



# An Operating Condition For Maximizing Tyre Derived Liquid Fuel In Fluidized Bed Using Surface Response Methodology

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## ABSTRACT

Pyrolysis experiments using spent tyre feedstock were performed in a tubular transport reactor at atmospheric pressure under nitrogen atmosphere in a fluidized bed to maximize the yield of pyrolytic oil. The pyrolytic oil is storable, transportable and energy can be recovered easily. Investigations on the operating parameters were performed for various ranges of feed particle size (0.3-1.18mm), temperature (350°C–600°C) and feed rate (10-25 g/min). The experiments were planned using design expert 8.01 software according to the central composite design by keeping the sweep gas flow rate at 2 m<sup>3</sup>/h and the response in terms of percentage yield of pyrolytic products for each experiment was analyzed. The interdependency of the parameters was studied on the yield of the pyrolysis products and the maximum oil yield (44.90%) was obtained by experiment for feed rate of 13.55 g/min with average pyrolysis reaction temperature of 416°C and average feed particle size of 0.82mm. The pyrolytic oil yield obtained through the experiment at optimum conditions is in a proper proportion with the predicted value (45.09%). The fuel properties of the pyrolytic oil represented the broad properties of furnace oil.

**KEYWORDS:** Spent tyre, Fluidized bed, Electrically heated, Pyrolytic oil.

## ARTICLE INFO

### Article History

Received: 27<sup>th</sup> December 2015

Received in revised form : 29<sup>th</sup> December 2015

Accepted : 30<sup>th</sup> December , 2015

**Published online :**

**31<sup>st</sup> December 2015**

## I. INTRODUCTION

The disposal of solid residues from human activity is a growing environmental problem for modern society, especially in developing countries. One such residue is spent tyres, which are non-biodegradable. A possible solution would be to reuse them as raw materials in alternative processes. The complex nature of tyres makes it difficult to recycle them. In view of the exploding number of automobiles and the resulting tyre waste the pyrolysis process offers an ideal solution to an otherwise critical issue of waste disposal. Many researchers have attempted on different types of spent tyre pyrolysis such as atmospheric fixed bed batch Pyrolysis [3,13], fluidized bed pyrolysis [8] catalytic pyrolysis [12] vacuum pyrolysis [14], ablative pyrolysis [15]. Also incineration combustion, co-combustion with coal or other fuels, gasification, and

hydrogenation processes are attractive methods for recovering both energy and by-products (carbon black, activated carbon, etc.) from spent tyres. Pyrolysis basically involves the decomposition of the tyre rubber at high temperatures (300-900°C) in an atmosphere of an inert gas such as nitrogen or helium at atmospheric pressure. Three products are typically obtained from the rubber: gas, oil and char. The composition of each fraction depends on the pyrolysis conditions used and on the tyre composition. Pyrolytic oils (a mixture of paraffins, olefins and aromatic compounds) have a higher heating value and can be combusted directly or added to petroleum refinery feedstock. The gas fraction contains concentrations of methane, carbon monoxide, carbon dioxide, nitrogen, hydrogen and oxygen [9,10]. Pyrolytic char consists of carbon black and a mixture of products from the degradation of rubber compounds such as carbonised rubber polymer and nonvolatile hydrocarbons, and lower amounts of tyre rubber

additives such as zinc, sulfur, clays, and silica [7,15]. The inorganic constituents of spent tyres may end up as impurities or ash in the pyrolysis chars, or they may volatilize and become part of the liquid or gas product streams. The present work discusses on operating condition for maximizing tyre derived liquid fuel in fluidized bed using surface response methodology.

