

## Effect of wood material and pyrolytic conditions on biocarbon production

Sherif S. Z. Hindi

(Department of Arid Land Agriculture, Faculty of Meteorology, Environment and Arid Land Agriculture, King Abdul-Aziz University, Saudi Arabia)

### Abstract

Outer and inner-zone wood of characterized eucalypt (*Eucalyptus camaldulensis*), casuarina (*casuarinas glauca*), tamarisk (*Tamarix aphylla*) and acacia (*Acacia saligna*) were pyrolyzed in a flowing nitrogen atmosphere at conditions of maximum final temperature (MFT) of 450° and 600°C and heating rates (HR) of 10° and 25°C/min. for a residence time (RT) of 90 and 120 min. Yield and the proximate analysis of biocarbon differed significantly according to species and pyrolysis conditions. Tamarisk and eucalypt gave the highest yields. Further, the yield decreased significantly with the increasing of the MFT and HR decreased. Apparent density (AD) of biocarbon was affected inversely only by the pyrolysis conditions, especially HR. The volumetric shrinkage (VS) of biocarbon is dependent on species, wood zone and pyrolysis conditions. The VS, ash (AC) and fixed carbon (AC) of biocarbon were affected directly only by the MFT. The gross heat of combustion (GHC) of biocarbon was affected by wood zone and pyrolysis conditions. For all species, the GHC As well as volatiles of biocarbon obtained at 450°C were lower than those at 600°C.

**Keywords:** Biocarbon, Apparent density, Gross heat of combustion, Proximate analysis, Shrinkage, Wood, Yield.

### 1. Introduction

Each year, more than 40 million tonnes of inedible plant material, including wheat stems, corn stover (the stalks and leaves) and wood shavings from logging, are produced-much of which is thrown away [1]. The increasing environment protection requirements for waste timber materials attract interest regarding the use of traditional slow pyrolysis equipment, which is employed to produce charcoal, for their utilization, since the raw material is lump wood [2]. It was concluded that their species studied were the most suitable species for short rotation fuel wood forestry programmes because of their high wood density, biomass yield, low ash and moisture content, and good heat of combustion at the juvenile stage [3]. In both Egypt and Saudi Arabia, each of wood, biocarbon made by earthen pits and their briquettes were used as sources of energy by direct burning especially in desert and villages for domestic purposes. Biocarbon is being used in Egypt in combination with sulfur and cellulosic pulp in synthetic silk production. Further, it is used frequently to manufacture special sizes of electrodes for electric analysis of copper in some factories.

The characteristics of the wood charcoals depend not only on the wood used, but also the carbonization system. In particular, the eucalyptus charcoal produced in continuous furnace has interesting characteristics: the lowest ash content ( $\sim 2\%$ ), the highest fixed carbon content ( $\sim 90\%$ ) and the highest total pore volume ( $\sim 1 \text{ cm}^3 \text{ g}^{-1}$ ) [4].

Increasing reaction temperatures decreased charcoal yield, increased its carbon-to-oxygen ratio, and promoted cellulose carbonization [5]. The char yield in pyrolysis decreased rapidly with an increase in temperature until 400 °C, after which there was a gradual decrease in the yield to ca. 40% at 750 [6]. The char yield decreases and the char structure becomes more deranged with increasing temperature [7].

Empirical models of shrinkage as a function of conversion are presented. The volume shrinkage of birch was found to be 45–70% and the shrinkage in the different directions 5–25, 25–40 and 15–40% for longitudinal, tangential and radial directions, respectively. Longitudinal shrinkage commenced after about 60% mass loss and is not strongly dependent on heating rate or on cellulose chain scission. A maximum shrinkage was found for tangential and radial directions at 400 and 500–700°C, respectively, and above these temperatures the shrinkage decreases [7]. It was found that under high heating rates the char particles underwent plastic deformation (i.e. melted) developing a structure different to that of the virgin biomass [8].

It was concluded that both the temperature and heating rate had a significant effect on both yield of pyro-oil and bio-char resulting from pyrolysis of beech trunkbark. The yield of bio-char decreases from 59 to 42% for 2 K/s run and it decreases from 43 to 29% for 100 K/s run as the final temperature was raised from 550 to 800 K [9]. When the pyrolysis temperature is increased, the bio-char yield decreases. The bio-char yield increased with increasing particle size of the sample. A high temperature and smaller particles increase the heating rate resulting in a decreased bio-char yield. The higher lignin content in olive husk results in a higher bio-char yield comparison with corncob. Bio-char from olive husk was more reactive in gasification than bio-char from corncob because of the higher ash content [10].

