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ORIGINAL ARTICLE

Study on fuelwood and carbonization characteristics of *Prosopis juliflora*

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Abstract In this study, fuel properties (calorific value, proximate and elemental parameters), chemical and elemental composition of *Prosopis juliflora* wood has been investigated. Result shows high calorific value (21 MJ/kg), high fixed carbon content (25 %) and low ash content (1.7 %) in *P. juliflora* wood. Ash elemental analysis shows the presence of high calcium (68 %) and low silica content (1 %) in *P. juliflora* wood. Presence of high lignin content (25 %) in *P. juliflora* wood makes it a suitable raw material for charcoal production. *P. juliflora* wood was carbonized at different experimental temperatures (300, 400, 500, 600, 700 and 800 °C). Effect of carbonization temperature on yield and other fuel properties of charcoal were investigated. The result shows that charcoal yield and its properties are greatly influenced by carbonization temperature. The charcoal yield reduced from 49 to 34 %, when carbonization temperature was increased from 300 to 800 °C. Fixed carbon content, ash content and calorific value of charcoal ranged between 55–84 %, 2–4 % and 24–33 MJ kg^{-1}, respectively. The highest calorific value (32.8 MJ/kg) was found in charcoal obtained at 600 °C. The yield of charcoal at 600 °C was found to be 37 %. Burning profile of wood and charcoal prepared at different carbonization temperature were studied under oxidizing atmosphere using thermo gravimetric analyzer. The variation in the fuel properties and combustion characteristics of charcoal prepared at different temperatures was mainly due to difference in chemical and elemental composition of chars.

Keywords *Prosopis juliflora* · Charcoal · Calorific value · Ash · Fuel · Lignin

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Introduction

Wood is the predominant source of fuel in many developing countries. It is either burned directly or converted into high energy content fuels through thermo-chemical and bio-chemical conversion processes (McKendry 2002). In India around 70 % of the energy requirement is met from fuelwood, which is collected from forest or nearby non forest areas. In recent years there is an increasing interest in the use of charcoal as a domestic fuel for cooking and heating. The other important application of charcoal includes water purification, food industry, pharmaceutical industry, chemical industry, metallurgical industry etc. (Muylaert et al. 1999). Wood charcoal is a highly porous material and its application includes water filters, gas masks, decolorizing agent, antigastric tablets etc. Charcoal is also widely used in the area of agriculture practices. It is used for improvement of soil, enhancement of plant growth and vigor, and to promote seed germination. It is also used to decontaminate soil from chemical spills, pre-emergent herbicides, pesticide, and effect of fertilizer applications.

Charcoal can be made from any lignocellulosic raw material, however, not all the species produces good quality charcoal (Kumar and Chandrashekar 2014). The charcoal making process is affected by heating rate,

residence time, particle size, chemical composition and anatomical properties, moisture content of lignocellulosic material and final carbonization temperature (Degroot and Shafizadeh 1984). Kumar et al. (1992) have investigated the effect of wood species on charcoal and found variation in its properties. Fuwape (1993) reported that yield, heating value and proximate analysis of charcoal is affects by final carbonization temperature. The chemical properties of charcoal have been studied in terms of ash content, volatile content, fixed carbon content and ultimate analyses (Liu et al. 2008; Kumar and Chandrashekar 2013). Yang et al. (2007) reported the degradation of chemical functional groups and a comprehensive understanding about the pyrolysis of cellulose, hemicelluloses, and lignin, with a focus on the gaseous product releasing properties.

In this study, we have focused on utilization of *P. juliflora* wood for production of charcoal. It is one the major fuelwood species which is used for charcoal production in many parts of the world. *P. juliflora* wood is reported to have very good heat of combustion, mainly because of high carbon content and lignin content (Goel and Behl 1996). Low moisture and ash content gives added advantage to this feedstock (Goel and Behl 1995). This species was purposely introduced in India, for its usefulness to produce fuel, timber, food for humans and livestock etc. (Walter 2011).

