

## Vacuum pyrolysed biochar for soil amendment

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### Abstract

Biochar, a highly carbonaceous charred organic material obtained from biomass conversion can be deliberately applied as a conditioner/amender in order to improve soil quality and associated environmental services. Napier grass (*Pennisetum purpureum*), a lignocellulosic biomass, can potentially be used to produce biochar. The aim of the present work is to manufacture, comprehensively characterize, and apply biochar obtained from the vacuum pyrolysis and investigate its potential for soil amendment. Biochar produced from Napier grass was characterized for its pH, electrical conductivity, soil water retention capacity, surface acidity and/or basicity, elemental composition, Infrared spectra, X-ray diffraction spectra, surface area, porosity, soil–water relation and morphological properties. Experiments on the methylene blue adsorption of the biochar indicated an equilibrium uptake capacity of 35 mg.g<sup>-1</sup> and showed good agreement with the Langmuir–Freundlich model. Kinetic studies revealed Lagergren pseudo-first-order fit with intra-particle diffusion appearing to be one of the rate controlling mechanisms. Pot trials with *Cicer* grown in neutral and acidic soil amended with biochar validated that biochar augmented plant growth in terms of enhanced biomass weight and number of seed germinations. The entire investigation revealed that the properties of the produced biochar are in line with those necessary for it to act as a suitable agent for soil amendment.

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**Keywords:** Napier grass; Pyrolysis; Biochar; Soil amendment; Adsorption; Crop trials

### 1. Introduction

Thermo-chemical, bio-chemical and physico-chemical conversion of lignocellulosic biomass represents a coherent renewable source for valuable industrial products [1]. Amongst these processing techniques, thermo-chemical conversion is the fastest and includes vacuum pyrolysis, gasification, and combustion. Vacuum pyrolysis has been extensively used for the conversion of unused biomass and its advantage lies in the process's ability to yield gaseous, liquid and solid products, which is not the case for combustion or gasification. Biochar, a highly carbonaceous charred organic material obtained from biomass pyrolysis, has been deliberately applied as a soil conditioner with the intent of improving soil quality and associated environmental services [2]. Its ability to be a good soil

amendment agent lies in its physico-chemical properties, porous morphology, and surface functionalities; these properties, however, are strongly dependent on the operating conditions of the pyrolysis and the nature of the feedstock [3].

Biochar obtained from the pyrolysis of lignocellulosic materials has an expected half-life in the range of 100 to 1000 years, which is approximately 10–1000 times longer than the life times of most soil organic matter. Thus, biochar addition to soil could provide a potential sink for organic carbon [3]. It can also augment pH, plant and microbial growth, nutrient retention, water holding capacity and carbon sequestration in soils while preventing groundwater eutrophication; this, in turn, can reduce fertilizer requirements and environmental deterioration associated with fertilizer use [4–8]. However, prior to soil application, biochar has to be comprehensively characterized since it yields differential effects on different types of soil and/or crops. Furthermore, while the pyrolysis of waste biomass like sugar cane bagasse, pecan shells, bio-solids, household wastes, etc. have been investigated earlier for the production of activated carbon and biochar, their application as a soil amender has received relatively less attention [9–11].

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Napier grass (*Pennisetum purpureum*), a lignocellulosic biomass, can potentially be used to produce biochar through pyrolysis. The grass undergoes harvesting after 3–4 months of planting and can last up to five years. It is drought-resistant but sometimes may become an aggressive plant that spreads underground and if uncontrolled, can invade crop fields as a weed. Our literature review revealed that, although biomass based biochars have been investigated earlier, Napier grass pyrolysed biochar has not been investigated for soil amendment. Thus, the aim of the present work is to comprehensively characterize as well as utilize biochar produced from Napier grass pyrolysis. Properties investigated include char pH, electrical conductivity, surface acidity and/or basicity, ash and elemental content, surface functionality (infrared spectroscopy), X-ray diffraction spectra, internal surface area, and surface morphology through scanning electron microscopy. Lastly, to evaluate its suitability for soil amendment, the adsorption capacity of the prepared biochar and its effect on plant (*Cicer*) growth were also investigated.

## 2. Methods and materials

### 2.1. Materials

Napier grass was obtained from Kirloskar Oil Engines Limited, Pune, India. Initially, it was ground in a mixer to obtain particle size in the range of 1–1.5 mm and then oven dried at 378 K for 5 h until constant weight was obtained. After the removal of moisture, 30 g of dried grass was fed into a SS 316 batch reactor for pyrolysis. The reactor was equipped with 1–15 bar range pressure gauge and a temperature indicator with K-type sensor. The grass was pyrolysed at different temperatures up to 873 K with biochar samples obtained at 573, 673, 773, and 873 K, respectively, in separate experiments.

