



## Research paper

# Characterization of castor plant-derived biochars and their effects as soil amendments on seedlings



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## ARTICLE INFO

## Article history:

Received 7 July 2016

Received in revised form

13 September 2016

Accepted 27 June 2017

## Keywords:

Feedstock

Castor bean

Tomato

Biochar

Slow pyrolysis

Root architecture

## ABSTRACT

Castor bean (*Ricinus communis*) is an important non-edible oilseed crop and source of castor oil which has multiple applications ranging from cosmetics to biofuels industry. However, the extraction of castor oil generates large amounts of de-oiled castor cake containing ricin, a highly toxic glycoprotein that requires treatment prior to its valorization. In this study, biochar was produced by slow pyrolysis at 550 °C from castor stalks and de-oiled castor cake, both by-products of castor oil production, in view of sustainable valorisation of a potential large scale cultivation of castor beans in Greece. The produced biochars were highly alkaline and had significant differences in terms of surface area, morphology, nutrients content and ratios of atomic C:N and H:C. The biochars were added to potting mix at different rates (0, 1 and 5% mass fraction of the dry mix) for the cultivation of tomato and castor seedlings, and their growth was monitored over a period of two months without fertilization. Based on the results, castor biochars improved castor seed germination, achieving 90% success rate earlier when compared to control. On the contrary, biochar did not affect significantly the germination of tomato seedlings. However, in both species, the biochar treatments promoted lateral root initiation but increased the developmental rate only in castor. In addition, biochars affected soil cation exchange capacity (CEC), PO<sub>4</sub>, total N and P, K, Na, Ca, and Al levels.

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## 1. Introduction

Currently, castor seeds are used for the extraction of castor oil which has numerous and diverse applications ranging from cosmetics to biofuels industry. The cultivation and processing of castor plants and seeds results in the production of significant amounts of dry residues at harvest (−0.8–3.0 Mg ha<sup>−1</sup> for short hybrids and up to 10 Mg ha<sup>−1</sup> for taller plants) in terms of biomass (i.e stalks and leaves) and de-oiled cake, considering that the oil content of the seeds is about 50% [1,2]. In addition to the amounts of these residues, the de-oiled cake contains ricin which is a highly toxic substance to humans and animals, therefore the need for their processing prior to discarding them arises (reviewed in Ref. [3]).

Thermochemical conversion processes are among the most widely applied technologies for biomass valorization [4]. A

commonly employed thermochemical process is pyrolysis which can be roughly defined as the heating of organic materials at temperatures higher than 300 °C under inert conditions. Pyrolysis at different temperatures and heating rates is used to transform low-energy-density biomass into three main products: (1) a relatively low energy-density gas, (2) a high-energy-density liquid known as bio-oil (fast-pyrolysis) or (3) a high-energy-density solid known as biochar (slow-pyrolysis) [5]. Consequently, biochar can be defined as the carbon-rich product obtained when biomass is pyrolysed, preferably at a slow rate, and it is differentiated from charcoal and similar materials by the fact that it is produced with the intent to be applied to soil as a means of improving soil productivity, carbon (C) storage, or filtration of percolating soil water [6]. Biochar has a high carbon content, low H:C atomic ratio and retains most of the nutrients contained in the original biomass. The important properties of biochar in relation to its use as a soil amendment are the surface area, porosity, electrical conductivity, pH, water holding capacity and cation exchange capacity (CEC). All of these properties are influenced by the original biomass composition and the pyrolysis conditions [7–9]. At high pyrolysis

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temperature and long durations, the biochar aromaticity increases, as demonstrated by the C content, but this is accompanied by less surface groups (due to decomposition) that contribute to the pH and CEC. In addition, nutrient content might decrease (due to formation of volatile species). On the other hand, it has been demonstrated that the surface area and porosity generally increase with longer pyrolysis time and higher temperature [10]. The increased surface area is positively correlated to the water holding capacity however on the downside, a high surface area is connected to well-developed porosity and consequently increased adsorption capacity [11]. Therefore, biochar production conditions should be carefully selected, taking into account its subsequent application.

Intensification of agricultural production to satisfy food demand rapidly depletes soil nutrients and requires heavy fertilizer usage to replenish them. Biochar is being investigated as a promising geo-engineering tool that could potentially mitigate the effects of over-cultivation, improve soil quality and reduce the extensive use of fertilizers [12]. Negative effects of biochars include the binding and deactivation of herbicides and nutrients, release of toxic compounds that may affect plant germination and growth, and a undesirable increase in soil EC and pH that may influence the bioavailability of nutrients (reviewed in Ref. [12]).

Climate change is expected to have a negative impact on soil fertility especially in regions such as the Mediterranean, where increased temperatures will deplete soil organic matter faster as a result of intensified microbial activity. Soil organic matter (SOM) depletion reduces soil fertility which is an important factor in functionality, plant growth and resilience of ecosystems [13]. The frequency of extreme weather events is also expected to increase which may eventually lead to nutrient runoff and topsoil erosion.