In this paper, fuelwood properties (calorific value, ash, volatile and fixed carbon content), ash elemental analysis and combustion characteristic of *P. juliflora* wood has been investigated. Study on chemical and elemental properties of wood has also been carried out. *P. juliflora* wood was carbonized at different temperatures (300, 400, 500, 600, 700 and 800 °C) and its effect on charcoal yield and other fuel properties were investigated. The combustion characteristic of charcoal prepared at 300, 500 and 700 °C were investigated using thermogravimetric analyser (TGA). The burning profiles of the samples were derived by applying the derivative thermogravimetric technique. The combustion properties was studied to characterise the thermal decomposition of *P. juliflora*, pre-charred at temperature, below and above the temperature of primary decomposition of biomass material i.e., <400 and >400 °C, respectively.

Materials and methods

The test samples of *P. juliflora* were procured from Bellary Forest Division of Karnataka, India. Minimum four trees were obtained for this experiment. All the trees were from similar soil and climatic conditions. After initial oven drying at 102 °C, samples were converted into desired form for further analysis. The oven dried wood was pulverized into fine powder for fuel property analysis. Samples for carbonization experiments were prepared from discs cut from trunk of trees at different positions.

Estimation of lignin and holocellulose content

The powdered *P. juliflora* sample was subjected to soxhlet extraction for 8 h using a mixture of ethanol–benzene, 1:2 (v/v) (TAPPI, T204 om-88) (TAPPI 1992). The samples obtained after solvent extraction was further subjected under hot water for removing water soluble extractives. The extractive free sample thus obtained was oven dried to constant weight at 80 °C and used for chemical analysis. The determination of lignin content was carried out by digesting extractive free bamboo sample with 72 % sulfuric acid for 2 h (TAPPI. T222 om-88) (TAPPI 1992). The holocellulose content was determined using standard method (Timell et al. 1959).

Calorific value (MJ/kg) and fuel value index

The powdered *P. juliflora* wood and charcoal samples were pelleted, oven dried to constant weight at 80 °C and burned in an oxygen bomb calorimeter (LECO AC-350) to estimate the calorific value. For each sample (wood and charcoal) the calorific value was determined three times and their average value was obtained.

Proximate analysis and elemental analysis

Ash and volatile matter were determined according to ASTM D5142, using a proximate analyzer (LECO TGA-701). The elemental parameters (carbon and hydrogen) were determined using a CHN analyzer (LECO- CHN-2000). The fixed carbon content (FCC) of the sample was estimated using following equation:

$$\mathrm{FCC}\,(\%) = [100 - (\%\,\mathrm{Ash\,dry} + \%\,\mathrm{Volatile\,dry})] \qquad (1)$$

In order to overcome the experimental and instrumental errors, experiments were repeated four times and the average values were obtained.

Ash elemental analysis

The elemental composition of wood ash was determined using Energy Dispersive X-ray Analysis (EDAX) attached to scanning electron microscope (SEM). The ash samples were finely grounded, oven dried and used as pellets for analysis. The results were obtained as elemental oxides. The average of values determined at five different areas of the samples are reported.

Table 1 Physical and Chemical properties of *P. juliflora* wood

Wood species	Proximate analysis (wt% dry basis)			Ultimate analysis (wt% dry basis)		Holo-cellulose (wt%)	Lignin (wt%)	OSE (%)	TSE (%)	CV (MJ/kg)
	AC	VMC	FCC	C	H					
P. Juliflora	1.7	66.6	25.5	48.4	6.26	65 (±1.9)	25 (±1.3)	12.3	20.5	21 (±0.3)

VMC Volatile matter content, *FCC* Fixed carbon content, *CV* Calorific value, *C* Carbon, *H* Hydrogen, *OSE* Organic solvent soluble extractives, *TSE* Total extractive content

Thermogravimetric analysis (TGA)

The combustion characteristic of *P. juliflora* wood and charcoal was studied under air atmosphere (21 % oxygen and 79 % nitrogen). Thermo-gravimetric analysis (TGA) was carried out using TGA Q500 V20.2. A known quantity of powdered sample (~10 mg) was placed in a platinum crucible and heated from ambient to 800 °C at a heating rate of 10 °C min^{-1}. The air flow rate (60 ml min^{-1}) was kept uniform during the experiment. General guidelines of ASTM D 3850 were followed. The burning profiles of the samples were derived by applying the derivative thermogravimetry technique.

