

FAST PYROLYSIS FOR BETER UTILIZATION OF TARMARIND SEED FROM RENEWABLE ENERGY POINT OF VIEW

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Abstract- The conversion of tamarind seeds into pyrolytic oil by fixed bed fire-tube heating reactor has been taken into consideration in this study. The major components of the system were fixed bed fire-tube heating reactor, liquid condenser and collectors. The raw and crushed tamarind seed in particle form was pyrolyzed in an electrically heated 10 cm diameter and 27 cm high fixed bed reactor. The products are oil, char and gases. The parameters varied were reactor bed temperature, running time, gas flow rate and feed particle size. The parameters were found to influence the product yields significantly. The maximum liquid yield was 45 wt% at 400°C for a feed size of 1.07cm³ at a gas flow rate of 6 liter/min with a running time of 30 minute. The pyrolysis oil was obtained at these optimum process conditions were analyzed for physical and chemical properties to be used as an alternative fuel.

Keywords: • Pyrolysis, Fixed bed, Tamarind seed, Renewable energy, Biofuel

1. INTRODUCTION

Agricultural waste is one of the members of biomass, which is an attractive renewable energy resource because it is widely dispersed and could contribute zero net carbon dioxide emission to the atmosphere [1]. Biomass can be used as raw material for pyrolysis to generate liquid, gaseous and solid fuels. Pyrolysis conversion technology for biomass has been investigated over the last three decades and is used commercially [2]. Pyrolysis produces gas, vapor that can be collected as liquid and solid char. The yields of each of these products depend on process conditions [3]. Solid char has a good gross calorific value (GCV) of 17-36 MJ/kg [4] and may be used to provide heat for pyrolysis process itself. Solid char can also be used as a fuel either directly as briquettes or as char-oil or char-water slurries or it can be used as feedstocks to prepare activated carbon. The pyrolysis gas fraction contains high concentrations of CO₂, CO, H₂, CH₄, C₂H₄, C₃H₆, and other hydrocarbons with a GCV of 6.4-9.8 MJ/kg and can be used to provide process heat, recirculated as an inert carrier gas or exported for applications such as feed drying. The usages of char and gas products in the pyrolysis process itself reduces production costs for the heating system. Bio-oil derived from agricultural wastes has moderate GCV ranging from 15-38 MJ/kg with some advantages in transportation, storage, combustion, retrofitting and flexibility in production and marketing [5]. The liquid product, is useful as a fuel, may be added to petroleum refinery feedstocks or upgraded by catalysts to produce

petroleum grade refined fuels. The bio-oil is a mixture of about 300 types of major and minor organic compounds that belong to acids, alcohols, ketones, aldehydes, phenols, ethers, esters, sugars, furans, nitrogen compounds and multifunctional compounds [6]. Bio-oil has several environmental advantages over fossil fuels as a clean fuel. It is CO₂/GHG neutral. Therefore, bio-oil can generate carbon dioxide credits. No SO_x emissions are generated, because plant biomass contains insignificant amounts of sulfur. Thus, bio-oil would not be subjected to SO_x taxes. Bio-oil fuels generate more than 50% lower NO_x emissions than diesel oil in a gas turbine. The tamarind, a slow-growing, long-lived, massive tree reaches, under favorable conditions, a height of 24-30 m, and may attain a spread of 12 m and a trunk circumference of 7.5 m. Tamarind grows in many part of the tropical based climate of the world which is suitable for its production. About 35-40 wt% of tamarind is generated as biomass solid waste in form of seed and shell. The tamarind was certainly introduced into tropical America, Bermuda, the Bahamas, and the West Indies much earlier. In all tropical and near-tropical areas, including South Florida, it is grown as a shade and fruit tree, along roadsides and in dooryards and parks. Mexico has over 10,000 acres of tamarinds, mostly in the states of Chiapas, Colima, Guerrero, Jalisco, Oaxaca and Veracruz. There are commercial plantings in Belize and other Central American countries and in northern Brazil. In India there are extensive tamarind orchards producing 250,000 metric tons annually. The climate of Bangladesh is also suitable for growing tamarind plants. Here the

