Upgrading Pyrolysis Oil for Alternative Sources of Petroleum

A. Khaleque[1]*, M. Afroze[2], M. Khan[2], M.S Hossain[3], M.M. Islam[4] and M. Nurunnabi[1]

[1]*Department of Applied Chemistry and Chemical Engineering, Bangabandhu Sheikh MujiburRahman Science and Technology University, Gopalganj-8100, Bangladesh*
[2]*Designated Reference Institute for Chemical Measurement Lab, Bangladesh Council of Scientific and Industrial Research, Dhaka-1205, Bangladesh*
[3]*Department of Mechanical Engineering, Rajshahi University of Engineering and Technology, Rajshahi-6204, Bangladesh*
[4]*Department of Chemistry, Bangladesh Army University of Engineering and Technology (BAUET), Qadirabad Cantonment, Natore-6431, Bangladesh*

Abstract: In this research the selection of suitable tire oil liquid is our main task. For this the upgrading pyrolysis oil has been analyzed by GC-MS and tries to get similarity with conventional diesel fuel. The GC-MS analysis it is shown the following compounds- octane, n-octane, benzene, nonane, cyclohexane, naphthalene, pentadecane are commonly available at upgrading (171-270) °C pyrolysis oil and conventional diesel fuel. But the other pyrolysis oil fraction is nearly matched by this analysis. From the experiment it is also tested the (171-270) °C pyrolysis oil give about similar efficacy that of conventional diesel fuel. The physico-chemical parameter, GC-MS analysis and efficacy testing are responsible such type of nature.

Introduction: The limited reserves of stored fossil fuels will be nearly exhausted in recent future. For this mankind already tries to investigate another energy sources such as biomass, hydropower, geothermal energy, wind energy, solar energy, and nuclear energy [1-4]. The developed alternative fuel research is ensuring to deliver the replace of fossil fuel. The focused sources are bio-ethanol, bio-diesel, lipid derived bio-fuel, waste oil recycling, pyrolysis, gasification, biogas and Fischer-Tropsch synthesis [2, 3].

Now a day the organic wastes are increasing day by day. Among the common organic wastes the scrap tire are most important. The various investigation the estimated amount of wastes tire are 15 million ton in the European Union, 2.5 in North America, 2.4 in UK, 1 in China, 0.5 in Japan, 0.17 in Korea, 0.09 in Bangladesh. This organic solid waste is non- biodegradable. For this the final destination of this waste is landfill; but this solution seems to be not advantageous [3-5].

The product obtained in pilot scale waste tire pyrolysis process is about 47wt%, 43wt% and 10wt% of solid, liquid and gases, respectively at optimum reactor operating temperature of 450°C. The liquid phase is a complex mixture of hydrocarbons. The liquid oil is easily transported, can be burnt directly in the power plant, petroleum refinery, gas turbine or upgraded to obtain light hydrocarbons for transport fuel [1, 3, 5]. The upgrading oil in the temperature range (171-270) °C can be used as light oil fuel; which gross calorific value (GCV) of 42.79 mj/kg. This encourages us trying to replace the conventional diesel fuel. For this the upgrading pyro oil can be demand the replace of conventional diesel fuel; but not the crude pyrolysis oil [8-11].

Corresponding author details:
E-mail address: akhaleque1989@gmail.com
Tel: +88 01731454640

The authors of the previous research work have developed a packed distillation column (Lab scale). Where the fractions were characterized by analyzing physico-chemical parameter, elemental analysis and FT-IR techniques and compared with conventional diesel fuel. Upgrading experiments of product oils have been conducted by fractional distillation, oxidative desulfurization and de-colorization. In fractional distillation 33.08%, 25.33%, 10.67% and 2.17% oils by volume are obtained over the temperature range (171-270) °C, (121-170) °C, (71-120) °C and (40-70) °C respectively. Then the desulfurization step removed around 60-65wt% sulfur. The heat of the fractionating column is control by lab made heating apparatus. Hydrogen peroxide and formic acid (2:1 ratio) were used at constant temperature and magnetic stirring rate in desulfurization process. The color of fractionated oil automatically changes by air oxidation. In oxidative desulfurization the sulfur are usually transformed into the sulfoxide and sulfone species and finally removed by extraction or adsorption. For further decolorization thermally activated bentonite powder is used [1, 10-13]. The details of this column and upgrading of waste tire pyrolysis oil are presented our previous research publication [1].

