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Waste Tyre Pyrolysis Oil: Production, Sulfur Removal and Fractionation for Value Added Products

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Abstract

Scrap tyres, one of the most common solid wastes, are increasing with the increase of vehicles number all over the world. Gradual depletion of fossil fuel requires a rapid shift towards alternative fuel sources including wastes. Globally, a lot of pyrolysis oil is generated from the management of waste tyres. The product Tyre pyrolysis Oil (TPO) has a parametric effect on the temperature, feed size, and reaction or pyrolysis time. This study explores the conditions for optimum pyrolytic oil yield (42% wt.) which was 500 ºC for 50 minutes, with a sample size of 4 cm^3. The obtained TPO were fractionated at various temperature ranges using a distillation column. The fuel characteristics of TPO, e.g., viscosity, flash point, pour point, calorific value, density, carbon residue are almost similar to that of the conventional oil standards but it cannot directly be used in the combustion process due to its higher sulfur content. For the removal of the sulfur compound, a various concentration ratio (10, 15, 20, 25, 30 percentages) of a mixture of H_2O_2 and HCOOH (2:1) solution (maintaining a pH of 4) were mixed with TPO. A maximum of 64.52% sulfur was removed from the TPO by using 25% of H_2O_2 + HCOOH solution and the pyrolysis gas was passed through the $Ca(OH)_2$ solution for wet scrubbing of SO_2, CO_2 and water vapor. The fractionated TPO (based on temperature range) provides valuable products e.g., bio-gasoline, biodiesel and other desirable chemicals. The production and eco-friendly use of TPO and its fractionated products can mitigate the disposal problem of waste tyre and help the eco-system to attain a sustainable environment.

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Introduction

In the recent years, the demand of energy and environmental pollution are of utmost concern, because of the rapid industrialization and population all over the world. Developing countries like Bangladesh need huge amount of energy to tackle the population's need. Globally, various efforts have been taken to meet the energy demand of power, transport and related sectors (Mia *et al.,* 2017; Unapumnuk *et al.,* 2008).

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Every year, more than 1.5 million tons of tyres generated from different tires and tube industries around the globe (Machin *et al.,* 2017) whereas Bangladesh produces about 120,000 tons per annum (Aziz *et al.,* 2017). In Bangladesh, every year, approximately 20.5 million automobile tyres are scrapped which accounts for 37% of the total tyre garbage production of the country. Typically, waste tyres are disposed by incineration and landfilling which have a tremendous adverse effect on the environment as well as on human health and limiting the land spaces (Jimoda *et al.,* 2018; Trongkaew *et al.,* 2011). For the utilization of waste tyres, various techniques like incineration, grinding, retreating, reclaiming, etc. have been applying, however, all of these have significant drawbacks (Kader *et al.,* 2015). Pyrolysis, burning of the substances in the absence of air, can be used for the production liquid oil (Tyre Pyrolysis Oil, TPO) from waste tyres which represents a source of energy that can be utilized as an alternative renewable fuel. This TPO can minimize the use of fossil fuel in combustion engine.

TPO is the mixture of different compounds e.g., aliphatic, aromatic, hetero-atomic and various polar fractions, having a calorific value is 41- 44 MJ/Kg, which is almost similar to that of the conventional fuels (Doğan *et al.,* 2012). The cetane number, a measurement of the quality or performance of diesel like fuel, of TPO reported to 40 - 44 while that of convention diesel fuel is 44 - 55. Although low cetane number of TPO shows poor thermal efficiency, rough performance in the engine and undesirable level of exhaust smoke and emissions (Martínez *et al.,* 2013), but, better homogeneity, low solid content and comparatively higher flash point of TPO are desirable (Hossain and Davies, 2013). The TPO produced from a variety of feedstocks and using different pyrolysis techniques, against those of fossil fuels. High acidity, the presence of solid particles, high water content, high viscosity, storage and thermal instability, and low energy content are typical characteristics of pyrolysis liquids. The TPO contains impurities like char which affect the engine performance and block the combustion chamber, piston ring grooves as well as exhaust valves of engines. The TPO also contains polycyclic aromatic sulfur hydrocarbons (PASH), which results in exhaust of SO_x gas during combustion (Bunthid *et al.,* 2010; Lewandowski *et al.,* 2019; Murugan *et al.,* 2008). TPO has a higher sulfur content compare to other petroleum products like diesel (Ilkiliç and Aydin 2011) which creates numerous problems including health hazards emission and air pollution during the utilization of TPO (Toteva and Stanulov 2020). The TPO used engine performance can be improved by decreasing the sulfur & moisture content from TPO (Ribeiro *et al.,* 2016; Hossain *et al.,* 2019). It is reported that the sulfur content of the initial pyrolysis product can be reduced by 34.25% when the TPO was treated with 5% $Ca(OH)_2$ (Aydin and Ilkiliç, 2012). The fractionation of TPO via distillation at atmospheric pressure can give various fractions (e.g., light, low-middle, high-middle, and heavy) and each fraction can be used for specific purposes (Campuzano *et al.,* 2021). However, very few studies explored the structural characteristics of each fraction. The characteristic behavior of various distillates at temperatures e.g., 30 - 80 ºC, 81 - 140 ºC, 181 - 220 ºC and 221 - 300 ºC as well as residues should require an in-depth study.