total production is almost 12,000 metric tons per year. The amount of seed is almost 4800 metric tons (40 wt% of fruits). The weight percentage of seed is very high but it has not any important usage. The seeds are considered as waste material. A mature tree may annually produce 150-225 kg of fruits, of which the pulp may constitute 30 to 55 %, the shells and fiber, 11 to 30 %, and the seeds, 33 to 40 %. Thus, an endeavor from the point of view of the energy recovery from this tamarind seeds by thermochemical process may be worthwhile. Usually oil is extracted from oil seeds by crushing it in oil mill, but oil extraction from tamarind seeds is not possible by this method. For this reason pyrolysis technology is used to derive oil from tamarind seeds. Characterization studies on tamarind seed have been carried out through proximate, ultimate and thermo gravimetric (TG) analysis. The result of these analyses showed that tamarind seed has high potentials to produce liquid fuel by pyrolysis conversion process. Although pyrolysis of biomass has achieved commercial status, there are still many aspects of the process which are largely empirical and require further study to improve reliability, performance, product consistency, product characteristics and scale-up. A quite number of the previous research groups worked for the potential recovery of fuels and chemicals from different agricultural residues and by-products have already been studied, but most of them have focused on the effect of reactor temperature on product yields and product compositions. Ablative, circulating fluid bed, fixed bed, cyclone, fluid bed, and entrained flow reactors, but not fixed-bed reactors with fire-tube heating have been studied. Therefore, the objective of this study was to develop a new heating system for pyrolysis for recovery of liquid hydrocarbons from biomass solid wastes. A fixed-bed fire-tube heating pyrolysis reactor system was designed and fabricated considering all the process parameters. The available biomass solid waste, tamarind seed, was pyrolyzed in the internally heated fire-tube heating reactor system under N₂ atmosphere. The effects of operating temperature, feed size and vapor residence time on the product yields were investigated. The whole pyrolysis liquids obtained at optimum operating conditions were characterized by physical properties, GCV, FT-IR and distillation.

2. MATERIALS AND METHODS

2.1 Materials

The ripe tamarind fruits were collected locally in Rajshahi and seeds were separated. The size of raw seeds is almost 1.5 cm³. The seeds were crushed and sieved to the sizes 0.20 cm³, 0.60 cm³, 1.07 cm³, 1.52 cm³ and oven dried to remove moisture for 12 hours at 110°C prior to pyrolysis. The proximate and ultimate analysis and gross calorific values of the tamarind seeds are presented in Table 1.

Table 1. Proximate and ultimate analysis, and gross calorific values of solid tamarind seed

Biomass samples	Proximate Analysis (wt%)				Ultimate analysis (wt%)					GCV (kJ/kg)
	Ash	Volatile	Fixed carbon	Moisture	C	H	O	S	N	
Tamarind seed	1.8-2.5	65-70	12-22	4.9-5.9	45.76	9.59	44.39	0.04	0.22	18108.3

2.2 Experimental set-up and procedure

The schematic diagram of the experimental set-up is presented in elsewhere [7]. The experimental unit consists of a fixed-bed fire-tube heating reactor chamber, a gravity feed type reactor feeder, two ice-cooled condensers, N₂ gas cylinder, N₂ gas pre-heater, an air compressor, char collection bag and thermocouples with temperature controller. The reactor chamber was heated internally by fire-tubes containing insulated electric coil. In each pyrolysis run, by the action of gravity force a quantity of 300 (±2.0) gm of tamarind seed sample was supplied from the feeder into the reactor chamber by opening the feed control valve. Then the reactor was purged before experiments by the flow of N₂ gas of 6 l/min for 5 min to remove the inside air. The reactor heater (power system) and LPG burner were switched on, and the temperature of the reactor was increased to a desired value of 300, 350, 400, 450, 500 or 550°C. During pyrolysis of tamarind seed a reddish brown visible vapor usually flared into the atmosphere. When the decomposition was completed colorless (no visible) N₂ gas came out from the reactor. The colorless flaring was the significance of the completion of the thermal decomposition of the tamarind seed. The reaction time was 50 min for every of all pyrolysis runs after which usually no further visible vapor product came out. Nitrogen gas was supplied in order to maintain the inert atmosphere in the reactor and also to sweep away the pyrolyzed vapor product to the condensers. Pyrolysis vapor product was passed through two sets of condenser pipes to quench into liquid and then collected in the reagent bottles. The uncondensed gases were flared to the atmosphere. The bottles were completely filled up with liquid so that no air could be trapped into the bottles. When pyrolysis of the feed material in the reactor was completed, the vapor exit port was closed and the reactor heater and LPG burner were switched off. N₂ gas supply was also stopped. After cooling down the system, the char product was pushed out from the reactor chamber with the aid of compressed air supplied from the air compressor by opening char exit port. Char was collected in the char collection bag and weighted. The liquid was then weighted and gas weight was determined by

