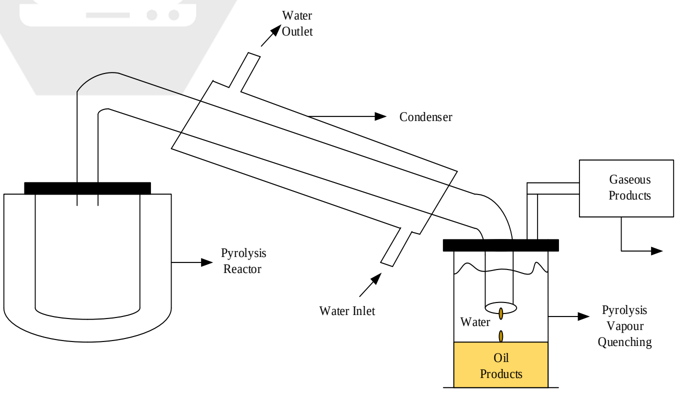

Figure 1: *Schematic diagram of the pyrolysis setup*

Performance Evaluation of the Produced Fuel Oil

The produced tyre pyrolyzed fuel oil was characterized for combustion and performance characteristics such as kinematic viscosity, specific gravity, flash point and heating value to determine the suitability of the produced used pyrolyzed tyre oil for used in ignition engine.

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Characterization of the Produced Pyrolyzed Oil

The product was characterized to evaluate the properties of the produced oil. The FTIR and GC-MS analysis were used to determine the constituent of the produced pyrolyzed oil.

III. RESULTS AND DISCUSSION

Production of Fuel Oil from the Catalytic Pyrolysis of Used Tyres and the Effects of the Operating Conditions to Oil Yield

The results of the production of fuel oil from used tyre catalytic pyrolysis at varying operating conditions are as shown in table 1 below. The experiments were carried out at a temperature range of 300 to 700 oC. The results are tabulated in Table 1. The results confirm that pyrolysis favours higher temperature. The heating rate is another important factor. The heating rate is directly proportional to the oil yield. The result above indicated that the pyrolysis of used tyres requires just about 12.5 % of catalyst to achieve optimum yield in conjunction with the other parameters.

Table 1: *Fuel oil yield at varying operating conditions*

Run	Temp. (oC)	Catalyst (%)	Heating Rate (oC/min)	Fuel Oil Yield (%)
1	300	5	25	10.330
2	300	12.5	10	12.632
4	300	12.5	40	18.850
5	300	20	25	20.290
6	500	5	10	22.310
7	500	5	40	29.110
8	500	12.5	25	49.920
9	500	20	10	40.015
10	500	20	40	49.945
11	700	5	25	38.100
12	700	12.5	10	45.575
13	700	12.5	40	52.075
14	700	20	25	51.098

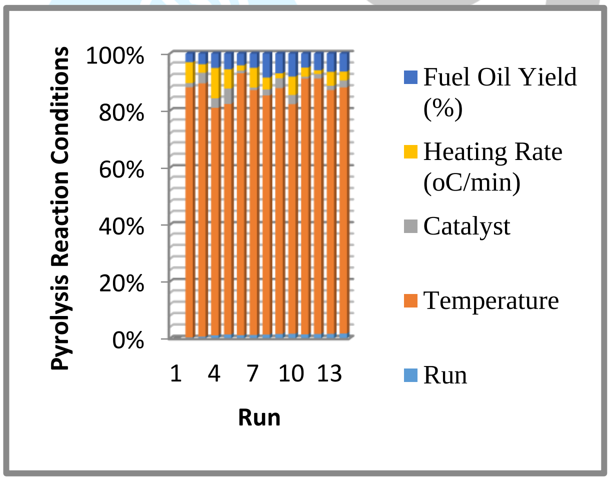

Figure 2: *Effect of temperature, catalyst and heating rate on percentage fuel oil yield*

The effects of temperature and the other reaction conditions are as depicted in fig. 2. The oil yield is proportional to increase in temperature. In addition, no matter the temperature and catalyst concentration, heating rate is a major contributor to the yield. The result showed that Catalytic Pyrolysis of used tyres using NaOH requires 12.5 % of the catalyst concentration at a heating rate of 40 oC/min and a maximum temperature of 700 oC to yield the optimum fuel oil of 52.075 %.

Characterization of the Produced Fuel Oil

The produced pyrolyzed tyre oil was characterized using FTIR and GC-MS to evaluate the functional group in the produced pyrolysis oil and GC-MS was used to examine the constituent of the produced pyrolysis oil. The results obtained are discussed.