Although the growing interest in pyrolysis of tyres mainly focused on the parametric study of TPO production, not many studies have focused on the removal of sulfur and its related components from TPO and fractionation of TPO.

In this study, the optimum conditions e.g., pyrolysis temperature, particle sizes etc. for waste tyre pyrolysis as well as a method for the removal of sulfur based components from pyrolysis oil using a solution containing specific ratio of hydrogen peroxide and formic acid was explored. Furthermore, the fractionation of TPO at various temperature ranges using a fractional distillation column was studied to identify different grades fuels e.g., gasoline, diesel etc. obtained from TPO. The physico-chemical properties of the liquid fuel derived from waste tyres, fractionated products were determined and compared with commercial fuels.

Materials and Methods

Pyrolysis of the waste tyres

The scrap tyres, collected from various disposal sites of Dhaka City Corporation, Bangladesh, were chopped into three different sizes and cleaned with water thoroughly to remove any soil or extraneous elements. The pyrolysis of the scrap tyres were carried out in a pyrolyzer, which was made of stainless-steel having a length and inner diameter of 30.0 cm and 3.0 cm, respectively. The overall process for the TPO production from waste tyres are reported in our previously published article (Galib *et al.,* 2022). The TPO production experiments were conducted by varying the temperature within the range of 400 – 550 °C for different sizes of the raw materials. For each run, 300 - 500 g sample was loaded manually and Nitrogen (N_2) gas was passed into the reactor for 2 minutes to purge the reactor for the removal of inside air before pyrolysis started. Furthermore, a continuous supply of nitrogen gas was maintained to ensure inert environment inside the reactor and to move away the pyrolyzed product from within the reactor to the condensers. A two steps condensing system (first step in a closed condenser with counter current water condensation and second step in a cascade of glass condenser with ice condensation) was maintained to ensure the complete condensation of TPO. The condensed TPO were then collected and processed for further experiments. A simplified form of experimental set up with apparatus and process flow chart for waste tyres pyrolysis is shown in Fig. 1.

Desulfurization and de-colorization of TPO

The TPO contains high value of sulphur which should be removed before further use of it. An integrated technology to remove oxygenated compounds, sulphur and color from the TPO, a replication of the reported method by Khaleque *et. al.*, is illustrated in Fig. 2 (Khaleque *et al.,* 2016).

For extraction of oxygenated compound from TPO, N, N- dimethyl formamide, for desulfurization of TPO, a mixture of hydrogen peroxide (H_2O_2) and formic acid (HCOOH) solution (a ratio of 2 : 1) at various concentrations was used. For decolorization, activated bentonite powder was mixed with TPO. To accomplish the process, 100 mL of crude TPO was taken and various ratio of N, N- dimethyl formamide and kept in shaking for 2 hours at room temperature, followed by addition of H_2O_2 + HCOOH solution. Then, thermally activated bentonite powder was added to TPO at room temperature and the mixture was stirred at 700 rpm and kept for 10 minutes. The processed sample was then used for fractionation to receive value added products.