The higher heating values of oven dry poplar were from 15,787 to 24,275 kJ/kg. The average calorific values of willow wood (whole tree with bark) were from 16,169 to 22,572 kJ/kg [11]. It was showed that there was no significant difference in calorific values between the three eucalypt species studied when considering a similar age category. Similarly, there was no significant difference in calorific values between disc positions and wood types (heartwood versus sapwood) within species. However, calorific values tend to increase along the tree stem towards the tree top for each of the three species [12].

It was showed that high temperature carbonized coconut shell char and activated carbon samples derived from high temperature carbonized coconut shell chars had higher BET surface area, total volume, micropore volume and yield as compared to those of low temperature carbonized coconut shell char and activated carbon derived from low temperature carbonized coconut shell char [13]. The pyrolysis temperature has an important impact on the yield and composition of bio-oil, char, and gases [14].

It was found high negative correlations of the wood heat value with holocellulose and ash, and high positive correlations with wood density, lignin, and alcohol-benzene and hot-water solubles. The exotic species studied gave equal or better charcoal, in terms of yield and quality, compared with the traditionally preferred talh [15].

Very good charcoal yields (35% for karkadeh and 38% for usher) with high energy transformation (58% and 62%, respectively) was obtained. The karkadeh charcoal, except for a somewhat high ash content, was good for domestic uses (79% fixed carbon and 30.3 MJ kg<sup>-1</sup> heat value). The usher charcoal was better with respect to fixed carbon (86.5%) and gross heat value (32.4 MJ kg<sup>-1</sup>). Both charcoals were of low density (140–160 kg m<sup>-3</sup>) [16]. The effects of final carbonisation temperature on the yield, heating value and proximate analysis of charcoal from the tree species were determined. *Gliricidia sepium* gave the highest charcoal yield of 51.6% with a gross heating value of 31.45 MJ/kg at 300°C final carbonisation temperature. There was no significant difference in the charcoal yield of *Gmelina arborea* and *Leucaena leucocephala* at 5% level of testing. The charcoal yield decreased with an increase in carbonisation temperature. The percentage volatiles in charcoal decreased with an increase in temperature, while there was an increase in the percentage fixed carbon [17].

Virtually, nothing has been carried out in Saudi Arabia and Egypt relevant to pyrolyze woods from eucalypt, casuarina, tamarisk and acacia at the selected pyrolysis conditions. Therefore, this study was conducted to evaluate the physico-chemical characterization of biocarbon and the parent wood from each of the four species.

## 2. Materials and Methods

Four hardwood species were used in the present investigation for biocarbon production, namely eucalypt (*Eucalyptus camaldulensis* Dehn.), Casuarina (*Casuarina glauca* Sieb.), tamarisk (*Tamarix aphylla* (L.) Karst.) and acacia (*Acacia saligna* (Labill.) H. Wendl.). These species are promising fast-growing timber trees for new reclaimed lands, deserts and sand dune fixation. Due to the different physical, chemical and anatomical characteristics of wood between and within species, study of outer-and inner-zone wood was additionally considered in the study. Four trees were selected from each species from different locations near Alexandria, Egypt. The age of the selected trees was about 30 years except for acacia whereby its age was 5 years. The diameters outside bark of the trees varied from 15-40 cm.

### 2.1. Samples preparation

After the selected trees were felled, one bolt, 0.5 m long each was cut from each stem at a height of 0.5 m above ground level. One diametric strip of 1.5 cm tangentially and 0.5 m longitudinally was removed from each bolt. Selected sticks from each of outer- and inner-zone woods were isolated and subsequently cross cut into cubic samples free of visible defects. Each twenty subsequent samples were specified for each pyrolysis run to determine the different properties of biocarbon as well as the volume of the wood samples assigned for volumetric shrinkage of biocarbon.

### 2.2. Pyrolysis process

The pyrolysis runs were carried out with a bench scale reactor (Fig. 1) which consists of an electric tube furnace (carbolite furnace modle MTF 12/338) controlled by a microprocessor temperature programmer with an error of  $\pm 5^{\circ}\text{C}$  with an accuracy of  $1^{\circ}\text{C}$  and heating rate of  $0.01^{\circ}\text{C}/\text{min.}$ , digital thermometer with a chromel alumel thermocouple (type K with an error of  $\pm 1^{\circ}\text{C}$ ), nitrogen regulator system consisted of a regulator and a flowmeter with an accuracy of  $\pm 2\%$  of the full scale and the reactor body made up of Pyrex glass that consists of the samples tube (with outer diameter of 2.8 cm) and the train of traps. This train is consisted of two connected traps: The first one is immersed in covered Dewar flask filled with liquid nitrogen and the second one is simply a water scrubber, held at room temperature.