At our present research work the previous characterize upgrading (171-270) °C pyrolysis oil is analyzed by GC-MS and efficacy testing in a diesel engine.

Materials & Method: Tyre pyrolysis crude oil was collected from the pilot plant of Department of Mechanical Engineering, Rajshahi University of Engineering and Technology. The fractionation was performe from the Department of Applied Chemistry and Chemical Engineering, University of Rajshahi, Rajshahi-6205, Bangladesh. Where are the fractions (40-70) °C counts as petrol, (71-120) °C as naptha, (121-170) °C as kerosene and (171-270) °C as diesel and the remaining as residue. The (171-270) °C upgrading pyrolysis oil engine test is done from the Department of Mechanical Engineering, Rajshahi University of Engineering and Technology, Rajshahi-6204, Bangladesh; and the GC-MS analysis is perform in Designated Reference Institute for Chemical Measurement Lab, Bangladesh Council of Scientific and Industrial Research, Dhaka-1205, Bangladesh. The heat of the fractionating column is control by using 2000 watt heating coil; which is control by adjusting the voltage of voltage regulator.

In this analysis GC-MS column specification: HP-5MSI (Agilent J and W capillary column) 5% phenyl, 95% dimethyl polysilloxne (Length 30m Diam 0.250 mm, film 0.25 μm). GC parameter: 1μl sample was injected in split less mode. The inlet temperature was set at 250 °C and oven temperature was programmed as 60 °C for 0 min then 3°C/min to 240 °C 0 min. Total run time was 60 min and column flow rate 1 ml/min He gas. The aux temperature was set to 280 °C. MS parameter: the MS was set in scan mode. The ionization mode was EI (electron ionization) type. The mass range was set in the range of 50-550 m/z. MS Quad temp: 150 °C, MS source temp: 230 °C.

For the determination of calorific value the following bomb 1341 calorimeter is used. The 1341 Calorimeter is an improved version of a plain, static jacket, oxygen bomb calorimeter that has been made by Parr for more than eighty years. It is a reliable calorimeter that can be used for the same broad range of solid and liquid combustible samples as the 6000 Series. Its modest cost and simple design recommend it primarily for users whose work load does not justify the purchase of an automatic model. The calorimeter requires no permanent connections. It can be set up and ready to operate in a few minutes and, when not in use, can be disassembled easily and stores on a shelf.

The diesel engine used in this research was a single cylinder, water cooled, 4-stroke and 7.5 HP-rated powers. For determine engine Efficacy the engine speed was measured directly from the tachometer attached with the dynamometer. The dynamic fuel injection time is measured accurately by attached a burette of the engine. A stop watch was used to measure fuel consumption time for every 10ml fuel.

Engine efficacy is one of the main parameter of an oil fuel. To determine brake thermal efficacy the brake power (B.P) and calorific value (C.V) known is necessary. This parameter is determined by using the followings [14]. Engine brake power (B.P) is the power available at the crank shaft of an I.C engine. It is the product of the torque and angular engine speed.

$$B.P = \frac{2\pi NT}{60 \times 1000}$$

<div align="right">eq. 1</div>

Where, B.P $=$ Engine brake power in kW and N $=$ Angular speed of engine in rpm (rpm $=$ rotation per minute).

The percentage of brake thermal efficacy (η_{th}) of the engine is the ratio of the heat equivalent to one KW hour to the heat in fuel per brake power (B.P) hour. It is also known as overall thermal efficiency.

$$\eta_{th} = \frac{B.P \times 3600}{m_f \times C.V} \times 100$$

<div align="right">eq. 2</div>

Where, $m_f =$ Mass of fuel and C.V $=$ Calorific value.

GC-MS Analysis and Efficacy Test Result Discussion:

GC-MS analysis: The Gas chromatography (GC) and mass spectrometry (MS) make an effective combination for chemical analysis. Gas chromatography (GC) is used to separate mixtures into individual components using a temperature-controlled capillary column. Smaller molecules with lower boiling points are travel down the column more quickly than larger molecules with higher boiling point. The mass spectrometry (MS) is used to identify the various components from their mass spectra. Each compound has a unique or near unique mass spectrum that can be compared with mass spectral databases and thus identified. Through use of standards quantification is also possible. The (71-120) °C upgrading pyrolysis oil, (121-170) °C upgrading pyrolysis oil, (171-270) °C upgrading pyrolysis oil and conventional diesel fuel presented compounds are detected by GC-MS analysis.