Fractional distillation of TPO

Measured volume of TPO (200 mL) TPO was taken in the round bottle flask and the sample in the flask

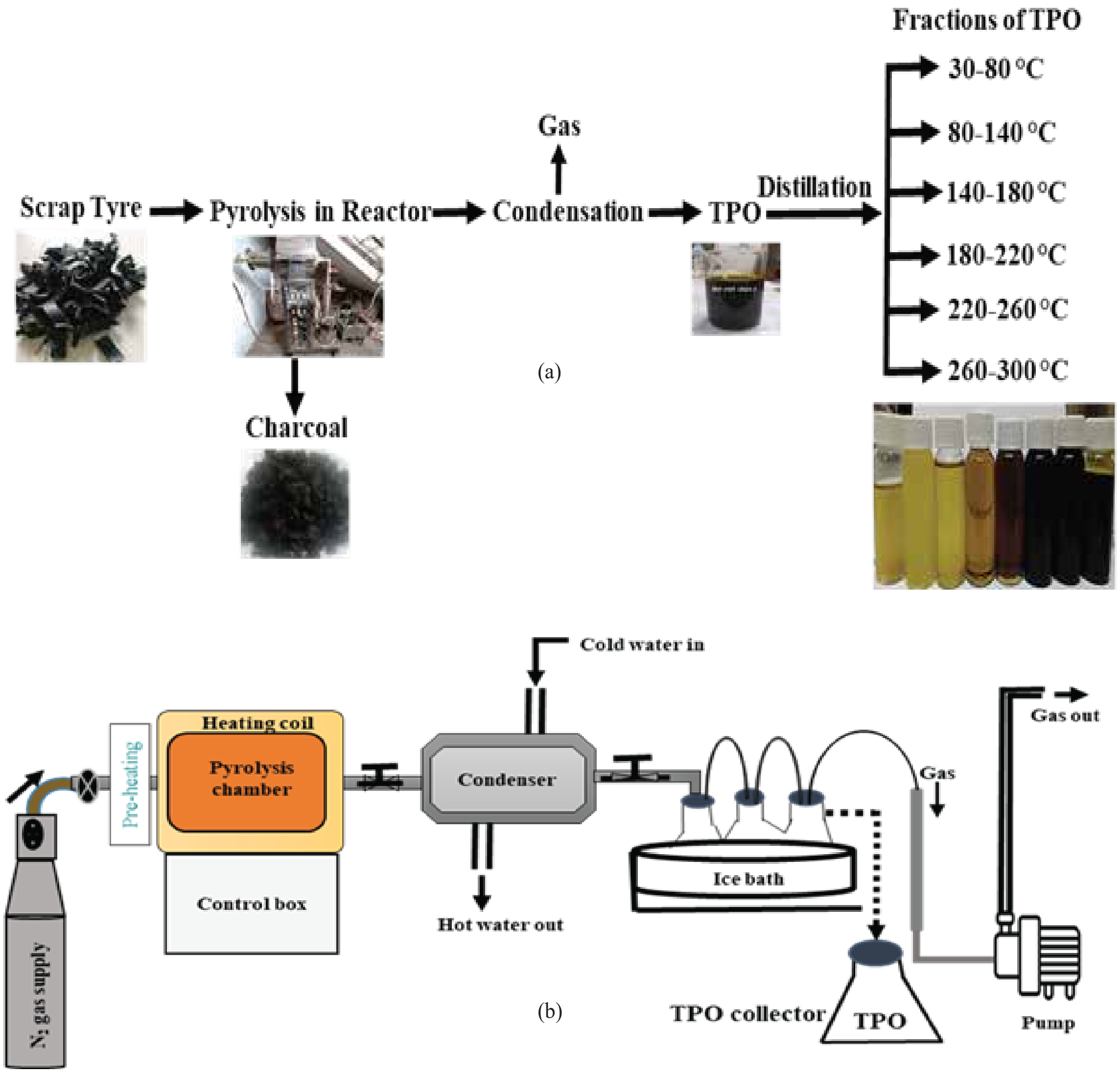

Fig. 1. (a) A schematic diagram of the overall experimental setup, and (b) A flow diagram of the pyrolysis process setup