## II. EXPERIMENTAL

### 2.1. Pyrolysis Unit

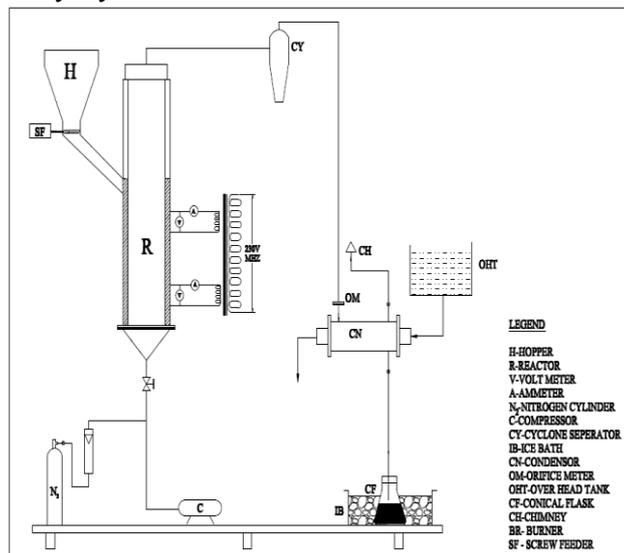


Fig.1. Electrically heated fluidized bed research reactor assembly

The Reactor (R) is made up of stainless steel tube of internal diameter 50mm. The reactor is filled with sand to a height of 30 cm with particle size in the range of 0.71-1mm in the perforated base for enabling fluidization. The bed is heated using electrical heaters connected to an ammeter (A) and autotransformer to control the heating. The reactor and the heaters are insulated with mineral wool. A temperature probe is kept inside the reactor, to measure the temperature at six different points. Fluidization is first done by air till uniform temperature is obtained in the bed and then with nitrogen. Compressed air is supplied through a compact heatless dryer and rotometer. The reactor is connected with a mercury manometer which indicates the fluidization in the reactor. The feeder unit is attached to the reactor. The unit consists of a hopper and a pulley driven screw feeder (SF) attached to variable speed motor. Raw material in powdered form is kept in the hopper and the screw feeder feeds the material in to the reactor. The cyclone separator (CY) is attached to the reactor which traps the particulates and char and prevents them from reaching the condenser. The condenser (CN) is of a shell and tube type. The condensate which is formed in the shell of the condenser passes through a tube and falls into the condensate receiver (CR) kept in an ice bath.

**2.2. Feed stock:** Spent tyre powder may be obtained by crushing the feedstock in a ball mill or from the waste produced during tyre retreading. This powder is fractionated into different sizes with the help of a sieve shaker in various ranges between 0.3-1.18 mm and used in the experiments.

**2.3. Experimental design:** The experiments were designed using the Central composite design of design expert software 8.01 for three variables in twenty experiments as follows:

X1 = Temperature (°C) = 350–600

X2 = Size for waste tyres (mm) = 0.3-1.18

X3 = Feed rate (g/mm) = 10-25

Experiments were performed using the test setup described in fig.1 according to the experimental plan to evaluate the yield of products fundamentally the solid char, liquid fuel and non-condensable gas. For each run the feeder was loaded to its maximum capacity and the feed rate was varied according to the experimental design keeping the sweep gas flow rate (2m<sup>3</sup>/h), for constant bed height of (30cm) and bed particle size (0.710-1.00mm). The heating system was switched on and maintained at the target temperature. In each experiment the yield of solid and liquid fractions were determined once the system had cooled to the room temperature. The gas yield was calculated from the material balance. The table 1 shows the experimental plan according to the central composite design.

TABLE 1: Design layout

Run	Temperature (A) (°C)	Particle size (B) (mm)	Feed rate (C) (g/min)
1	350	0.71	17.5
2	400	0.48	22
3	400	1	22
4	400	0.48	13
5	400	1	13
6	475	0.3	17.5
7	475	0.71	17.5
8	475	0.71	25
9	475	0.71	17.5
10	475	0.71	17.5
11	475	0.71	17.5
12	475	0.71	17.5
13	475	0.71	17.5
14	475	0.71	10
15	475	1.18	17.5
16	550	0.48	22
17	550	0.48	13
18	550	1	22
19	550	1	13
20	600	0.71	17.5

## III. RESULT AND DISCUSSION

The response of experiments conducted according to the central composite method was fitted to a second order polynomial mathematical model from which the optimum response was calculated. The software was also used for estimating the mathematical model representing the second order response surface fitted to the design points and the responses.

Multiple regression analysis of the experimental data gave the following second order polynomial equations:

In terms of actual factors

$$Y_{oil} = -32.00617 + 0.37493 * \text{Temperature} + 23.31042 * \text{Particle size} - 0.86137 * \text{Feed Rate} + 0.023785 * \text{Temperature} * \text{Particle size} + 1.05595E-003 * \text{Temperature} * \text{Feed rate} - 0.23570 * \text{Particle size} * \text{Feed rate} - 5.11276E-004 * \text{Temperature}^2 - 16.65127 * \text{Particle size}^2 + 0.010512 * \text{Feed rate}^2$$