Castor seeds and castor cakes have been scarcely reported in the literature as potential materials for the production of biochars [14–17] and activated carbons [18,19], but to the best of the authors' knowledge there is no report for the use of castor derived biochar for soil amendment. Currently, there is a growing interest in the use of biochar in sustainable agriculture [20,21], although its current use is limited because of lack of understanding of its effects in plant productivity. Previous studies on biochar amendment in a few agricultural species such as wheat [22] and maize [23] often gave contradictory results concerning crop yield.

The objective of the current study is to explore the potential of residual de-oiled castor cake and castor stalk field remnants in the production of biochar using castor and tomato as indicator plants. The working hypothesis was that castor biochar could be used as soil amendment for increasing soil fertility and enhancing plant growth. The two biomass species were subjected to slow pyrolysis and the resulting biochars were characterized physicochemically. Subsequently, to test the above hypothesis their effect as soil amendments was investigated during germination and growth of castor and tomato seedlings.

## 2. Materials and methods

### 2.1. Biomass characterization

Castor plants of hybrid H14 [24] were cultivated in Northern Greece (Thessaloniki, Krithia, 22°59'E, 40°50'N) as annual crop (~5 month growth cycle) during the years 2014 and 2015. The castor beans were collected and pressed mechanically for oil extraction. The spent castor cake (thereafter referred as CC) was collected and used in this study as feedstock along with the castor stalks (thereafter referred as CS) remaining during the plant cropping. Both the CC and CS were characterized physicochemically prior to their pyrolysis, in terms of moisture and ash content, elemental analysis and protein, hemicellulose, cellulose and lignin content.

The moisture determination was conducted in an oven at 105 °C where the sample remained for 24 h. The ash content was measured after combustion of biomass in a muffle furnace at 575 °C for 16 h (NREL/TP-510-42622) [25]. The metals were determined by ICP-OES using the Optima 4300DV instrument, by Perkin Elmer. The extractives, hemicellulose, cellulose and lignin content were determined according to the NREL methods (NREL/TP-510-42618 and NREL/TP-510-42619) [26,27]. Finally, the protein content was measured according to the N-Kjeldahl method (AOAC 955.04).

### 2.2. Slow pyrolysis – biochar production

The dried biomass (CC or CS) was milled to 2 mm particles and afterwards submitted to slow pyrolysis. In each case, around 75 g of biomass were loaded in a vertical tubular stainless steel reactor located in an electrically heated three-zone split-tube furnace. Continuous flow of N<sub>2</sub> at 50 cm<sup>3</sup> min<sup>-1</sup> was applied throughout the pyrolysis in order to ensure inert conditions. The oven temperature was increased at a steady rate of 20 °C min<sup>-1</sup> up to 550 °C. The temperature inside the reactor was monitored by a thermocouple, while a reaction time of 30 min was considered from the moment the biomass reached the final temperature. At the end of the reaction time the reactor was removed from the oven and rapidly cooled to room temperature. The gases and liquids produced during the pyrolysis were collected in a cooling trap connected at the exit of the reactor. The resulting samples are denoted as BCC for the castor cake derived biochar and BCS respectively for the castor stalks derived biochar.

### 2.3. Biochar characterization

N<sub>2</sub> adsorption/desorption measurements were employed for the evaluation of the textural properties of biochar by the use of a Quantachrome Autosorb Automated Gas Sorption instrument. Prior to the measurement a sample equal to ~200 mg was heated for 18 h at 250 °C. The surface area (S<sub>BET</sub>) of each biochar was calculated by the Brunauer-Emmett-Teller (BET) equation using the data of the N<sub>2</sub> adsorption isotherm. The micropore volume (V<sub>micro</sub>) was calculated through the t-plot method. Finally total pore volume (V<sub>total</sub>) was measured through the use of Barrett-Joyner-Halenda (BJH) method. The determination of the ash content was performed according to ASTM D2866. The elemental analysis was performed on a CHN-628 elemental analyzer (LECO instruments, USA) according to ASTM D 5291. The presence of further metals was evaluated by ICP-OES spectroscopy using the Optima 4300DV instrument, by Perkin Elmer. Briefly, an appropriate amount of biochar was calcined at 575 °C for 16 h and the resulting ash was diluted with 1 cm<sup>3</sup> of concentrated HF. The mixture was heated to dryness, followed by addition of 1 cm<sup>3</sup> of concentrated HCl and HNO<sub>3</sub> and mild heating until complete dissolution. Finally the sample was diluted to 100 cm<sup>3</sup> with double distilled H<sub>2</sub>O prior to analysis. Each sample analysis was performed in triplicate. Finally, the pH value of the biochars was measured by adding 1.0 ± 0.01 g of sample to 74.55 kg m<sup>-3</sup> (1 M) KCl in deionized water at a mass ratio of 1:10 and measuring the pH value of the supernatant after removal of the biochar [28].

### 2.4. Amendment of growth medium by biochar

The different biochars, BCC and BCS, were thoroughly mixed with a commercial type of soil which contained 40% blond peat, 10% black peat, 20% clay and 30% organic humus (Agricult, Athens, Greece). Subsequently, the biochar-soil mixtures of 0%, 1% BCC, 1% BCS, 5% BCC and 5% BCS (mass fraction on dry weight basis) were transferred to 80 cm<sup>3</sup> pots. Each biochar treatment was

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