Before starting a pyrolysis run, nitrogen flow was introduced at 354 ml/minute until a steady gas flow was obtained, increased to 825 ml/minutes for 10 minutes and re-adjusted at 354 ml/minute for the duration of the pyrolysis run. The cover of the Dewar flask was raised only while the liquid nitrogen was being poured. Then, after ten minutes of pouring, the liquid nitrogen trap was connected to the water scrubber trap. However, this period is long enough for the nitrogen gas inside the reaction body to approach, relatively, its original state after addition of N<sub>2</sub>-liquid to prevent a back suction that force water to move inversely from the water scrubber toward the pipe attached to N<sub>2</sub>-liquid trap where the water freezes and plug it. This can prevent the nitrogen gas to pass through it increasing the pressure inside it and explores the glassy reactor. At the beginning of the run, the furnace was switched on and the temperature program was set on the desired maximum final temperature (MFT) of 450°C with a heating rate (HR) of  $2.5^{\circ}\text{C}/\text{min.}$ , starting from a room temperature. After the MFT was reached, the heating system was shut off and the samples were allowed to cool to 100°C with maintenance of nitrogen flow at 354 ml/minute.

### 2.3. Determination of wood and biocarbon properties

Wood properties, namely extractives (EC), lignin (LC) and ash (AC) contents were determined according to the ASTM standard methods [19], [20] and [21], respectively. The volumes of wood samples assigned for specific gravity (SG) and volumetric shrinkage (VS) of biocarbon were determined using mercury displacement using Amsler volume meter. The SG of biocarbon was determined based on the oven-dry weight and volume, whereas the VS was based on the oven dry volume of the parent wood. Gross heat of combustion of biocarbon was determined using an adiabatic oxygen bomb calorimeter, Parr 1341, according to the procedures recommended by Parr instruction manual and in accordance with ASTM [22].

The biocarbon yield was calculated based on the oven-dry weight of wood. The moisture, volatile matter, ash, and fixed carbon contents were determined according to the ASTM [23] for proximate analysis of wood charcoal.

Split-split plot design in three replicates was applied and the statistical analysis of the recorded data was done according to Steel and Torrie [24] using the analysis of variance procedure and least significant difference test (LSD) at  $P \leq 0.05$ .

## 3. Results and Discussion

Mean values of extractives content (EC), lignin content (LC) and ash content (AC) of wood were presented at Table 1. It can be seen that the three properties was affected significantly by species. The eucalypt wood had the highest EC and LC while acacia was the lowest. Further, tamarisk wood contained the highest ash among the four species while eucalypt wood had the lowest one.

Biocarbon properties, namely yield, volumetric shrinkage, ash, volatiles and fixed carbon contents of biocarbon made under the controlled pyrolysis conditions of maximum final temperature (MFT) of 450° and 600°C, heating rate (HR) of 10° and 25 °C/min. and residence time (RT) of 90 and 120 min.

Yield of biocarbon differed significantly according to species and pyrolysis conditions. The yields from tamarisk and eucalypt (33.45% and 32.86%, respectively) were higher than those from casuarina and acacia (27.62% and 27.58%, respectively) as shown in Fig. 2. Further, the yield decreased significantly with the increasing of the MFT and HR decreased without any effect due to RT (Fig. 3). There is no clear effect due to wood zone on biocarbon yield. The differences between species in their productivity of biocarbon may be attributed to their differences in extractives, lignin and ash contents of the parent wood. Eucalypt and tamarisk woods with the greatest extractives content (Table 1.) produced more biocarbon comparing with casuarinas and acacia with the lowest extractives content (Fig. 2). This is supported by the finding of Kryla [25] who stated that removing extractives from wood caused greater mass loss and therefore lower yield at carbonization temperatures of 350°C and higher. He attributed this behavior to the fact that extractive remaining in the unextracted samples are degrading at a slower rate than the corresponding wood substance. Furthermore, lignin content of wood is the most important factor influencing the productivity of biocarbon. It has been stated that species with higher contents of lignin give higher yields than those with lower contents of lignin [25] and [26]. This is can be attributed to that lignin generally leaves a larger residue, due to its thermal stability, than do the polysaccharide at the end of carbonization process [10]. Accordingly, eucalypt and tamarisk with the greatest lignin content of wood produce more biocarbon than casuarinas and acacia with the lowest lignin content (Table 1. and Fig. 2.). In addition, ash content of wood may affect the biocarbon yield. This is obvious in the case of tamarisk which contained the highest ash content of wood (5.4 %) and reasonably the highest biocarbon yield (Fig. 1.). However, other researchers did not find a species effect on biocarbon yield [10].