In terms of coded factors for oil and other products

$$Y_{oil} = +41.09 - 5.52 * A + 1.53 * B - 0.74 * C + 0.46 * A * B + 0.35 * A * C - 0.28 * B * C - 2.82 * A^2 - 1.13 * B^2 + 0.22 * C^2$$

$$Y_{char} = +26.46 - 0.51 * A - 2.42 * B + 0.67 * C - 0.47 * A * B + 3.10 * A * C - 3.10 * B * C + 0.96 * A^2 + 3.74 * B^2 + 1.71 * C^2$$

$$Y_{gas} = +32.44 + 6.04 * A + 0.89 * B + 0.065 * C + 0.021 * A * B + 1.85 * A^2 - 2.61 * B^2 - 1.93 * C^2$$

Where Y is the response for oil, char and gas and A, B and C are the coded values of the test variables namely temperature, feed particle size and feed rate respectively.

TABLE 2: Results of experiments planned according to central composite rotatable design

Run No	Temperature (A)	Particle size (B)	Feed rate (C)	Pyrolytic oil	Char	Gas
	°C	mm	g/min	wt%	wt%	wt%
1	350	0.71	17.5	40.54	31.41	28.05
2	400	0.48	22	40.5	32.5	27
3	400	1	22	44.5	26.5	29
4	400	0.48	13	44	33.5	22.5
5	400	1	13	44.95	37.87	16.18
6	475	0.3	17.5	36.12	45.5	18.38
7	475	0.71	17.5	41.5	24.5	34
8	475	0.71	25	40.6	34.5	24.9
9	475	0.71	17.5	41.5	24.5	34
10	475	0.71	17.5	41.5	24.5	34
11	475	0.71	17.5	40.6	34.5	24.9
12	475	0.71	17.5	41.5	24.5	34
13	475	0.71	17.5	40	26	34
14	475	0.71	10	42.4	29.85	27.75
15	475	1.18	17.5	39.25	30.25	30.5
16	550	0.48	22	29.3	40.54	30.16
17	550	0.48	13	28.25	26.25	45.5
18	550	1	22	32	30.45	37.58
19	550	1	13	35.2	30.8	34
20	600	0.71	17.5	25.3	28.7	46

3.1. Effect of operating parameters: A series of graphs (fig.2-6) were plotted using data obtained from the second order polynomial equations based on the responses obtained through the experiments for the oil yield to study the effect of individual parameters on the oil yield.

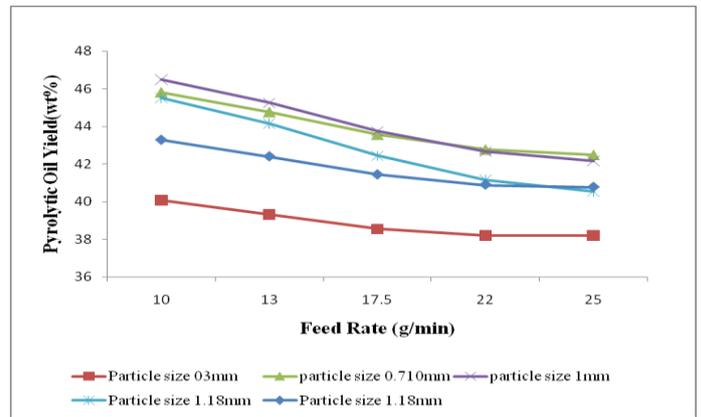


FIGURE.2. Effect of Feed rate on the oil for various particle sizes at constant temperature (416°C)

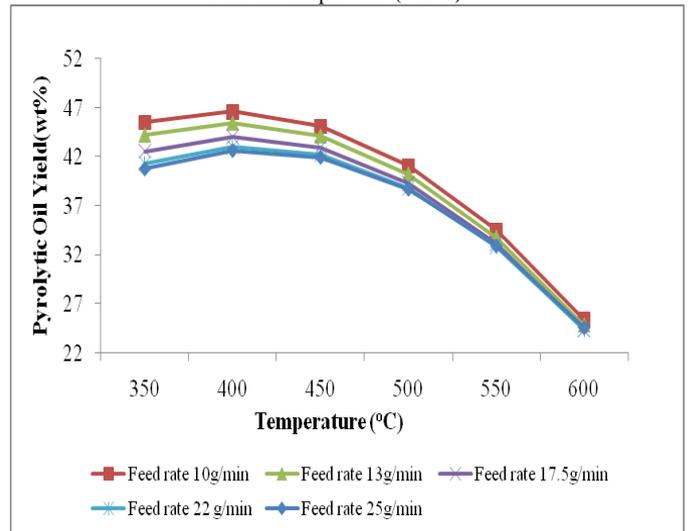


FIGURE.3. Effect of Temperature on the oil yield for feed rates at constant particle size (0.82mm)