The detected compounds and their percentage area compared to the total area of the chromatogram, which gives an estimate for their relative concentration in the pyrolyticoilsare shownat thetable 1, 2 and 3.And the conventional diesel is at the table 4. From GC-MS identified data of table 1 show the major following-cyclopropane, benzene, cyclopentadiene, cyclopentene, cyclohexadiene and 2,4 hexadiene compounds are

present in (71-120) °C upgrading pyrolysis oil.The following compounds and other rest compound remain in the table 1 show the nearest same physico-chemical properties of petroleum derived (71-120) °C gasoline fuel. The main identified data in table 2 are the followings- benzene, cumene and benzene derivatives. In this fraction (121-170) °C upgrading pyrolysis oil benzene concentration is highest than other remaining compound at the table 2. This benzene can be considered as a source of benzene which is generally done normally (121-170) °C obtaining naptha petroleum fuel.

But the assigned data in table 3the major compounds are- naphthalene, bezene, dl-limonine and 1H-indine. Among this total naphthalene and benzene is large concentration value; so the concentration of napthalene and benzene in (171-270) °C upgrading pyrolysis oil is bigger than other compounds presented in this oil fuel sample. This temperature range fractionated upgrading pyrolysis oil support the results obtain from FT-IR analyses in our previous research work. The identified compounds are mainly of unsaturated aromatics. The aromatic compounds are single and double ring alkyl aromatics and good thermal properties. This fraction oil also supports the nearly same physico-chemical properties of conventional diesel fuel. Which indicate the (171-270) °C upgrading pyrolysis oil can be further used as an alternative fuel instead of conventional diesel fuel. Otherwise most of the compound presents in upgrading pyrolysis oil have the nearly same physico-chemical properties.

By GC-MS identification of remaining (271-325) °C residue pyrolysis oil which is not physico-chemically treated give us un-desirable result. In this fraction the main identified compound is naphthalene and it derivatives; which have no better thermal properties, engine efficacy and other remarkable application.

For conventional diesel fuel the GC-MS data identify the following major following compounds are benzene, decane, undecane, nonane, dodecane, octane, naphthalene, tridecane, tetradecane and hexadecane. The most abundant compound present in the conventional diesel are pentadecane and hexadecane whose total concentration are 11.48 % and 10.50%. So the heavy hydrocarbon compound present in conventional diesel fuel by compare with (171-270) °C upgrading pyrolysis oil.

The factors which determine the internal engine performance; heating value is one of the most. The light hydrocarbon heating value is comparatively high than higher hydrocarbon. Here the heating value of conventional diesel fuel and (171-270) °C upgrading pyrolysis oil are 43.81mJ/kg and 42.79mJ/kg. The heating value of (121-170) °C upgrading pyrolysis oil is slight less than conventional diesel fuel. The water and ash content in (121-170) °C upgrading pyrolysis oil is responsible for such type of nature. From the recent article study it is shown the petrol, kerosene is higher heating value than conventional diesel fuel. And the burning spontaneity of lower hydrocarbon is better than heavy hydrocarbon. By considering this phenomenon the (171-270) °C upgrading pyrolysis oil burn easily and produces less smoke than conventional diesel fuel. But in the case of engine performance parameter the upgrading pyrolysis oil give slight les efficacies for the presence of bounded water and ash present in upgrading pyrolysis oil. To obtain better engine efficacy the upgrading pyrolysis oil need further purification.