The slight differences in the apparent density (AD) of biocarbon made from the four hardwood species are presented at Fig. 4 with the highest value for casuarinas and the lowest one for acacia. Actually, the AD of biocarbon was affected only by the pyrolysis conditions, especially HR. The AD decreased as HR increased from 10° to 25°C/min. (Fig. 5), whereas neither the MFT nor RT had a significant effect on it. This is adapted to that concluded for the much greater effect of heating rate on the pyrolysis of biomass and the quick devolatilization of the biomass rapid pyrolysis that favors the formation of char with high porosity [10] and accordingly lower apparent density. In addition, the AD of biocarbon was directly related to the specific gravity of the parent wood.

The volumetric shrinkage (VS) of biocarbon is dependent on species, wood zone and pyrolysis conditions. The VS of biocarbon made from inner-zone wood of eucalypt was much lower than that from outer-zone (Table. 2). This effect was not evident in the case of the other species. For the pyrolysis conditions, the VS of biocarbon was affected only by the MFT, whereby it increased as the MFT increased (Fig. 3). Examining Fig. 3 revealed that most of the VS took place up to 450°C, thereafter, the increase in shrinkage was small due to most of thermal degradation of the polysaccharides takes place below 450°C [25], [26] and [10].

Gross heat of combustion (GHC) of biocarbon was affected by wood zone and pyrolysis conditions. The GHC of tamarix biocarbon made from outer-zone wood was greater than that from innerzone wood (Table 2). However, this behavior is not evident for the other species. Generally, at all pyrolysis conditions, the GHC of biocarbon for tamarisk biocarbon was lower than those for the other species (Fig. 6). For all species, the GHC of biocarbon obtained at 450°C were lower than that at 600°C. There were no evident effects on the GHC of biocarbon due to HR and RT (Fig. 7). The differences in the GHC of biocarbon between species at different pyrolysis conditions may be related to their fixed carbon (FC) and ash (AC) contents [27], [28] and [29]. Accordingly, the highest GHC values of eucalypt, casuarinas and acacia biocarbons can be attributed to their higher FC and lower AC values (Fig. 3). On the other hand, tamarisk biocarbon with the lowest FC and highest AC contents gave the lowest GHC value.

The ash content (AC) of biocarbon varied according to species and pyrolysis conditions. Tamarisk biocarbon had generally much higher content of ash than the other species (Fig. 2). However, this content was increased only with

increasing of MFT (Fig. 3). The volatiles of biocarbon made from different species was affected by pyrolysis conditions. At the same conditions of MFT of 450°C, HR of 10° or 25°C/min. and RT of 90 or 120 min., the volatiles of biocarbon was generally higher than that at 600°C, HR of 10 or 25°C/min. and RT of 90 or 120 min. (Fig. 3). Up to 450°C, the volatiles of acacia and casuarina biocarbon were lower than those for eucalypt and tamarisk. However, up to 600°C, tamarisk biocarbon contained higher volatiles than the other species. In addition, Fixed carbon (FC) of biocarbon was affected by species and pyrolysis conditions as single variables. At pyrolysis conditions of MFT of 450°C, HR of 10° or 25°C/min. and RT of 90 or 120 minutes, the FC of biocarbon was generally lower than that at 600°C, HR of 10° or 25°C/min. and RT of 90 or 120 min. Up to 450°C, the FC of acacia and casuarina biocarbon were lower than those for eucalypt and tamarisk. However, up to 450° or 600°C, the FC content of tamarisk biocarbon was lower than the other species (Fig. 3).