Table 2: *FTIR result of constituent functional group of the produced pyrolyzed tyre oil*

No.	Wavelength (cm-1)	Functional group	Class of compounds
1	655.82	C–H	Aromatic, out-of-plane bend/ Alkyne
2	759.98	C-H bending	Aromatic ring
3	794.7	C-H bending	Alkene+ Phenyl ring substitution

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4	887.28	C-H bending	Alkene
5	949.01	C-H bending	Alkene
6	1o87.89	C–H, C=S	Aromatic, in-plane bend, thiophenes
7	1300.07	C-N stretching	Amines
8	1438.94	C-H bending	Alkane
9	1527.67	C=C-C	Aromatic ring stretch
10	1674.27	C=C stretching	Aromatic ring stretch
11	2087.05	C≡C	Alkyne
12	2364.81	C≡N	Alkyne
13	2596.27	S–H stretch	Thiols and thio-substituted compounds
14	2750.58	C-H stretching	Aldehyde
15	2904.89	C-H stretching	Alkene
16	3066.92	C–H stretch, =C–H stretch	Aromatic
17	3240.52	C–H stretch	Alkane
18	3371.68	O–H	Hydroxy group, H-bonded O–H stretch
19	3456.55-3892.48	O–H, N–H	Alcohol, hydroxy group, phenol

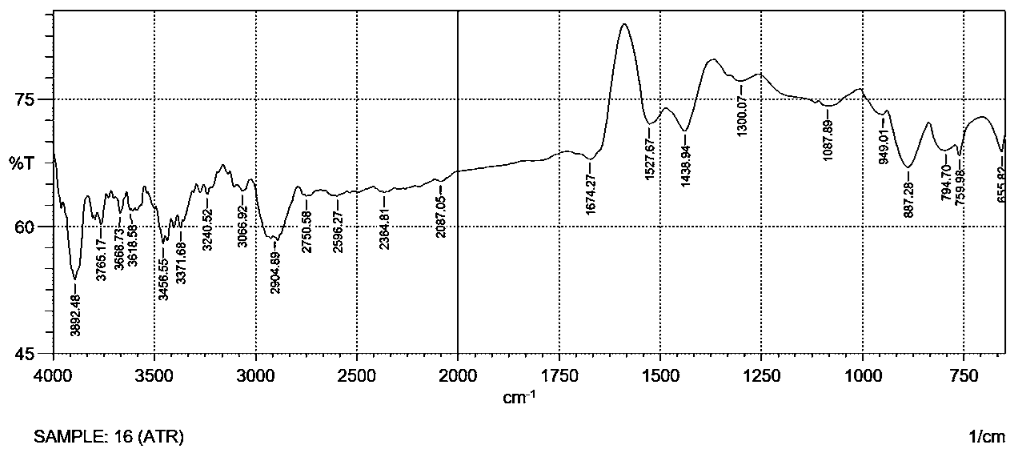

Figure 3: *FTIR spectrum of the produced pyrolyzed tyre oil*

Table 3: *GC-MS analysis of major compounds present in pyrolyzed tyre oil*

Retention Time	Area (%)	Compound Identified
3.29	5.2924	Methylcyclohexane
4.061	1.6863	m-Ethylmethyl benzene
4.274	3.8599	Decane
4.529	3.8546	1,2,3 Trimethyl benzene
4.859	2.0919	Benzene,
4.974	2.7389	1,4- Diethyl benzene
5.61	1.9682	2,4,4-Trimethyl-2-pentene
5.83	6.9800	Ethylcyclopentane
5.962	5.1090	Benzene, 1,2,3,4tetramethyl, o-Cymene
5.979	1.7644	Benzonitrile
6.035	1.7399	D-Limonene
6.43	5.2134	1-Ethylcyclopentene
7.356	2.9236	Dodecane
7.55	0.2375	2-Methyl-1-heptene
7.62	2.1697	2-Methyl-1,3-butadiene
8.18	0.9923	2-Methyl-2-butene