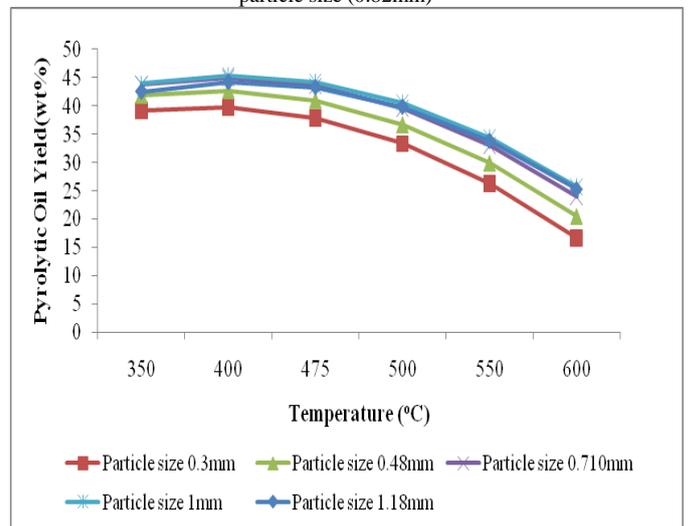


FIGURE.4. Effect of Temperature on oil yield for various particle sizes at constant feed rate (13g/min)

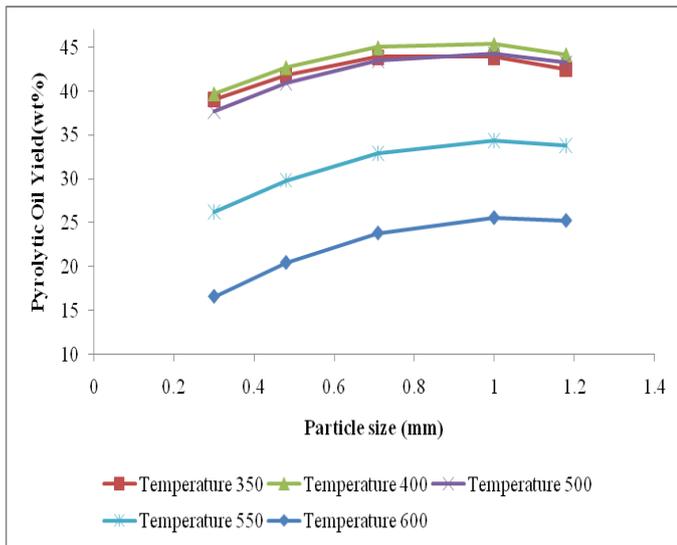


FIGURE.5. Effect of Particle size on Oil yield for Different Temperatures and at a fixed Feed rate (13g/min)

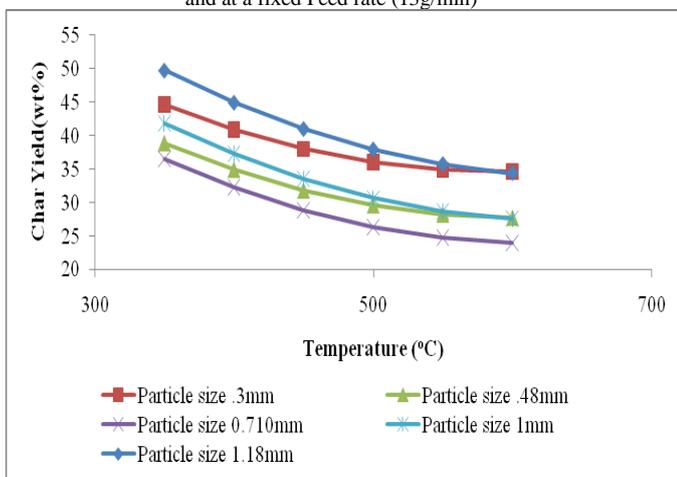


FIGURE.6. Effect of Pyrolysis Reaction Temperature on Char yield for different feed particle sizes at a fixed feed rate(13g/min)