Table 1. GC-MS analysis of upgrading (71-120) °C pyrolysis oil

R. time	Area (%)	Name of compounds
1.809	1.56	3-Butyn-1-ol,
1.846	2.58	Cyclopropane, 1,1-Dimethylcyclopropane
1.878	3.99	1,3-Cyclopentadien
2.028	1.92	1,3-Cyclopentadien, 2-Methyl- 1,3-Cyclopentadiene, 1-Methyl-1,3-cyclopentadiene
2.076	2.77	Cyclopentene, 1-Methy l-1-cyclopentene
2.130	0.61	Benzene
2.156	1.54	1,3-Pentadiene, 2,3-Dimethyl-1,3-pentadiene
2.199	2.71	Cyclopentene, 1,2 -Dimethyl-2-cyclopentene
2.253	2.23	1,3-Cyclopentadiene, 1,2-Dimethylcyclopentadiene
3.306	4.05	1,4-Cyclohexadiene, 1-methyl- 1,4-cyclohexadiene
3.338	2.59	2,4-Hexadiene, 2-Methyl- 2,4-Hexadiene
3.360	2.71	Toluene, Benzene, Methylbenzene, Methylbenzol
4.413	4.11	Cyclohexene, 1-Methylcyclohexene, 3-Methylcyclohexene
5.461	0.52	1,3,5-Hexatriene, 2-M ethyl-1,3,5-hexatriene
5.504	2.53	Cyclohexane, cis-1,3-Dimethylcyclohexane
5.605	1.20	Mesitylene
5.563	2.32	2,4 Hexadiene
5.600	2.82	2,4 Hexadiene
5.665	2.23	2,4 hexadiene
5.718	8.60	3 Hexene, Diethylacetylene
8.230	1.29	Cyclobutane
8.400	2.03	Cyclopentane
10.095	0.81	Fumaric acid

Table 2. GC-MS analysis of upgrading (121-170) °C pyrolysis oil

R. time	Area (%)	Name of compounds
2.413	0.36	Octane, n-Octane, Iso-octane, 2- Methyl-1,3-Cyclohexadiene
3.035	0.33	Cyclopentene, 1,2,3-Trimethylcyclopentene
3.460	0.36	Pyridine, 2-Methylpyridine
3.555	0.40	1,3-Dimethyl-1-cyclohexene, 1,3- Dimethyl cyclohexene
3.765	0.26	Cyclohexene, trans-3,5-Dimethylcyclohexene
3.870	0.45	Bicyclo-oct-3-ene, 4 -Vinylcyclohexene
3.911	2.29	Benzene, Ethylbenzene, Phenylethane, Ethylbenzol
4.165	7.39	Benzene, o- Xylene, o-Xylol, 3,4-Xylene, m-Xylene, m-Dimethylbenzene
4.590	0.55	Hexanenitrile, Hexanonitrile
5.090	0.41	2-Napthol
5.420	0.67	2-Pentanone
5.610	0.61	Ethylbenzene
5.815	0.50	1,6-Heptadien-3-yne
6.160	0.45	Cyclopentadien, 1,5,5-Trimethyl-1,3-Cyclopentadiene
6.230	3.03	Benzene, o- Xylene, o-Xylol, 3,4-Xylene, m-Xylene, m-Dimethylbenzene
6.450	0.50	Nonane, 2,5,5-Trimethyl-1,3-cyclopentadiene
6.695	0.28	Cyclohexanemethanol,4-ethylene cyclohexanemethanol
6.865	0.59	Benzene, Isopropylbenzene
7.205	1.31	2-Nitrocumene
8.050	0.34	1,3,6-Heptatriene, 2,5,6-Trimethyl-1,3,6-heptatriene
9.065	0.95	1H Indine
9.645	0.97	dl-Limonene
10.075	3.29	Benzene, p-Ethyltoluene
10.475	3.06	Cumene, 1,2,4-Trimethyl benzene
11.915	5.02	Benzene, 1,2,4-Trimethyl benzene