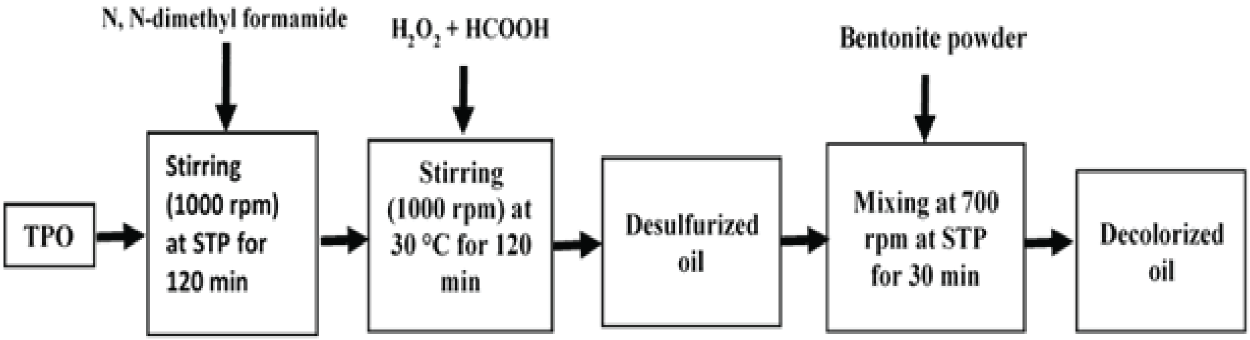

Fig. 2. A process flow chart of desulfurization and de-colorization of TPO

was fractionated using fractional distillation unit. Various fractions based on temperature range were passed through the condenser and collected as distillates. The heaviest (> 300 °C) fraction was collected as residue from the flask**.** The fractions were as follows: 30 - 80 °C, 81 - 140 °C, 141 - 180 °C, 181 - 220 °C, 221 - 260 °C, 261 - 300 °C, and residues at above 300 °C.

Characterization of TPO and its fractions

The physico-chemical properties e.g., viscosity index, density, flash point, pour point, carbon residue, ash content and sulfur content of TPO and its fractionated products from TPO were performed according to Institute of Petroleum (IP) and American Society for Testing and Materials (ASTM) test methods. The sulfur content of TPO and fractionated oil were measured by following IP 61/59 method. The presence of various functional groups in the TPO and fractionated oil were identified by FT-IR study using SHIMADZU FTIR 4500.

Results and Discussion

Optimization of tpo production: Effect of temperature, reaction time and particle sizes

The TPO products optimization depended on the temperature, reaction time and particles sizes of the tyre sample. In our previous study , it was reported that the maximum TPO production was 42 wt.% when the temperature, reaction time and particle sizes of tyres were 500 °C, 50 minutes and 4×2×0.5 cm^3, respectively (Galib *et al.,* 2022). Pyrolysis at low temperature and extended time promotes the secondary products, resulted in more char and less oil production (Hossain and Rahman, 2015). Pyrolysis at higher temperature (more than 500 °C) increases the heating value of volatile matters which decreased the yield of TPO but increased the yield of gaseous fraction because of the presence of secondary cracking reactions (Čepić *et al.,* 2021). Particle size of scrap tyre more than 4×2×0.5 cm^3 restricts homogenous heating and cannot get carbonized the core rubber which resulted in the decrease of TPO yield and increase of gas and char production. The following Table 1 illustrates the yields of various products at various reaction times when the reaction temperature was 500 °C and particle sizes was 4×2×0.5 cm^3.

Table 1. Yield of pyrolysis products at different reaction times (at 500 °C and particle size of 4×2×0.5 cm^3)

Temperature (°C) & Size of the scrap tyre (cm^3)	Reaction time (minutes)	Liquid (%)	Gas (%)	Char (%)
500 °C and 4×2×0.5 cm^3	30	36	10	54
	40	41	14	45
	50	42	14	44
	60	39	22	39

Desulfurization and de-colorization of TPO

For desulfurization, a mixture of hydrogen peroxide (H_2O_2) + formic acid (HCOOH) (at a ratio of 2 : 1) solution were mixed with TPO. Various concentration solutions e. g., 10%, 15%, 20%, 25%, 30% of the

TPO were used to oxidized the sulfur compound of TPO. The oxidizing compound was extracted by using different solvent such as acetone, ethanol, methanol, N, N- dimethyl formamide, but most effective compound was N, N- dimethyl formamide for extraction of oxygenated compound. A maximum of 64.52% sulfur content was removed by the use of 25% mixture of H_2O_2 + HCOOH in TPO as shown in Fig. 3. Fig. 4 shows the visual change of color of TPO before and after desulfurization and decolorization treatment.