Carbonization of *P. juliflora* wood

A small laboratory scale kiln with internally sealed chamber having dimensions of 8 cm length × 8 cm height × 12 cm depth was used for preparation of charcoal. The oxygen free environment was maintained inside the kiln by maintaining continuous flow of nitrogen gas. The temperature was controlled using a PID controller with an accuracy of ±1 °C. Wood samples for carbonization experiments were oven dried at 100 ± 2 °C till constant weight achieved. Oven dried and pre-weighed wood pieces (10 nos.) of size 5 cm length × 2 cm width × 2 cm thickness were used for carbonization. The oven dried wood samples were kept in the carbonization chamber at 300, 400, 500, 600, 700 and 800 °C, carbonization temperature. The ramp rate 5 °C/min and 1 h soaking time was kept constant for all the experiments. After attaining the desired carbonization temperature and 1 h soaking, the kiln was switched off and allowed to cool down. When the kiln temperature reduced below 200 °C, the samples were removed from the furnace and then sprinkled with water to arrest the carbonization process. The carbonized specimens were then oven-dried at 102 ± 2 °C. The yield of charcoal was estimated using Eq. 2.

$$Yield\,(\%) = [(W_1 - W_2)/W_1] \times 100 \quad (2)$$

where W_1 is oven-dry weight of wood sample and W_2 is oven-dry weight of charcoal. All the experiments were repeated four times to eliminate the effect of variation in furnace conditions on carbonization process.

Results and discussion

The quality of fuel depends on basic properties of raw material. The result on calorific value, proximate analysis (ash content, volatile matter content and fixed carbon content) and ultimate parameters (carbon and hydrogen) of *P. juliflora* wood is summarized in Table 1. The fuel characteristics of charcoal are affected by chemical characteristics of raw material. The basic chemical composition i.e., holocellulose, lignin and extractive content of *P. juliflora* wood is given in Table 1. The results on effect of carbonization temperature on yield and fuel properties of *P. juliflora* char are presented in Table 2. The combustion characteristic of charcoal was investigated under air atmosphere and burning profile results are summarized in Table 3.

Fuel properties of *P. juliflora* wood

The results on fuel properties, chemical and elemental analysis of *P. juliflora* wood are summarized in Table 1. The result shows high volatile matter content (67 %) and fixed carbon content (25 %) in *P. juliflora* wood (Table 1). Although, the presence of high volatile matter content meagerly contributes in heating value of a solid fuel, but for energy production through pyrolysis and gasification, high volatile content feedstock is generally preferred (Mangut et al. 2006). The calorific value is greatly influenced by elemental composition, moisture content and quantity of ash produced (Kumar et al. 2011). The calorific value of *P. juliflora* was found to be 21.0 MJ/kg (Table 1). High calorific value of *P. juliflora* wood can be attributed to its high lignin content (25 %) and high fixed carbon content (25 %) Table 1. The organic solvent soluble and water soluble extractives in *P. juliflora* wood were found to be around 12.3 and 10.5 %, respectively (Table 1). Extractives content has positive effect on calorific value of a biomass (Kataki and

Table 2 Yield and fuel properties results of *P. juliflora* charcoal

Carbonization temperature (°C)	Ultimate analysis (%)			Proximate analysis (%)			Calorific value (MJ/kg)	
	C	H	N	Ash	VMC	FCC		
300	65.8	3.7	0.41	2.5	39.1	63.4	24.0 (±0.01)	48.7 (±0.3)
400	76.1	3.9	0.52	3.0	25.8	77.2	29.9 (±0.04)	42.7 (±0.6)
500	79.6	3.3	0.63	3.2	18.6	84.6	31.2 (±0.04)	37.8 (±0.2)
600	83.3	2.8	0.61	3.6	11.2	92.4	32.8 (±0.01)	37.0 (±0.4)
700	82.9	2.1	0.70	3.7	8.9	94.7	32.1 (±0.03)	34.6 (±0.3)
800	83.4	1.7	0.52	3.4	7.9	95.5	31.7 (±0.02)	34.2 (±0.3)

Table 3 Characteristics of *P. juliflora* charcoal obtained by thermogravimetric experiment under oxidizing (air) conditions

Feed stock (°C)	Ignition temperature (°C)	Peak temperature (°C)	Maximum combustion rate (mg/min)	Temperature range (°C)
Char 300	265	449	1.399	275–475
Char 400	275	439	2.084	300–490
Char 500	310	451	1.867	300–510
Char 600	325	456	1.640	310–520
Char 700	375	483	1.659	350–530
Char 800	280	533	1.722	400–575