subtracting the liquid and char weight from the total weight of feedstock. Afterwards, the system had been made ready for the next run just repositioning the valves. Initially the pyrolysis were performed by varying the temperature within the range of 300–550 °C at every 50 °C for a particular feed size and vapor residence time. Once the temperature of maximum liquid yield (400 °C) was selected, additional tests were conducted at the optimum temperature by varying the feed size and the vapor residence time to find out the optimum process conditions.

2.3 Pyrolytic product liquid analysis

Pyrolytic liquids obtained under the maximum liquid yield conditions were well mixed and homogenized prior to analysis being made. Some physical properties of pyrolytic liquids including density, viscosity and GCV were determined by using the standard methods ASTM D189, ASTM D445, and ASTM D240, respectively. The functional group compositions of the product liquids were analyzed by Fourier Transform InfraRed (FT-IR) spectroscopy. The FT-IR instrument of model PERKIN ELMER FTIR 2000 was used to produce the ir-spectra of the derived liquids.

3. RESULTS AND DISCUSSIONS

3.1 Pyrolysis product yields

3.1.1 Effect of reactor temperature on the product yields

The percentage yields of the pyrolysis products, liquids, solid char, and gases obtained from at a temperature range between 300-550 °C, feedstock size of 1.07 cm³ and gas flow rate of 6 L/min are presented in Fig. 1. The Figure shows that when the temperature increased from 350 to 555 °C, the yield of liquid increased first to a maximum value at 400 °C, and then decreased to a minimum value at 550 °C. The gas yield increased over the whole temperature range, while char yield decreased over the whole temperature range. It is apparent that a fairly sharp temperature optimum exists at which maximum yield of liquid was achieved probably due to strong cracking of the selected crushed tamarind seeds at this temperature. The tamarind seed sample was not totally decomposed at a temperature less than 400 °C. The thermal decomposition of the sample increased (*i.e.* solid yield decreased) up to a temperature of 400 °C and hence increased the liquid and gas yields. In the temperature range of 400-550 °C, solid yields were almost equivalent to those obtained by TGA of tamarind seed at around 400 °C, which are presented elsewhere [8]. Solid char consists of a small amount of volatile hydrocarbons, solid hydrocarbons, and inorganic compounds. Therefore, there is no obvious mechanism for char loss with increasing temperature; only higher temperatures volatilize some of the solid hydrocarbons of the char. In competition with char loss reactions, a certain amount of char- or coke-like carbonaceous material is formed due to secondary reactions among the

polymeric compounds, derived primarily from depolymerization and fragmentation reactions of three key biomass building blocks: cellulose, hemicellulose, and lignin [9]. Since solid yields do not decrease significantly in the temperature range of 400-550 °C, it may be concluded that tamarind seed decomposition is complete and carbonaceous material has been formed. The decrease in liquid yields and increase in gas yields above a temperature of 400 °C is probably due to decomposition of some oil vapors into permanent gases, and secondary carbonization reactions of oil hydrocarbons into char. The increase in gas yields at higher temperatures is also caused by char loss reactions. Thus, at higher temperatures the gas yields gradually dominated; however, 400 °C seems to be the optimum temperature to obtain liquid products from thermochemical conversion of solid tamarind seed by pyrolysis, since decomposition is complete and liquid yields become maximized. Pyrolysis product yields and their distributions over the whole range of temperature depend not only on feedstock composition and operating temperature, but also on the specific characteristics of the system used, such as size and type of reactor, efficiency of heat transfer from the hot reactor surface to and within the biomass, feed particle size, and vapor residence time. Therefore, results from different studies are difficult to compare. Studies on fluidized-beds and fixed-beds [10] resulted in product distributions similar to those of the presented study. Solid yield decreased and gas and liquid yields increased up to an intermediate temperature. At increasing temperatures, solid yields remained almost constant, gas yields increased and liquid yield decreased. The liquid yields obtained in the presented study are slightly lower and char yields are higher compared to those of previous studies [11]. These variations are mainly due to compositional differences (moisture, volatile, fixed carbon and ash content) in biomass feeds and quality of pyrolytic product liquids. A higher volatile content of feedstock with lower amounts of fixed carbon and ash is desirable for higher percentage of liquid yields in the pyrolysis process. Higher amount of fixed carbon and ash content of a feedstock contribute to char formation. Moreover, water in bio-oils originates from the original moisture in the feedstocks and dehydration reactions occurring during pyrolysis [9]. Feedstock used (Table 1) in the presented study was of a lower volatile and moisture and a higher fixed carbon and ash content compared to that used in earlier studies. Some research groups [4,11] used an additional cooling solvent to quench pyrolytic vapors in the subzero range, while the product liquids contained 15-35wt% of water.