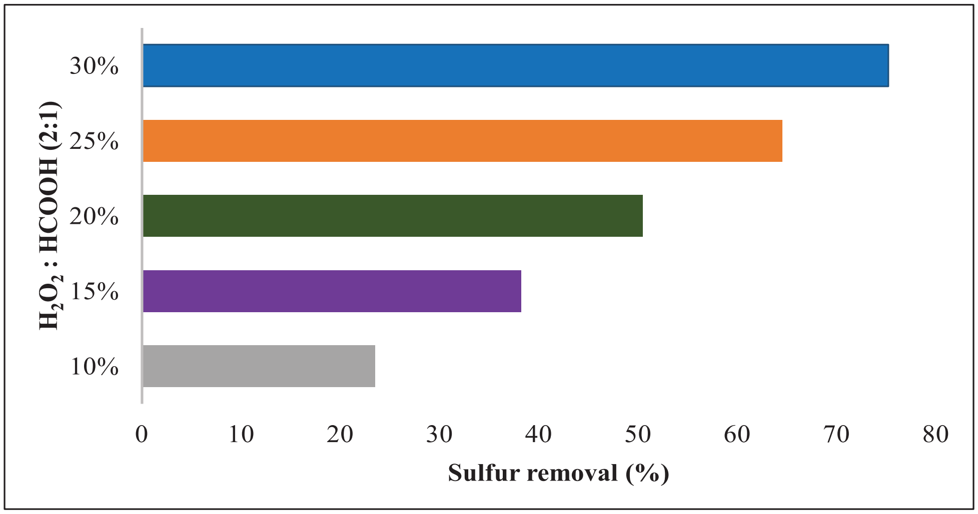

Fig. 3. Sulfur removal as a function of various concentration of H_2O_2 + HCOOH

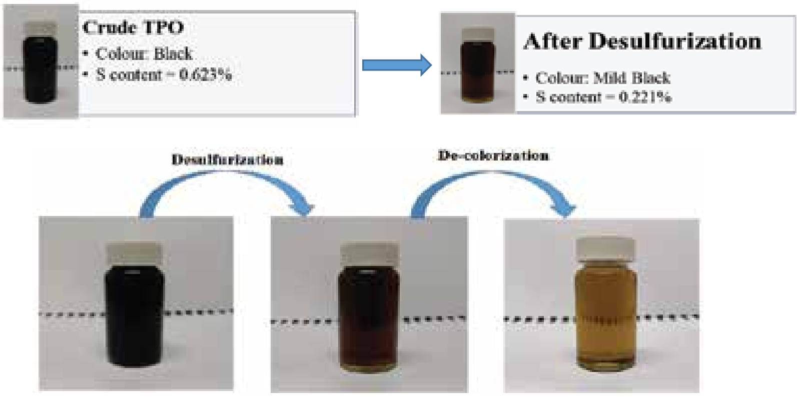

Fig. 4. A schematic representation of sulphur content in TPO and desulfurized TPO as well as color changes in decolorized TPO.

Fractional distillation of TPO

TPO was fractionated into seven parts, based on the temperatures, during distillation process. The fractions were as follows: 30 - 80 ºC, 81 - 140 ºC, 141 - 180 ºC, 181 - 220 ºC, 221 - 260 ºC, 261 - 300 ºC and residues above 300 ºC. Fig. 5 shows a photographic view of each fraction and Fig. 6 illustrates the amount of product obtained at the selected temperature ranges during fractional distillation.

Fig. 6 shows yields of the product fractions at various boiling ranges as stated above which was obtained from TPO by distillation process. The initial boiling point (IBP) of the TPO during distillation was found at 30 ºC. Then almost 40 vol.% of pyrolytic liquid were distilled out at a temperature within 180 ºC, 17 vol.% distilled out at 180-220 ºC and 20 vol.% distilled out at 220 - 300 ºC, respectively. These fractions were coincided with light naphtha, heavy naphtha and middle distillate as reported by Laresgoiti *et al.* 2004, where car tyre -derived pyrolysis liquids gives 20% light naphtha, 35% middle distillate and10% heavy naphtha and. The presence of middle distillates in TPO signifies the use of this fraction as diesel fuel and as heating oil (Ucar *et al.,* 2005). TPO distillation fractions have comparatively lighter products than that of commercial diesel fuels. The products obtained from distillation of TPO at 150 - 300 ºC may be atomized efficiently, allowing the combustion to begin at a lower temperature.