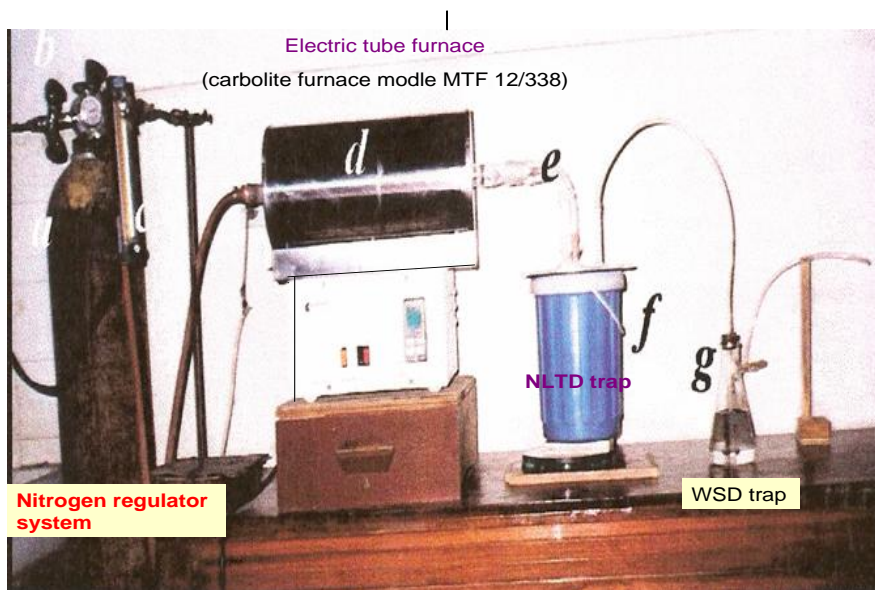


Fig. 1. Pyrolytic apparatus used for the wood pyrolysis.

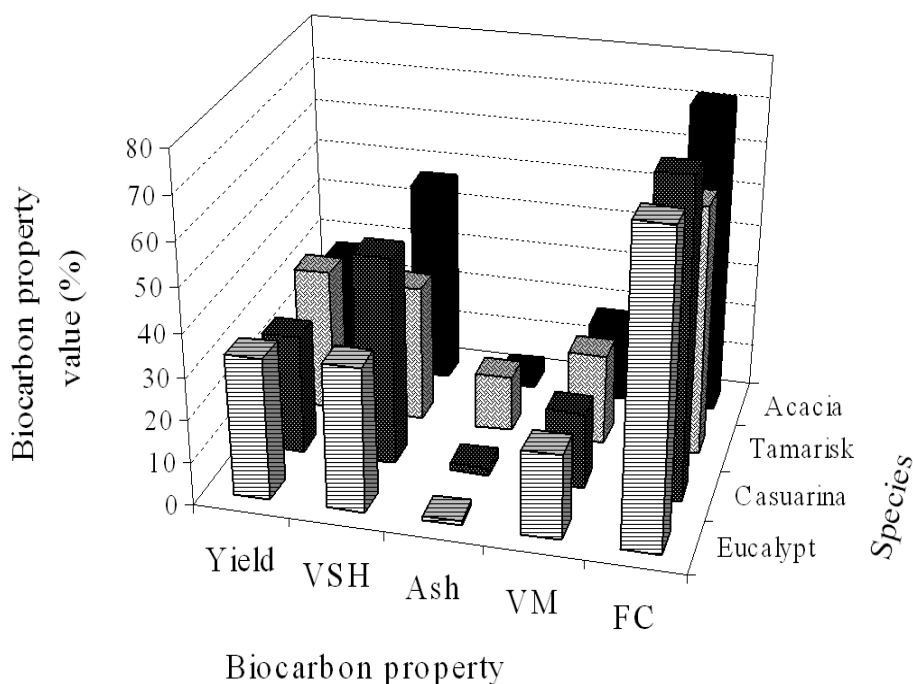


Fig. 2. Yield, shrinkage, ash, volatiles and fixed carbon contents of biocarbon made from the four wood species.