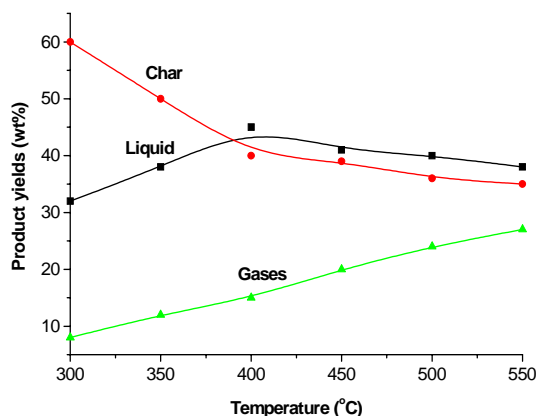


Fig. 1. Effect of temperature on product yields for feed size 1.07 cm³ and N₂ gas flow rate 6 l/min

3.1.2 Effect of feed size on the product yields

The effect of feed size on product yields under optimum reactor temperature and for a N₂ gas flow rate of 6 l/min is presented in Fig. 2. The figure shows that liquid yield first increased up to a maximum value for feed sizes of 1.07 cm³ and then decreased for larger feed size while the char yield increased and gas yield decreased for all particle sizes above 1.07 cm³. The thermal conductivity of biomass is very poor (0.1 W/m² °C along the grain, 0.05 W/m² °C cross grain), and hence biomass particles have to be very small to allow rapid heating to achieve high liquid yields. Theoretically, particle size has a great effect on pyrolysis product yields. It is generally assumed that a decrease in particle size will tend to generate higher liquid product yields [12]. During pyrolysis, a high heating rate (up to 10000 °C/s) may be achieved with a thin reaction layer but the lower thermal conductivity of biomass prevents such a heating rate through the whole particle. Smaller feed sizes provide more reaction surface and allow a high heating rate, but the decomposition of the biomass feed occurs too quickly. There is sufficient time for secondary reactions in the oil vapors and consequently gas yields increase and liquid and char yields decrease. In contrast, the heating rate in larger feed particles is low due to their lower thermal conductivity, and heat can flow only to a certain depth in the available pyrolysis time. Thus, the cores of the larger particles become carbonized and/or cannot be decomposed completely resulting in an increase in char yield and a decrease in liquid and gas yields. Pyrolysis experiments performed by Putun et al. [12] in a fixed-bed reactor suggested that particle size had little effect on product yields. In our study, we observed the same. For particle size 1.07 cm³, decomposition proceeds to completion and there is less of a chance for secondary cracking at the optimum reactor temperature and a gas flow rate 6 l/min.

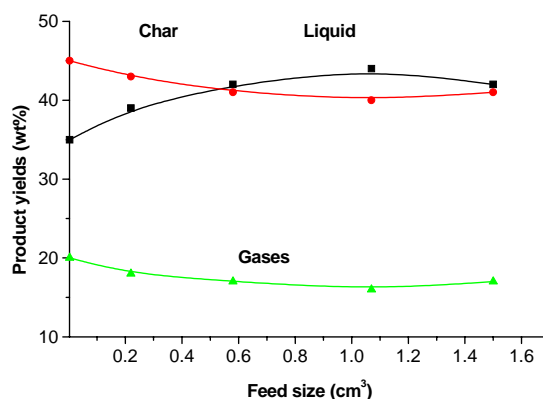


Fig. 2. Effect of feed size on product yields for temperature 400°C and N₂ gas flow rate 6 l/min