Fig. 5. Various fractions of liquid products distilled out from TPO

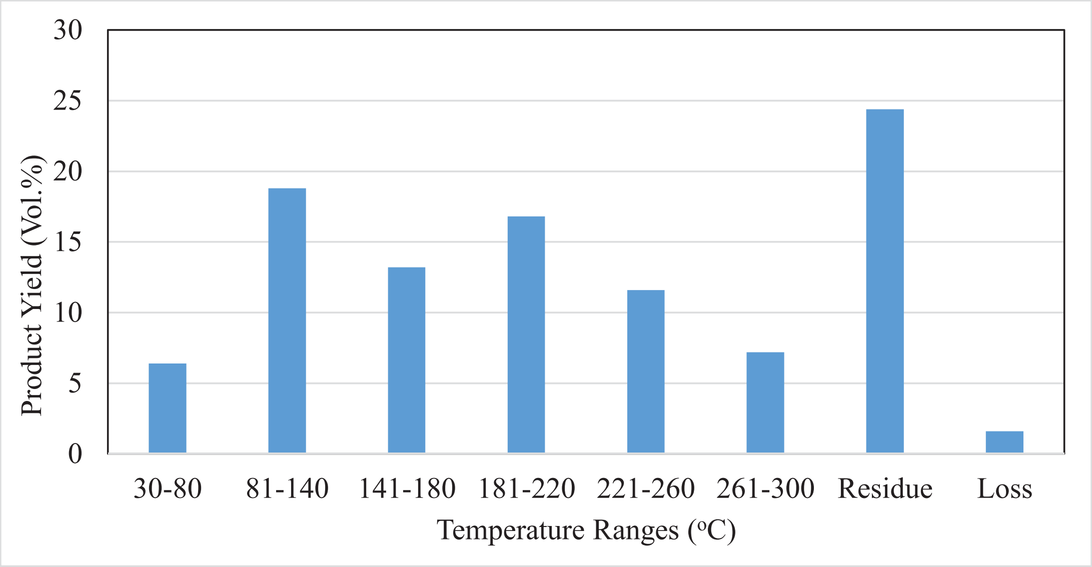

Fig. 6. Volume fractions of oil obtained at different temperature ranges during distillation

Physico-chemical Properties of TPO, its distillation fractions

A comparison of waste tyre-derived TPO fuel characteristics to that of the commercial diesel fuel and petrol/gasoline is shown in Table 2. Table 2. illustrated the physicochemical characteristics of TPO, its fractionated parts and their comparisons with commercial diesel and petrol. The density i.e., specific gravity of TPO was found 936.4 kg/m^3 which is higher than that of commercial diesel (820 - 845 kg/m^3) and petrol (720 - 775 kg/m^3). The density of the distillates 30-80 °C, 81-140 °C, 141-180 °C, 181-220 °C, 221-260 °C, 261-300 °C was 802.44, 847.98, 888.69, 903.31, 931.66 and 956.1 kg/m^3, respectively (at 15 °C) indicates that the heavier fractions have higher density.

Table 2. Comparison of the properties of crude TPO, its fractionated oil and conventional fuels

Properties	TPO	Upgraded fractions of TPO (°C)						Conventional Diesel	Conventional petrol
		30-80	81-140	141-180	181-220	221-260	261-300		
Density(kg/m^3) at 15°C	936.4	802.44	847.98	888.69	903.31	931.66	956.1	820-845 (Hönig 2015)	720-775 (Hönig *et. al.,* 2015)
Viscosity (at 40°C, cps)	4.6	-	-	1.6	2.1	3.3	5.7	6.06 (Prodhan *et. al.,* 2020)	4.4 (Gohary 2014)
Flash point (°C)	49	21	28.7	34.4	37.2	46.8	51.9	70 (Prodhan *et. al.,* 2020)	45(Hönig *et. al.,* 2015)
Fire point (°C)	95	72.1	81	130.5	155.2	167.5	174.2	76 (Ramnarayan, 2014)	-
Carbon Residue (%)	0.54	0.093	0.31	0.757	0.892	1.01	2.42	0.1 (Rao *et. al.,* 2021)	-
Ash Content (%)	0.031	-	-	-	0.012	0.022	0.041	0.01 (Rao 2021)	-
Pour point (ºC)	<-6	<-6	<-6	<-6	<-6			-20 (Prodhan *et. al.,* 2020)	-22(Gambo *et. al.,*2017)
Calorific value, MJ/kg	39.4	34.2	35.3	37.11	37.2	38.3	36.9	44.5 (Prodhan *et. al.,* 2020)	47.1 (Gambo *et. al.,* 2017)
pH	4.9	6.05	5.40	5.31	5.30	-	-	5.4	-
S (%)	1.623	0.039	0.282	0.777	0.536	0.555	0.614	0.11 (Prodhan *et. al.,* 2020)	0.015 (Fuel, *et. al.,* 2019)