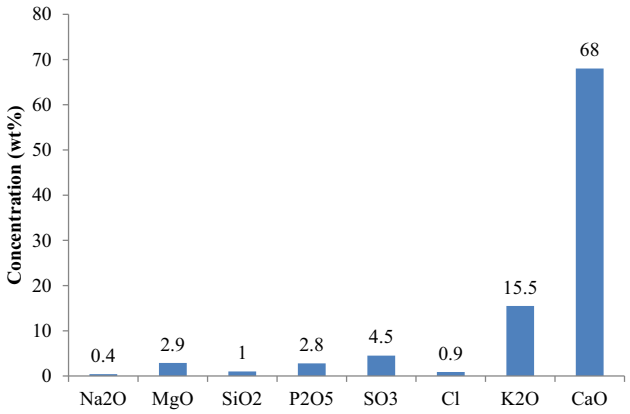

Fig. 1 Ash elemental composition of *P. juliflora* wood (wt%)

Konwrer 2001). The biomass feedstock having low ash content is considered as better feedstock. The ash in *P. juliflora* wood is found to be very low i.e., 1.7 %. The ash elemental composition of *P. juliflora* wood is reported in oxide forms Fig. 1. The major ash-forming constituents are potassium (K_2O) and calcium (CaO) Fig. 1. The presence of more calcium (68 %) and less potassium (15 %) in ash gives added advantage to *P. juliflora* wood. Alkali metal in ash, along with other elements such as silica and chlorine are responsible for many ash related problems like fouling, slagging, sintering and corrosion (Olofsson et al. 2002). On other hand, elements such as calcium and magnesium increase the ash melting point and therefore, prevent many ash related problems (Ohman and Nordin 2000). The main chemical elements in *P. juliflora* biomass are C (48.3 %) and H (6.3 %). The results of elemental analysis are presented in Table 1. In a biomass fuel, higher proportion carbon–carbon bond are more desirable, whereas, higher proportion of carbon–oxygen and carbon-hydrogen bonds reduces the energy value (McKendry 2002). The H/C ratio obtained from elemental analysis result is found around 0.13 (Table 1).

Effect of carbonization temperatures on char yield and fuel properties

The result on effect of carbonization temperature on charcoal yield in summarized in Table 2. Charcoal yield is found to be greatly influenced by change in carbonization temperature. The charcoal yield reduces from 48.7 % (±1.8) to 34.2 % (±0.8) when the carbonization temperature was raised from 300°C to 800°C. Reduction in charcoal yield at higher temperature can be attributed to the removal of the volatile matter contents from wood at high temperatures (Table 2). When carbonization temperature was increased from 300 to 800 °C, volatile matter contents were reduced from 39 (±0.1) to 8 % (±0.7). The amount of volatile matter content and fixed carbon content in char is a function of its chemical composition. The chemical composition of wood is greatly influenced by temperature. The thermal degradation of cellulose, which is major cell

wall polymer, dominates the chemistry of pyrolysis (Shafizadeh and Fu 1973). The decomposition of cellulose mainly leads to volatile gasses, whereas the thermal decomposition of lignin ends in tars and char formation (Demirbas 2001). At 325 °C the char yield from cellulose is reported be around 63.3 %, which significantly reduces to 16.7 % at 400 °C. On the other hand, char yield from isolated lignin has been reported to be very high i.e., 73 % at 400 °C (Rowell and Dietenberger 2013). The lignin, which makes up 25–30 % of the chemical composition of the wood, is thermally stable below 270 °C, while 90 % of lignin is thermally degraded at 400°C (Yang et al. 2007). The amount of holocellulose and lignin in *P. juliflora* wood is found around 65 and 25 %, respectively. Therefore, high charcoal yield from *P. juliflora* wood can be attributed to its high lignin content. The charcoal yield from *P. juliflora* at 400, 600, and 800 °C is around 43, 37 and 34 %, respectively which is significantly higher than other major fuelwood species (Table 2). Kumar et al. (1992), have carried out carbonization of *Acacia nilotica* and *Eucalyptus globulus* wood samples at almost similar experimental condition i.e., 4 °C/min heating rates and 1 h soaking time. They reported a charcoal yield of 32.0, 27.5 and 26.7 % at 400, 600, and 800 °C, respectively, in case of *E. globulus*. Similarly, the yield of charcoal from *A. nilotica* at 400, 600, and 800 °C was found to be 31.70, 26.10 and 22.76 %, respectively. Kumar et al. (2008) have studied carbonization characteristics of *Casurina equisetifolia* wood and found charcoal yield of 35, 31 and 29 % at 400, 600 and 800 °C, respectively.