Fig. 3 illustrates the relation between temperature and yield of pyrolytic oil. The pyrolytic oil yield passes through a maximum at temperature around 400°C and a high yield of oil 44.90% was obtained for an average temperature of 416°C by experiment. The predicted oil yield was 45.09% at the same temperature. Char is another product of tyre pyrolysis. The fig. 6 shows the relationship between temperature and char yield. As the temperature was increased, the char yield decreased and the gas yield increased (350°C-28.05% and 600°C-46.6%) for the same particle size and feed rate. At higher temperature (550<sup>0</sup> C) the char yield is so low and at lower temperature (350<sup>0</sup> C) the char yield is high. Just opposite trend was noticed in the gas yield which is similar to results obtained by other researchers [2.3]. The yield to gas increases as the yield to liquid decreases with increase temperature due to that there is a significant decrease in pyrolytic oil yield at higher temperatures(600<sup>0</sup> C) irrespective of the particle size and feed rate.

Fig. 4 shows the oil yield to different particle sizes. At higher particle size due to lower thermal conductivity of rubber, the effect of temperature is inferior for the large particles. Such an effect is correlated with the fact that heat can flow only to a certain depth in the large particles at the available pyrolysis time in comparison with almost complete thermal decomposition of the smaller particles

with longer residence time in fixed bed conditions. The production of gas is favoured by long residence time and high temperature. From the experiments it may be concluded that low oil yield is related with the particle size and as well as the temperature. The maximum yield of pyrolytic oil can be obtained at temperatures in the range of 400-425<sup>0</sup> C with relatively a higher feed particle size

Another parameter investigated on the influence of pyrolytic oil yield is feed rate Sample feed rates over the range 10-25 g/min showed that the oil yield is not only dependent on the particle size and reaction temperature but also on the feed rate. Increasing the feed rate may decrease the amount of energy available for heating each particle and likely to decrease the average temperature of the particles. If the particle size is very small average temperature would be very high resulting in high gas yield. Increasing feed rate may also have influence of the efficiency of heat transfer. It is inferred that at lower feed rates, higher pyrolytic oil yields can be obtained for larger feed particle sizes.

3.2. Effect of interdependency of operating parameters on the oil yield

The 3D response surface and the 2D contour plots are generally the graphical representations of the regression equation. Each contour curve represents an infinite number of combinations of two test variables with the other maintained at zero level. From the contour plots, it is easy and convenient to understand the interactions between two factors and also arrive at optimum conditions.

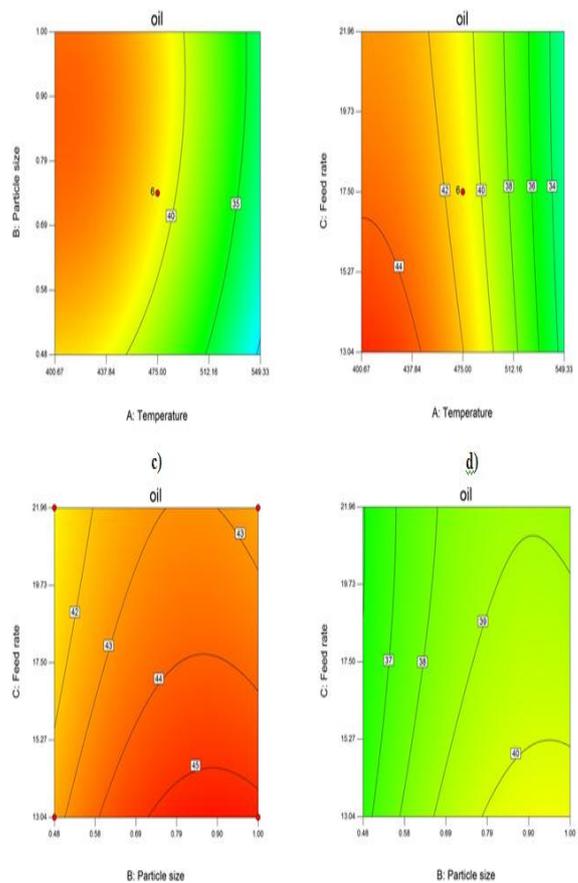


FIGURE.7. Contour plot of spent tyre pyrolytic oil yield(wt%): Effect of temperature and particle size (a) Temperature and feed rate (b) Particle size and feed rate constant temperature 400°C (c) Particle size and feed rate at constant temperature 500°C (d) Particle size and Feed rate.