The viscosity of TPO, 141-180 °C fraction and 261-300 °C fractions was found 4.6, 2.6 and 5.7 cps at 40 °C. The low viscosity of liquid is a favorable feature in handling and transporting of the liquid (Tüccar *et al.,* 2018). The flash point and pour point of the various fractions were comparable with commercial petrol, kerosene and diesel. The calorific value, a primitive property of the fuel, of the TPO and its various fractions was found comparable to that of the commercial petrol, kerosene and diesel indicates the potentiality of the use of tire derived oils as fuel. Carbon residues in the various distilled and residue product fractions increased with the increase of distillation temperature of TPO. Because of the carbon residue, fuel injector and cylinder scoring can be fouled within the engine which slow down the performance of any engine, even causes the failure of the functionality of engine (Thangarasu and Anand, 2019).

Functional Groups analysis (via FTIR) of TPO and derived fractions

The FTIR technique is generally used to recognize the functional group in the samples. Figure 7 illustrates the FTIR spectra of the various fractions of TPO obtained during distillation. The peaks indicates the presence of various functional groups of mainly alkenes, aromatics, ketones, alcohols and aldehydes. The absorbance peak of alkene group (=C-H) bending generally found at 730 - 770 cm^{-1}. For the various fractionated oils, it was observed at 731.02 cm^{-1}, 756.10 cm^{-1} and 752.24 cm^{-1}. The absorbance peaks of O-H bending & C-O stretching were found at 1300 - 1100 cm^{-1} in spectra of the fractionated oils. The peaks observed at 1039.03 cm^{-1}, 1045.42 cm^{-1}, 1026.13 cm^{-1}, 1092.07 cm^{-1}, 1274.95 cm^{-1} indicate the existence ether and related groups. Furthermore, peaks at 1778.37 cm^{-1}, 1897.95 cm^{-1}, 1899.88 cm^{-1} represents existence of the carboxyl (C=O) functional group in the TPO fractions. The presence of hydrocarbon groups C-H, C = C and alcohols in the TPO and its various fractions indicate that the liquid has potential to be used as fuel.