**Table 3.** GC-MS analysis of upgrading (171-270) °C pyrolysis oil

R. time	Area (%)	Name of compounds
3.020	1.00	1,2,4-Trimethylbenzene
4.186	6.03	Benzene, Cymol
4.491	6.79	dl-Limonene, Cyclohexene,
4.598	0.75	Benzene, Cyclopropane
5.165	0.97	Indene
5.807	0.54	Benzene, 1-methyl-3-propyl-Toluene
6.133	0.44	Oxirane, Cumene,
6.267	1.26	Benzene, 4-ethyl-1,2-dimethyl-benzene
7.615	1.31	Benzene, 1-methyl-2-(1-methylethyl)- benzene
8.027	1.34	Benzene, 2-Ethyl-p-xylene
8.150	0.23	Cyclohexene, 1,3-Cyclohexadiene
8.535	1.63	Benzene, Styrene,
9.230	0.65	O-Diethyl benzene
9.802	0.46	Benzene, 1,2,4,5-Tetramethylbenzene
10.139	0.51	Benzene, 2-ethyl-1,4-dimethyl-p-xylene
10.412	1.55	Benzene, 1,2,4,5-Tetramethyl benzene
11.940	0.56	1,5,9-Cyclododecatriyne
11.669	0.84	1H-Indene,
12.005	0.71	1,4-Dimethylazulene
12.252	1.96	2-Methylindene, 1H-Indene
12.418	1.15	2-Methylindene, 1H-Indene
12.610	0.74	2-Methylindene, 1H-Indene
12.787	1.00	2-Methylindene, 1H-Indene
12.870	0.40	Dodecane
13.691	1.59	Naphthalene, Naphthene
14.558	0.62	1H-Indene, 1,6-Dimethylindan, Benzene
15.25	1.12	Heptadecane
16.451	0.78	Benzothiazole, Benzosulfonazole
17.039	0.42	Naphthalene,
17.375	0.63	Heptadecane
18.553	1.66	1H-Indene, 1,3-Dimethyl-1H-indene
18.826	0.74	1H-Indene, 1,3-Dimethyl-1H-indene
21.243	2.89	Naphthalene, 1-Methyl naphthalene
22.179	1.53	Naphthalene, 1-Methyl naphthalene
23.244	0.36	Naphthalene,1,8-Dimethyltetrali 1H-Indene
26.346	1.15	1,2,3-Trimethylindene
27.630	1.71	Naphthalene,
28.405	1.93	Naphthalene, 1,6-Dimethyl naphthalene
29.106	1.93	Naphthalene, 2,7- Dimethyl naphthalene
29.384	1.74	Naphthalene, 1,8-Dimethyl naphthalene
37.850	0.59	Pentadecane
40.925	1.42	Naphthalene, 1,6,7-trimethyl naphthalene
43.963	3.35	Naphthalene, 1,4,6-Trimethyl naphthalene

Table 4. GC-MS analysis of conventional diesel fuel

R. time	Area (%)	Name of compounds
5.649	0.50	Octane, n-octane, Isooctane
8.275	0.61	Benzene, 1,3-dimethyl-m- xylene
9.810	1.28	Nonane, n-Nonane
11.597	0.59	Octane, 2,6-Dimethyloctane
13.046	0.84	Benzene, 1-ethyl-3-methyl- Toluene, m-Ethylmethylbenzene
13.218	0.80	Benzene, 1-Methyl-2-ethylbenzene, o-Ethylmethylbenzene
13.437	0.52	Nonane, 2-Methylnonan
13.817	0.76	Nonane, 3-Methylnonan
15.036	1.45	Benzene, 1,2,3-Trimethylbenzene, Cumene
15.694	2.79	Decane, n-Decane
17.090	0.81	Nonane, 2,6-Dimethylnonane
19.663	1.00	Benzene, 1-methyl 2-Propyltoluen
20.379	1.08	Decane, 3-Methyldecan
20.593	0.72	Benzene, 1,3-Dimethyl-4-ethylbenzene
21.000	0.80	Benzene, Cymene
22.251	0.75	Benzene, 1-Methyl-2-isopropylbenzene
22.481	4.07	Undecane, n-Undecane, Hendecane
23.380	0.69	Benzene, 1,2,4,5-Tetramethylbenzene
24.642	0.83	1-Methyl-2-Phenylcyclopropane
24.936	0.66	2-(4'-methylphenyl)-propanal benzene
26.530	1.17	Trans-1-methyl-2-indanol
26.899	1.05	Undecane, 2-Methylundecane
27.311	0.64	Undecane, 3-Methylundecane
29.451	5.36	Dodecane, n-Dodecane, Isododecane
30.274	1.19	Undecane, 2,6-Dimethylundecene
32.927	0.84	Undecane, 2,6-Dimethylundecene
33.382	1.01	Dodecane, 4-Methyldodecane
33.767	0.93	Dodecane, 2-Methyldodecane
34.168	0.95	Dodecane, 3,8-Dimethyldecane
34.297	1.27	Octane, 2,6-Dimethyloctane
35.174	1.25	Naphthalene, 1-Methylnaphthalene
36.265	7.61	Tridecane, n-Tridecane
36.891	1.06	Naphthalene, 1,2,3,4-tetrahydro-1, 4-Dimethyltetralin
37.260	0.84	Naphthalene, 1,2,3,4-tetrahydro-1, 5-Dimethyltetralin
39.966	0.90	Tridecane, 4-Methyltridecane
40.367	1.12	Tridecane, 2-Methyl-n-tridecane
41.089	2.02	Dodecane, 2,6,10-trimethyl- farnesane
42.774	9.57	Tetradecane, n-Tetradecane
43.330	0.85	Naphthalene, 2,6- Dimethylnaphthalene
46.652	2.01	Tetradecane, Methyltetradecane
47.042	1.21	Tetradecane, 3-Methyltetradecane
48.968	10.26	Pentadecane, n-Pentadecane
52.995	1.22	Pentadecane, 3-Methylpentadecane
54.825	10.50	Hexadecane, n-Cetane, n-Hexadecane