3.1.3 Effect of N₂ gas flow rate on the product yields

The effects of gas flow rate on gas, char, and liquid yields for optimum reactor temperature and for optimum feed size are shown in Fig. 3. When the flow of N₂ gas from cylinder was increased, the vapor residence time decreased, and the pyrolysis vapor product removal rate of biomass feed from the hot zone increased. It was observed that at the beginning with lower gas flow rate (2 l/min), the liquid product was 38 wt% of biomass feed with a char product of 40 wt% and a gas yield of 22 wt%. With the increase of N₂ gas flow rate up to 6 l/min, the liquid product was increasing while gas yield decreasing. The char yield appeared to be more or less constant. This may be due to the fact that at lower gas flow rate the pyrolysis reaction could not take place properly. When the gas flow rate was increased to values higher than 6 l/min for particle size 1.07 cm³, liquid product decreases from 45 to 36 wt% of biomass. The increase in gas yields with decreasing gas flow rate (increasing vapor residence time) is due to the decomposition of some oil vapor into secondary permanent gases. Primary vapors are first produced from pyrolysis of biomass sample at optimum reactor temperature and the primary oil vapors then degrade to secondary gases {for instance: oil vapors → heavy hydrocarbons + light hydrocarbons (CH₄ + C₂H₄ + C₃H₆ +) + CO + CO₂ + H₂} within the period of higher vapor residence time, which leads to less oils and more gaseous products. Besides, long contact time between the volatiles and the char leads to another parallel secondary pyrolysis reaction (for instance: C + CO₂ → 2CO) and hence reduces in char yields. Our results are in good agreement with those of other studies [12,13]. Our study also indicated that product yields varies little in relation to the feed sizes and sweeping gas flow, but are affected significantly by reactor operating temperatures.

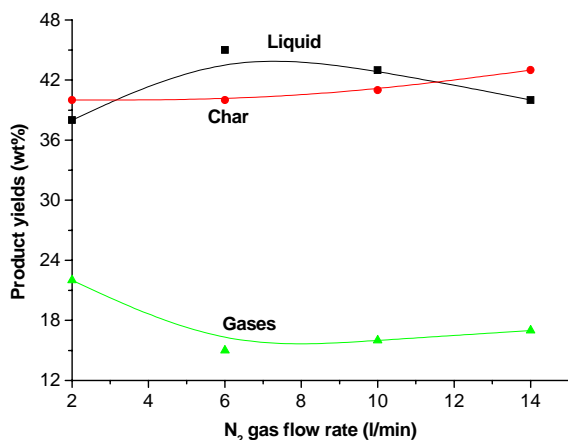


Fig. 3. Effect of N₂ gas flow rate on product yields for temperature 400°C and feed size 1.07 cm³

3.2 Analysis of product liquids

3.2.1 Fuel properties of the pyrolytic liquids

Bio-crude oils obtained from pyrolysis of tamarind seed were homogeneous and no phase separation took place in the storage bottles. The derived liquids were characterized in terms of fuel properties and compared to other pyrolytic derived fuel from date seed and heavy fuel oil in Table 2.

Table 2: Comparison of tamarind seed pyrolysis oil with biomass derived pyrolysis oil and petroleum fuel

Properties	Tamarind seed oil	Date seed oil [15]	Heavy fuel oil [16]
Kinematic viscosity at 30°C (cSt)	6.51	6.63	200 [#]
Density (kg/m ³)	1150	1042.4	980 [*]
GCV (kJ/kg)	19106.2	28636	42000

N.B.: [#] at 50°C ^{*} at 20°C

It is found that the density of pyrolytic liquids was higher than that of commercial diesel fuel, which corresponds to a higher energy content. Viscosity plays an important role in design and operation of the fuel injection system as well as in atomization quality and subsequent combustion properties of the fuel. The viscosity of liquid products from tamarind seed sample was considerably lower than that of heavy fuel oil (200 cSt at 50°C). A low viscosity of 6.51 cSt at 30°C is a favorable feature in the handling and transportation. The viscosity of bio-oils usually vary over a wide range (35-1000 cP at 40 °C) depending on the feedstock and

process conditions, and especially on the efficiency of collection of low boiling components [9]. The GCV of tamarind seed oil was near about other pyrolytic liquid but it was almost half of the heavy fuel oil.