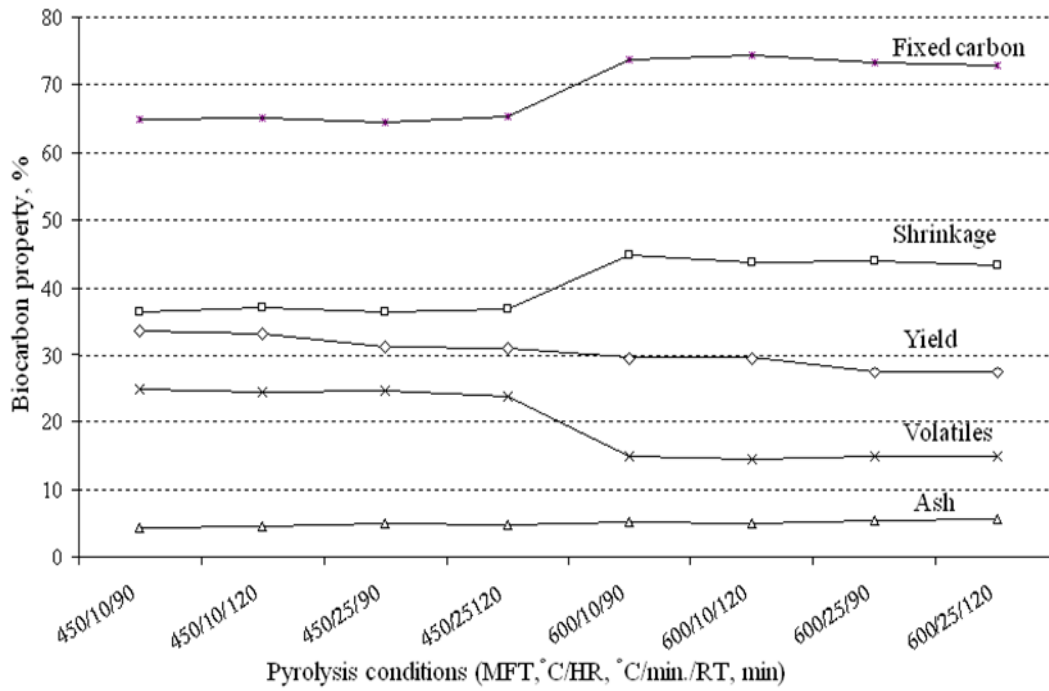


Fig 3. Yield, shrinkage, ash, volatiles and fixed carbon contents of biocarbon made under controlled pyrolytic conditions of maximum final temperature (MFT) of 450° and 600°C, heating rate (HR) of 10° and 25 °C/min. and residence time (RT) of 90 and 120 min.

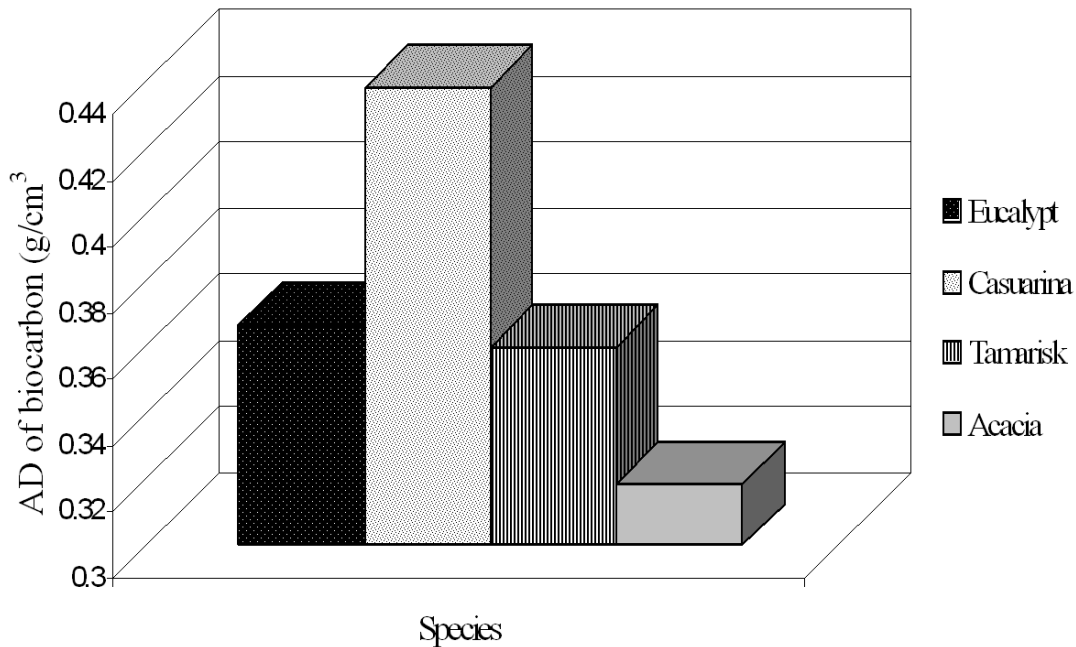


Fig. 4. Apparent density (AD) of biocarbon made from the four wood species.