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10.45	0.7342	1H-Indene, 2,3dihydro-1,1,5trimethyl-
12.53	1.8747	3-Methyl-octane
12.67	3.5408	2,2-Dimethyloctane
12.9	0.4593	2,3-Dimethyl-1,3-butadiene
13.587	1.5589	Naphthalene, 2,3,6trimethyl
16.35	5.6437	Dibenzothiophene
16.662	2.2716	2-Methyltetradecane
18.65	1.4502	Tricylene
19.25	1.0158	2-Propenylidene-cyclobutene
19.39	1.9511	Toluene
24.35	2.4439	p-Xylene
24.41	1.4114	Phenol
25.35	2.5750	1,7-Octadiyne
28.54	1.0996	5,9-Tetradecadiyne
29.51	0.7074	7,11-Dimethyl-3-methylene-1,6,10-dodecatriene
32.38	0.3285	8-Methylene-dispiro 2.o.2.5 undecane
33.66	0.3084	Heptadecane
34.83	0.7057	o-Cresol
43.88	0.1207	2,6-Dimethyl-2-trans-6-octadiene
52.22	2.9932	Naphthalene
54.78	0.9362	1,3,5-Trimethylbenzene-2-ethenyl
69.86	0.1080	Phthalonitrile
75.88	0.2377	1-Methylnaphthalene
95.27	0.4942	1,4-Dimethylnaphthalene
98.49	0.8403	1,2,3-Trimethyl-1,2-dihydro-2-quinoline

Fourier Transform Infra-Red (FTIR) Spectroscopy of Pyrolyzed Tyre Oil

The FTIR is a measure of the quantitative and qualitative analysis of functional group of organic and inorganic samples. The FTIR spectrum for the produced pyrolyzed tyre oil is as shown in Figure 5 and the results obtained from the transmittance spectrums are presented in Table 2.

These results showed that, the constituent functional groups of the produced pyrolyzed tyre oil are mainly, aliphatic chains, aliphatic compounds, and oxygenated functional groups, such as phenolic, hydroxyl or carbonyl groups, as well as few amine functional group. The bands obtained for the functional groups are further shown in Fig. 3.

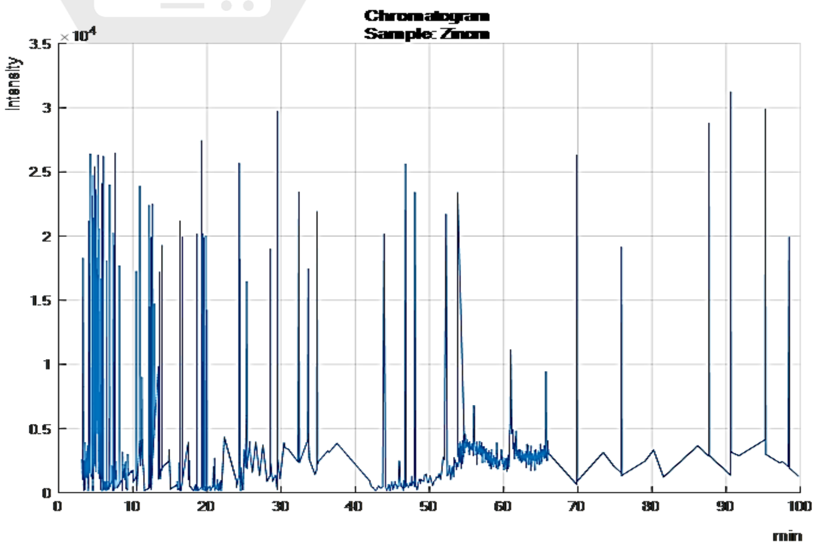

Figure 4: *GC-MS chromatogram of the produced pyrolyzed tyre oil*

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The intermolecular bonded hydroxyl groups (OH) and N–H were observed at 3800 – 3500 cm-1. This band is attributed to the presence of hydrogen-bonded O-H group of alcohols, carboxylic acids and phenols. The band observed at 650 – 950 cm-1 also indicates mainly the presence of aromatic and alkane functional. It was concluded that the presence of different functional group of compounds indicate the presence of hydrocarbons in the produced pyrolysis oil and also the O-H broad peak was attributed to the interaction of O-H bonding with hydrocarbon contents.

Gas Chromatography and Mass Spectrometry (GC MS) Analysis of Pyrolyzed Tyre Oil

The GC-MS analysis of the produced pyrolyzed tyre oil was carried out to determine the constituent of the produced oil. The GC-MS analysis was used to separate pyrolysis oil mixtures into individual components and to identify the various components from their mass spectra. Table 3 shows the compounds identified and their percentage area compared to the total area of chromatogram, which gives an estimate for their relative concentration in the pyrolyzed tyre oils while Figure 6 shows the chromatogram of the produced pyrolysis oil. From Table 3, it can be seen that the produced pyrolyzed tyre oil is a complex mixture with 84.4325 Area (%) of forty one (41) identified compounds. The pyrolyzed tyre oil contained mainly, paraffins, unsaturated hydrocarbons, aromatics (BTX) as well as minor traces of nitrogen and sulphur containing compounds as shown in Table 3.