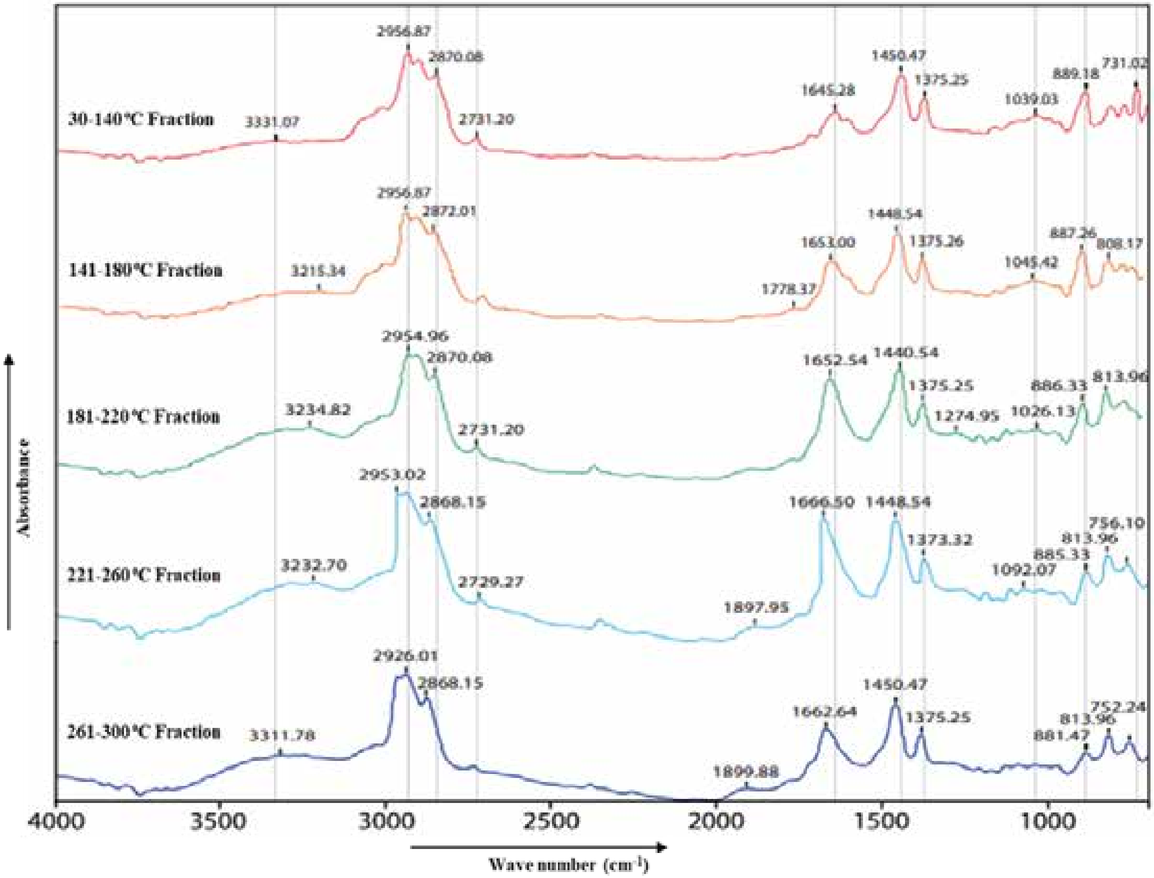

Fig. 7. FT-IR spectra of various fractions (obtained at different temperatures) of TPO

A comparison of the functional groups present in the different fractions (studied by FT-IR) is presented in Table-3.

Table 3. Comparison of FT-IR absorption bands of fractionated TPO at different temperature ranges

Peak at wave number (cm^{-1})	Fractionation range					Functional groups
	30 - 140 °C	141 - 181 °C	181 - 220 °C	221 - 260 °C	261 - 300 °C	
730-735	P	A	A	A	A	Alkene group (=C-H) bending
750-760	A	A	A	P	P	C-H bending
1000-1100	P	P	P	P	A	C-O stretching, S=O stretching,
1250-1300	A	A	P	A	A	C-O stretching, C-N stretching
1365 -1475	A	P	P	P	P	CH_2 and CH_3 bending
1450-1600	P	P	P	A	P	C=C stretching (Aromatic Rings)
1560 -1640	P	P	A	A	P	N-H bend (Amine Group)
1680-1630	P	P	P	P	P	C=O Stretching, N-H bend
1750-1800	A	P	A	A	A	C=O stretching
1850-1900	A	A	A	P	p	C=O stretching, C-H bending
2750 - 2850	P	P	A	P	P	C-H (Aldehydes) (doublet)
2850 - 3000	A	P	P	P	P	C-H stretching (Alkanes)
3200-3400	P	P	P	P	P	O-H group (Alcohol, Phenols)
3475-3150	P	A	P	A	P	N-H stretch

*A= Absent, P= Present

Conclusions

Commercially usable pyrolysis oil (TPO) was produced from waste tyre, its desulfurization and decolorization were performed and fractions of TPO at different temperature ranges were carried out. The study showed that the produced TPO and its fractions have a comparable properties of commercial fuels. The maximum TPO yield was found 42.0% from the waste tyre at the temperature of 500 °C while the reaction time and particle size were 50 minutes and 4cm×2cm×0.5cm. The desulfurization and decolorization process can successfully remove the most of the sulphur compounds and unexpected colors. The fractionated parts have comparable fuel properties. The physico-chemical properties and fractionation portion especially middle distillate fraction of TPO was similar with commercial diesel and other conventional fuels. The TPO needs to further purify before application because of higher sulfur content. The utilization of the TPO and their derivatives product will be significantly changed in the country's fuel economy.

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Authors Contribution

Mohammad Ismail (The corresponding author): Supervision, Conceptualization, Reviewing and Editing. Md. Galib Mahmud: Sample Collection, Experimental investigation, methodology development and thesis writing. Hd. Razu Ahmmed: Draft Preparation, Visualization. Md. Saiful Islam: Writing- Original draft preparation, Methodology, Reviewing. Kawnish Kirtania: Reviewing and Editing.

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