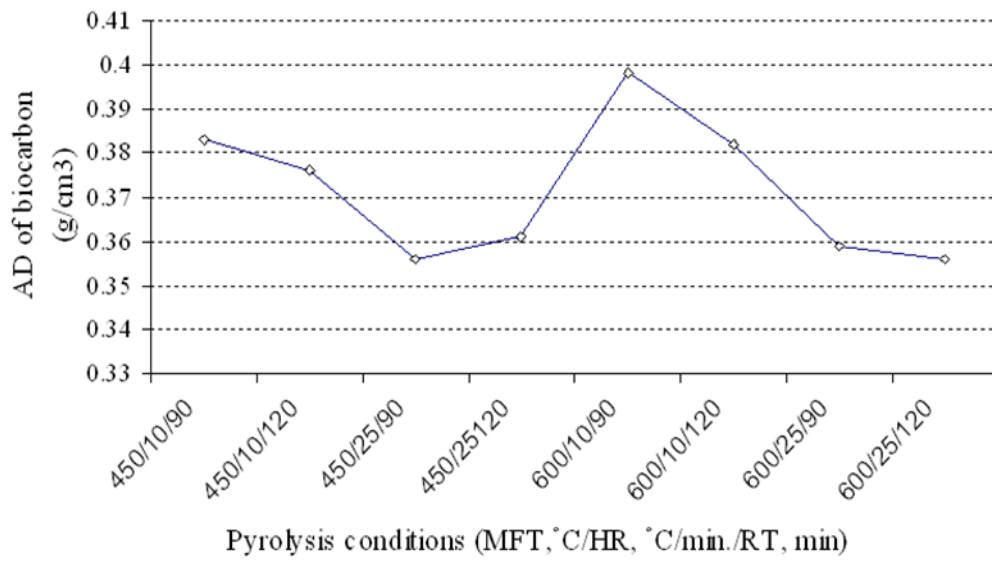


Fig. 5 Apparent density (AD) of biocarbon made under controlled pyrolytic conditions of maximum final temperature (MFT) of 450° and 600°C, heating rate (HR) of 10° and 25 °C/min. and residence time (RT) of 90 and 120 min.

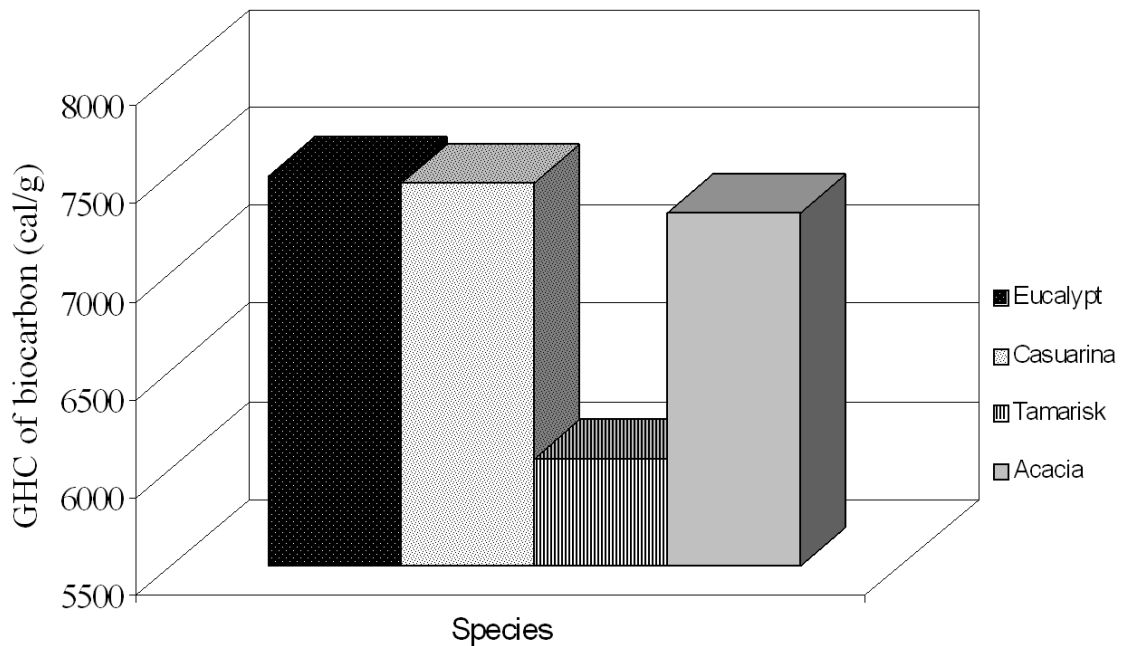


Fig.6. Gross heat of combustion (GHC) of biocarbon made from the four wood species.