From the result presented in the Table 2, it is observed that the proximate and ultimate values of char are greatly dependent on the carbonization temperature. When the carbonization temperature was increased from 300 to 800 °C, the FCC increased from 63 to 95 %, AC increased from 2.5 to 3.4 %, whereas, VMC of wood char reduced from 39 to 8 %. The higher value of FCC and AC in charcoal prepared at higher temperature may be due to the removal of VMC from the wood during the pyrolysis process. Calorific value of *P. juliflora* char is shown in Fig. 2. Calorific value of char is found to be greatly influenced by change in carbonization temperature. A marginal increase in calorific value was observed with increase in carbonization temperature up to 600 °C (Table 2). The increase in calorific value can be attributed gradual increase in FCC and reduction in VMC of charcoal with carbonization temperatures (Table 2). The heat of combustion of carbon is higher than that of volatile matter and therefore, the samples having high proportion of fixed carbon content have higher calorific value. The charcoal obtained at 700 and 800 °C were found to have marginally lower calorific value (32.1 and 31.7 MJ/kg) as compared to the one prepared at 600 °C (32.8 MJ/kg) Table 2. The

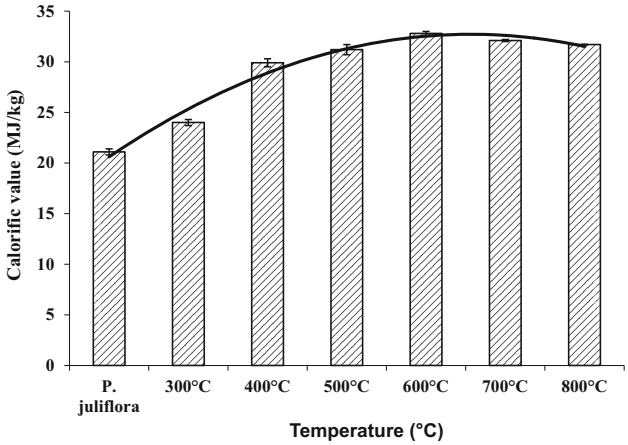

Fig. 2 Calorific value of *P. juliflora* wood and charcoal

same can be attributed to the presence of high ash content in char prepared at higher carbonization temperatures (700 and 800°C), which has a negative effect on the calorific values of samples (Fuwape 1993; Kumar and Chandrashekar 2013, 2014). The calorific value of *Eucalyptus* hybrid and *C. equisetifolia* wood char at 600 °C is 33.8 and 32.7 MJ/kg, respectively (Kumar et al. 2008). The calorific value of *P. juliflora* wood (21 MJ/kg) was compared with char of the same species in Fig. 2. The ash, volatile and fixed carbon content of *P. juliflora* wood were found to be 0.83, 80.39 and 17.38 %, respectively (Table 1).

The values of ultimate carbon and hydrogen were also found to be changing with carbonization temperature. As shown in the Table 2, the ultimate carbon of the char increased with the increase in the carbonization temperature, whereas, the ultimate hydrogen in chars decreases with the increase in the carbonization temperature. When the carbonization temperature was increased from 300 to 800 °C, the ultimate carbon increased from 66 to 83 %, whereas, the amount of hydrogen decreased from 3.7 to 1.7 % (Table 2).This increase in the carbon % with increase in the temperature may be due to the release of volatile matter (H_2O, CO, CO_2, non- cyclic hydrocarbon, etc.) from the wood and enrichment of remaining wood char matrix with aromatic compounds (Biagini et al. 2008). The loss in the hydrogen percentage also supports the aromatisation of the wood char matrix (Leckner and Karlsson 1993).