Efficacy Test: This efficacy test was performed by using 100% diesel and (171-270) °C upgrading pyrolysis oil. The performance parameter specially the engine break power and finally engine break thermal efficacy is calculated. The break thermal efficacy of the diesel engine is represented by following figure-
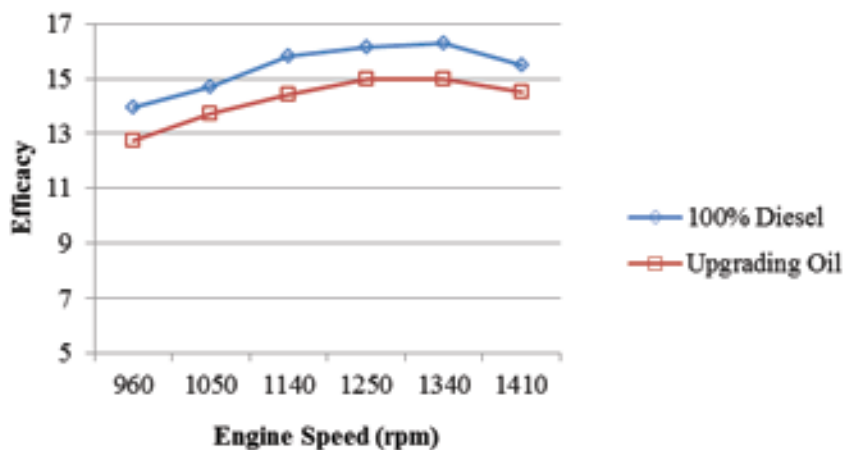
Fig.1: Variation of efficiency with respect to engine speed

It is seen from the figure that the brake thermal efficiency of the engine increases with an increase in engine speed up to 1250 rpm. This is due to the increase of fuel supply. This causes the increase of output power in the cycle. The brake thermal efficiency decreases with further increase of fuel supply consequently the increase of engine speed. The cause of decreasing brake thermal efficiency at an increased engine speed is insufficient amount of air supply. This causes improper burning of fuel in the combustion chamber. The engine speed of 1250 rpm at which the brake thermal efficiency reaches to a maximum value is chosen as standard for the experiments of (171-270) °C upgrading pyrolysis oil and conventional diesel fuel.

Conclusions: In this research we are very glad to perform such types of analysis by using modern instrument GC-MS and by obtaining the similarity of conventional diesel fuel and (171-270) °C upgrading pyrolysis oil. Both have about same composition, heating value, burning spontaneity, diesel engine efficacy and other relative properties. We also get comparatively same engine efficacy; but for a few undesired reason the engine efficiency slight less than conventional diesel fuel. Already we evaluate the previous and present study the ash and water content is responsible such types of undesired engine efficacy. The remaining upgrading fraction can also be tested by other types of petroleum engine. By considering all these results, it can be concluded that this upgrading pyrolysis oil can be used as any alternate fuel sources for various applications. If we get proper funding and further instrumental facilities than we try to perform upgradation of pyrolysis oil to increase engine efficacy by using upgrading pyrolysis oil.

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