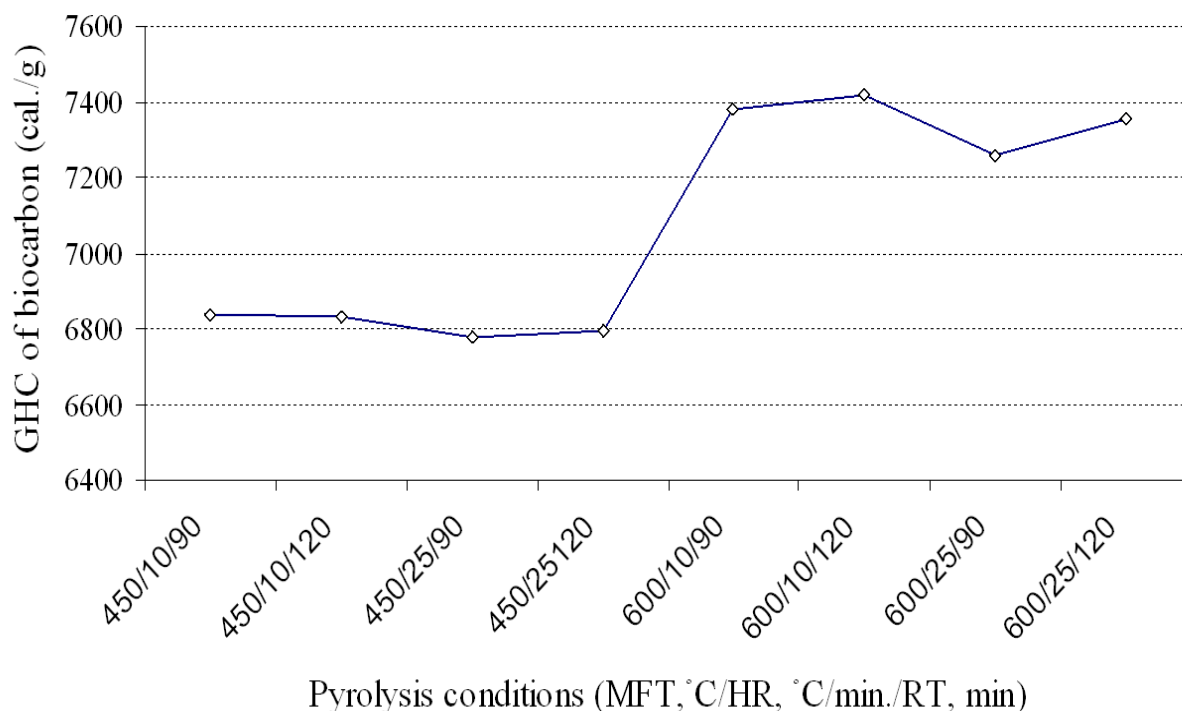


Fig. 7. Gross heat of combustion (GHC) of biocarbon made under controlled pyrolytic conditions of maximum final temperature (MFT) of 450° and 600°C, heating rate (HR) of 10° and 25 °C/min. and residence time (RT) of 90 and 120 min.

Table 1. Mean values\* of extractives content (EC), lignin content (LC) and ash content (AC) of wood.

Species	EC (%)	LC (%)	AC (%)
<i>Eucalyptus camaldulensis</i>	19.62 <sup>a</sup>	31.65 <sup>a</sup>	0.169 <sup>c</sup>
<i>Casuarina glauca</i>	5.38 <sup>c</sup>	28.37 <sup>b</sup>	0.922 <sup>bc</sup>
<i>Tamarix aphylla</i>	15.77 <sup>b</sup>	27.92 <sup>b</sup>	5.429 <sup>a</sup>
<i>Acacia saligna</i>	2.78 <sup>d</sup>	19.88 <sup>c</sup>	1.308 <sup>b</sup>

\*Means with the same letter are not significant.

Table 2. Mean values\* for volumetric shrinkage (VS) and gross heat of Combustion (GHC) of biocarbon made of the four wood species from each of the outer-and inner- zone woods.

Species	VS (%)	GHC (cal/g)
<i>Eucalyptus camaldulensis</i>		
a-outer-zone wood	40.74	7517
b-inner-zone wood	26.52	7488
<i>Casuarina glauca</i>		
a-outer-zone wood	51.56	4749
b-inner-zone wood	45.90	7472
<i>Tamarix aphylla</i>		
a-outer-zone wood	29.94	6250
b-inner-zone wood	33.97	5852

<i>Acacia saligna</i>		
a-outer-zone wood	46.75	7372
b-inner-zone wood	47.43	7252
LSD <sup>1</sup> <sub>0.05</sub>	7.56	89
LSD <sup>2</sup> <sub>0.05</sub>	11.26	97

\*Each value is an average of 32 specimens.

<sup>1</sup>LSD<sub>0.05</sub> = Least significant difference at 95% level of confidence, to test the difference between two wood-zone means at the same level of species.

<sup>2</sup>LSD<sub>0.05</sub> = to test the difference between two species means at the same or different levels of wood zone.

#### 4. Conclusion

Biocarbon produced from tamarisk, irrespective of the yield, was generally lower in its quality than other species at the same pyrolysis conditions. This is due to its higher contents of ash and volatiles as well as its lower GHC and FC. *Acacia* biocarbon is generally of better quality compared to that of tamarisk but is lower in some quality parameters due to its lower AD and relatively higher ash content than the biocarbons made from casuarinas and eucalypt. Accordingly, *Acacia saligna* biocarbon is of better industrial quality subsequent by *Casuarina glauca*. They are suitable as reductants in metallurgy as well as for production of CS<sub>2</sub> required for silk synthesis, internal and external fuels and sorbents.

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