3.2.2 Fractional distillation

The boiling point distribution of hydrocarbons in pyrolytic liquid from pyrolysis of tamarind seeds at the temperature of 240°C is presented in Fig 4. It shows that the pyrolysis liquid has a wide boiling point range. This figure also shows that about 35 % of pyrolysis liquids can be distilled at a temperature of 130°C, 40 % between 130 and 175°C and 25 % between 175 and 280°C, which correspond to the limit boiling points of light naphtha, heavy naphtha and middle distillate respectively. From Fig. 4 it can be seen that the pyrolytic liquid fractions under boiling point range of 125-225°C, which is about 70 % of total volume, are important to take into consideration to be usable as diesel fuels. It can also be realized from Fig. 4 that the pyrolysis liquid fractions have slightly lower products than the diesel fuel. For a diesel fuel smooth spontaneous ignition at relatively low temperature is essential. Thus the pyrolytic liquid fractions within the boiling point range of 125-225°C may be atomized well and hence it is possible to initiate the combustion at a lower temperature. Therefore on the basis of the boiling point distribution results, pyrolytic liquid fraction (bp: 125-225°C) may be used both as automotive diesel fuels and as heating diesel oil.

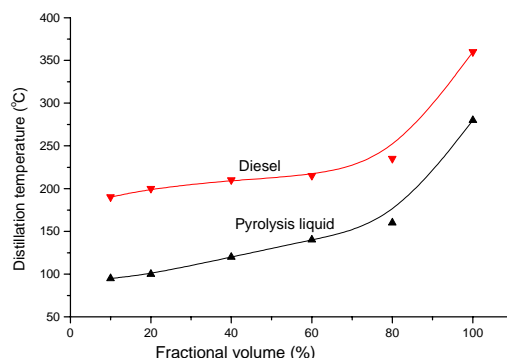


Fig. 4. Boiling point distribution of pyrolytic liquid compared to diesel.

3.2.2 Fourier Transform Infra-Red (FT-IR)

The tamarind seed pyrolysis oil and pyrolysis aqueous fraction were analyzed by Fourier Transform Infra-Red (FT-IR) spectroscopic technique. Pyrolysis oil consists of mainly alkanes, alkenes, aromatic rings and phenols. From the Fourier Transform Infra-Red (FT-IR) spectroscopy of derived pyrolytic oil, the FT-IR fractional groups and the indicated compound of the liquid product are presented in Table 3. The presence of water impurities and other polymeric O-H in the oil are indicated by the broad absorbance peak of O-H stretching vibration between 3600 and 3200 cm⁻¹. The presence of alkenes is indicated by the strong absorbance peak of C-H vibrations between 3050 and 2800 cm⁻¹ and

the C-H of deformation vibrations between 1490 and 1325 cm^{-1} . The possible presence of alkenes are indicated by the absorbance peaks 1680 and 1575 cm^{-1} representing C=C

Table 3. FT-IR functional groups and indicated compounds

Frequency range(cm^{-1})	Groups	Class of compounds
3600-3200	O-H stretching	Polymeric O-H, water impurities
3050-2800	C-H stretching	Alkanes
1680-1575	C=C stretching	Alkanes
1550-1475	-NO ₂ stretching	Nitrogenous compounds
1680-1575	C=C stretching	Alkenes
1490-1325	C-H bending	Alkenes
1300-950	C-O stretching	Primary, secondary, tertiary alcohols.
900-550	O-H bending	Aromatic compounds

4. CONCLUSIONS

The pyrolytic product yields and liquid product composition of the presented study prove that “fire-tube heating” in fixed-bed pyrolysis technology is an efficient and effective method of heating. The optimum liquid yields conditions for the reactor system are: operating temperature 400 °C, feed size 1.07 cm^3 and gas flow rate 6 L/min with the running time 30 min. The physical properties analysis showed that the oil is heavy in nature with moderate viscosity. The heating value of the oil is moderate, that can be used in power generating plant, heating and cooking purposes.

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6. NOMENCLATURE

Symbol	Meaning	Unit
T	Temperature	(K)
GCV	Gross Calorific Value	(kJ/kg)
wt%	Weight percentage	