The 2D contour plots are presented in fig. 7. The contour plot in fig. 7(a) shows that with the increase in particle size and decrease in temperature, higher yield of oil can be

obtained. It is observed from fig. 7(a) & 7(b) that there is a significant interaction between temperature, feed particle size and feed rate at the temperature range of 400 – 430 °C. It is also understood from fig. 7(c) & 7(d), that there is a significant influence of particle and feed rate at the temperature range of 400 – 430 °C and moderate influence of feed rate and particle size at higher temperatures (above 500°C)

**3.3. Contour plots for the optimized condition:** From this model the optimum conditions were determined and the 3 dimensional representation is presented below to show the effectiveness of each parameters on the production of pyrolytic oil.

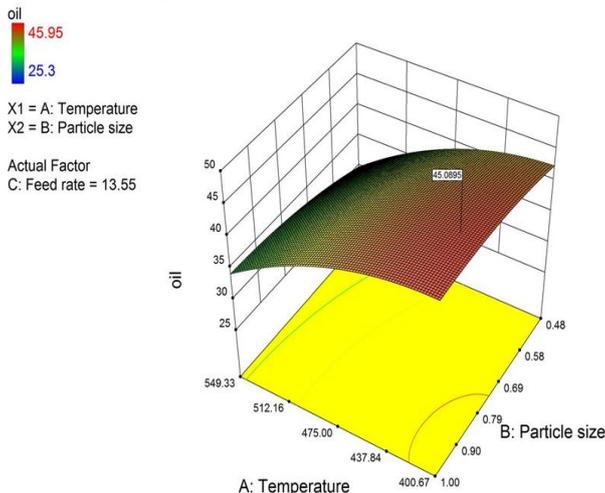


FIGURE.8. 3D Representation of various parameter

The optimal values of test variables in coded units are as follows:- A = 416.44 , B = 0.82 , C = 13.55 with the corresponding  $Y_{oil} = 45.0895$ . The results of the optimization are :-

- Temperature :- 416.44°C
- Particle size :- 0.82mm
- Feed rate :- 13.55 gm/min

At these optimum conditions the experimental pyrolytic oil yield is 44.90% against the predicted value of 45.09%.

### 3.4 Properties of pyrolytic product

The calorific value, carbon residue and the viscosity were measured for three samples at the lower, middle and upper ranges, investigated and tabulated as shown below.

**TABLE 3: Properties of Char and oil samples**

Sl No	Reaction Temperature	Average Particle size	Feed Rate	Viscosity	Calorific value	Carbon residue	Surface area of char (As received)
	<sup>o</sup> C	mm	g/min	Centipoises	(MJ/kg)	(%)	m <sup>2</sup> /g
1	350	0.71	17.5	14.9	36.8728	2.3	55.10
2	475	0.71	17.5	9.54	38.454	2.4	42.98
3	600	0.71	17.5	11.4	36.0197	2.8	35.4

**TABLE 4: Composition of gas**

Serial no	Component	350°C	475°C	600°C
1	Hydrogen (%)	0.57	11.64	13.072
2	Methane (%)	0.21	1.239	1.133
3	Nitrogen (%)	95.86	83.52	83.025
4	Oxygen (%)	3.2	2.55	1.887
5	Carbon dioxide (%)	0.13	0.621	0.799
6	Carbon monoxide (%)	0.11	0.413	0.08

## IV. CONCLUSION:

The evaluation of optimum operating conditions for a fluidized bed pyrolysis process of spent tyre using a computer program “Design Expert 8.01” was performed and the following conclusions can be drawn :-

- In view of the exploding number of automobiles and the resulting tyre waste the process offers an ideal solution to an otherwise critical issue of waste disposal.
- In case of tyre waste the process ensures practically zero effluents (except for a very small quantity of acid sludge which could be neutralized and disposed off is the sulphur in the oil is to be removed). The pyrolytic oil exhibited the broad properties of furnace oil.
- By recovering valuable pyrolytic oil, this process could save large amounts of petroleum fuels. The production of carbon black may be used for low end applications such as those for fillers in the plastic industries if it is not upgraded.
- The optimum conditions for maximum pyrolytic oil yield are: temperature, 416<sup>o</sup> C, average particle size, 0.82, feed rate 13.55 g/min under the sweep gas flow rate of 2 m<sup>3</sup>/h and sand bed height of 30cm.
- Quantitative relationships could be obtained between the experimental variables and the yield of oil.

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