### 2.2. Characterization

The pH of the biochar was measured by shaking 2 g biochar in 40 cm<sup>3</sup> distilled water and 0.1 M KCl solutions for 30 min [12]. The electrical conductivity (EC) was determined using a Jenway 4510 conductivity meter by shaking 1 g biochar with 20 cm<sup>3</sup> distilled water for 1 h. The elemental composition of the biochar viz., C, N, H, O, and S content, was determined using a Perkin–Elmer 240B Elemental Analyser. Ash content was analysed by heating the produced biochar to 1073 K in a muffle furnace in the presence of oxygen for 6 h. The ash content was estimated as the difference in the weight of the char, before and after heating it.

The surface acidity and/or basicity of the biochar were estimated by using a Metrohm Tiamo Auto-titrator [13]. Briefly, 0.15 g biochar was shaken with 15 cm<sup>3</sup> of 0.1 N NaOH for 30 h, filtered and an aliquot of 5 cm<sup>3</sup> of the NaOH filtrate was transferred to 10 cm<sup>3</sup> of 0.1 N HCl solution that neutralized any un-reacted base; the solution was back-titrated with 0.1 N NaOH. Similarly, for surface basicity, 0.15 g biochar was shaken with 15 cm<sup>3</sup> of a 0.1 N HCl solution for 30 h. Further, the slurry was filtered and an aliquot of 5 cm<sup>3</sup> of HCl filtrate was transferred to 10 cm<sup>3</sup> of a 0.1 N NaOH solution, which neutralized the unreacted acid. The solution was back-titrated with a 0.1 N

HCl solution. The base or acid uptake of the biochar was converted to surface acidity or basicity (mmol/g) [14].

The surface functionality of biochar was measured by infrared spectroscopy (IR). To do this, the chars were mixed with KBr (spectrograde; Merck) in a ratio of 1:200 (w/w) and pelletized using a hydraulic press under constant pressure and further analysed using a Bruker/Vertex 80V FTIR spectrophotometer. The IR spectra were recorded at room temperature (298 K) in the region 4000–400 cm<sup>-1</sup>. The internal surface morphology was studied using a JEOL-JSM-Scanning Electron Microscope (SEM) after coating with platinum. The electric current was 15 mA and the accelerating voltage was 20–30 kV. The XRD spectra of biochar were recorded on D8 Advance Bruker X-ray diffractor at the wave length 1.5406 Å.

The surface area, pore volume and pore size distribution of the biochar were determined with a Micro Meritics ASAP-2020 Surface Area Analyser. The samples were oven-dried at 378 K for 5 h before the BET surface analysis. Approximately 0.2–0.3 g of the sample was first degassed on a HighVac<sup>TM</sup> system at 373 K for 4 h, then for a minimum of 24 h at 523 K under a pressure varying as 6.5–9.5 Pa<sub>abs</sub>. The sample was then introduced into a gas adsorption chamber of the surface area analyser to study N<sub>2</sub> sorption at 77 K.

The effect of biochar on soil water retention capacity was determined by adding 1–5% (w/w) biochar into a suspension of soil (20 g) and water (20 cm<sup>3</sup>) and shaking it at room temperature for 24 h. Subsequently, the suspension was filtered and the residual weight was accounted for the measurement of the water retention capacity of the soil.

### 2.3. Biochar adsorption capacity

The adsorption capacity of biochar was studied by investigating its methylene blue (MB) uptake. This was performed by shaking 0.3 g biochar with 300 cm<sup>3</sup> of MB solution (5–60 ppm concentration) in a multi-magnetic stirrer (Biosystem Scientific) for 24 h at 300 K. During the experiments the pH was monitored with a PH-035 (ATC) pH meter. Further, to study the sorption kinetics, 3 cm<sup>3</sup> aliquots were withdrawn at regular intervals, filtered through a 0.45 μm syringe, and its absorbance was measured using UV–visible spectrophotometer at 630 nm. The equilibrium adsorption of MB was evaluated using Eq. 1; here,  $C_0$  and  $C_t$  are the initial and final adsorbate concentrations (mg/dm<sup>3</sup>),  $V$  is the volume of the solution (dm<sup>3</sup>), and  $W$  (g) is the mass of the biochar used.

$$q_e = \frac{(C_0 - C_t) \times V}{W} \quad (1)$$

### 2.4. Biochar application in crop growth trials

The application of biochar for plant (*Cicer*) growth was performed at the Institute of Chemical Technology, Mumbai, located at 19°1'4.73 N and 72°, 50'41.15 E with an altitude of 9 m above from sea level. The mean annual maximum and minimum temperatures of the site are 304.2 K and 296.7 K, respectively, with relative humidity varying between 67 and 78%. Further, the mean annual rainfall of the study area is

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