From Table 3, the pyrolyzed tyre oil contains 30.24 percent composition of the aromatics comprising of Benzene, Toluene and Xylene compounds, 32.43 percent composition of paraffinic compounds, 30.52 percent composition of the Alkenes and Alkynes compounds, 6.68 percent composition of sulphuric compound and 0.11 percent of nitrogen compound. The GC-MS results confirmed the FTIR results obtained for the produced pyrolyzed tyre oil. The presence of oxygen, nitrogen and sulphur compounds established

oils while Fig. IV shows the chromatogram of the produced pyrolysis oil. From Table 3, it can be seen that the produced pyrolyzed tyre oil is a complex mixture with 84.4325 Area (%) of forty one (41) identified compounds. The pyrolyzed tyre oil contained mainly, paraffins, unsaturated hydrocarbons, aromatics (BTX) as well as minor traces of nitrogen and sulphur containing compounds as shown in Table 3.

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Therefore, both the FTIR and GC-MS analysis affirmed that the produced pyrolyzed tyre oil contained complex mixture of compound with C6 – C18 carbon of paraffins, naphthenes, olefins, and aromatics compound that are found in standard petroleum diesel fuel. These compositions are similar to the compositions of a conventional diesel fuel. Dibenzothiophene is a known component of petroleum derived oil. The GC-MS and FTIR analysis further confirmed the characteristics properties of the pyrolyzed tyre oil. This pyrolyzed oil can be used as fuel oil for engines, undergo further treatment for transport fuel or as reformate (due to its high aromatic contents) for other useful chemicals.

The pyrolyzed tyre oil produced was characterised for suitability and fuel performance. The kinematic viscosity, density and specific gravity, flash point and heating value and octane number of the produced oil were evaluated based on the ASTM standards. The fuel properties determined for the produced fuel oil are shown in Table 4.

The fuel oil was further tested to confirm its suitability for use in any type of compression-ignition engine/diesel engine. The result of the fuel properties for the pyrolyzed tyre oil were analyzed and compared with ASTM D–6751 diesel fuel standard value. From Table 4, the density and specific gravity of the pyrolyzed tyre oil was determined as 0.939 g/cm3 and 0.937 respectively.

The kinematic viscosity which is a measure of the fuel spray atomization and fuel system lubrication was evaluated for the pyrolyzed tyre oil. It can be seen from Table 4 that kinematic viscosity of 3.13 mm2/sec obtained for the pyrolyzed tyre oil is within the acceptable range of value recommended by ASTM D–6751 for diesel fuel.

Fuel Properties of Pyrolyzed Tyre Oil

Table 4: *Properties of the Produced Pyrolyzed Tyre Oil*

S/N	Properties	Diesel Standard Value (ASTM D6751)	Pyrolyzed tyre oil
1	Kinematic viscosity (mm2/sec)	1.3 – 6	3.13
2	Density at 40 °C (g/cm3)	0.820 – 0.900	0.939
3	Specific gravity	–	0.937
4	Flash point, (oC)	> 38	63
5	Heating value (MJ/kg)	42 – 44.5	42.407
6	Cetane Number	> 40	48.511

The flash point of a liquid is the minimum temperature at which the liquid gives sufficient vapours to ignite shortly when a flame of standard dimension is brought near the surface of the liquid. It is a measure of the safety in handling of fuel oil. It can be seen from Table 4 that the flash point of 63 oC obtained for the pyrolyzed tyre oil is very suitable. Typically, the flash point of diesel ranges between 52 to 82 Degree Celsius.

The heating value which is a measure of the fuel economy, was determined as 42.407 MJ/kg for the pyrolyzed tyre oil. The obtained value is absolutely within the recommended range for diesel fuel. Cetane number is one of the most important properties of diesel fuel. It is a measure of the ignition, smoking and emission quality of diesel fuel. From Table 4, the Cetane number obtained is 48.511. The obtained Cetane number is suitable for diesel fuel.

IV CONCLUSION

The catalytic pyrolysis of used tyres for the production of fuel oil was successful. The fuel properties of the produced pyrolyzed tyre oil compared favourably with that of fossil diesel fuel.

The FTIR and GCMS analysis affirms that the produced pyrolyzed tyre oil contained complex mixture of compound with C6 – C18 carbon of paraffins, naphthenes, olefins, and aromatics compound that are found in standard petroleum diesel fuel. Therefore, the use of NaOH as catalyst for the pyrolysis of used tyres has proven to have higher oil yield and good characteristic properties. It is therefore recommended for used tyre disposal and veritable means of waste to energy.

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