Combustion characteristics of *P. juliflora* wood biomass and charcoal

The most common technique used for studying thermal degradation of carbonaceous materials is by thermogravimetric analysis (TGA) (Haykiri-Acma 2003; Munir et al. 2009). Derivative thermogravimetric (DTG) curves of the

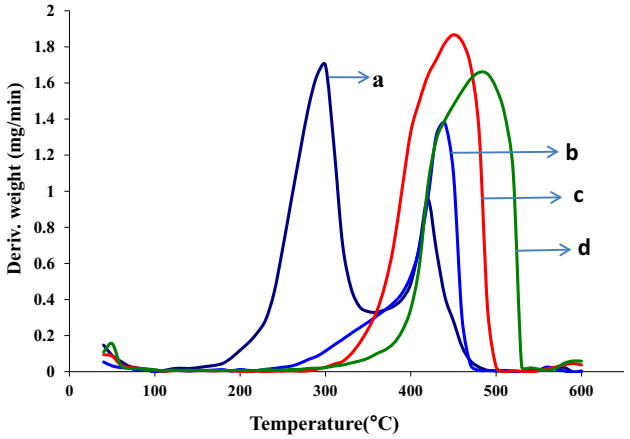

Fig. 3 TGA thermogram of thermal decomposition of *a P. juliflora* wood biomass; *b* char-300 °C; *c* char-500 °C; **d** char-700 °C

P. juliflora wood and char samples (300, 500 and 700 °C) are shown given in Fig. 3. The characteristic parameters of TGA analysis under air atmosphere are summarized in Table 3. The DTG curve shows an initial weight loss between temperatures 40–90 °C which is mainly due to the removal of moisture and lighter volatiles from wood and charcoal samples (Munir et al. 2009). In wood samples, a sudden weight loss was observed at around 200 °C which is mainly due to removal of volatiles and their combustion Fig. 3. This zone (200–300 °C) is termed as active pyrolysis zone. The third major weight loss occur at temperature range of 400–475 °C, which is mainly due to combustion of char Fig. 3. This zone is termed as passive pyrolysis zone. The ignition temperature corresponds to the point at which the burning profile underwent a rapid rise (Garcia-Ibanez et al. 2006). However, the temperature where the rate of weight loss due to combustion is highest is called as peak temperature (Haykiri-Acma 2003). The ignition temperature for active and passive zone in wood sample was found to be 217 and 419 °C, respectively.

Thermal decomposition of charcoal results in one major peak, at high temperature range of 250–575 °C Fig. 3. The ignition temperature of charcoal obtained at 300, 500 and 700 °C was found to be 265, 310 and 375 °C, respectively (Table 3). The variation in ignition temperature of charcoal obtained can be attributed to difference in their volatile matter content. The charcoal obtained at lower temperatures contains higher volatile matter content as compared to the one carbonized at higher temperatures (Table 2). During combustion process, around 300–375 °C, most of the carbohydrate polymers decompose into combustible volatile, leaving lignin as largely un-degraded wood component (Rowell and Dietenberger 2013). Degradation of cellulose produces combustible volatiles such acetaldehyde, propenal, methanol, butanedione, and acetic acid (Rowell and Dietenberger 2013). The temperature corresponding to peak height is inversely proportional to reactivity while the peak height is directly proportional to the reactivity. The maximum combustion rate of 1.399, 1.867 and 1.659 mg/min were found at peak temperatures of 449, 451 and 483 °C, for *P. juliflora* charcoal prepared at 300, 500 and 800 °C, respectively (Table 3). This shift in combustion rate and peak temperature of the samples can be attributed to different amount of carbon available in the samples. The charcoals prepared at higher temperatures have higher percentage of carbon and low amount of volatile matter as compared to the one obtained at lower temperatures (Table 2).

Conclusions

Following conclusion can be drawn from the results discussed above.

- High yield of charcoal from *P. juliflora* can be attributed to presence of high lignin content and high fixed carbon content in its wood.
- Decrease in yield of charcoal with increase in carbonization temperature can be attributed to reduction in volatile matter content at higher temperature.
- Volatile content of charcoal decreases with the carbonization temperature but the fixed carbon and ash content increases with rise in carbonization temperature.
- A definite trend of increase in the calorific value of char with carbonization temperature up to 600 °C has been noticed. However, further increase in temperature resulted in reduction in calorific value due to higher proportion of ash in char.
- Ignition temperature of the charcoal sample was found to be increasing with increases in reaction temperature. The reactivity (rate of combustion) of charcoal prepared at lower temperature (300 °C) was found to be more compared to charcoal obtained at higher temperatures.

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