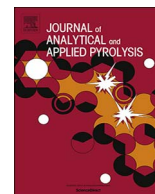




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Biochar from biosolids microwaved-pyrolysis: Characteristics and potential for use as growing media amendment

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ABSTRACT

Biochar, produced from biosolids using microwave pyrolysis technology, is energetically a more efficient alternative to that produced with conventional convective heating. However the potential of microwave generated biochar as a growing media amendment has not been sufficiently explored. Here we produced biochar from biosolids using microwave energy. The pyrolysis experiments were conducted in two stages, initially using a custom built single mode chamber to explore the energetics and product distribution of the pyrolysis process at different temperatures and secondly in a 1 m³ 6 kW multi-mode chamber, to explore potential scale-up of the process. The second phase of the pyrolysis experiments was focused on biochar generation for use in the remainder of this research. Microwave pyrolyzed biochar (MB) was characterised for its chemical and physical properties. Then, we conducted a greenhouse experiment, where we compared the ability of four growing media mixes that combined pine bark with (i) sphagnum peat and fertilizers; (ii) 20% MB and fertilizers; (iii) 60% MB and fertilizers; and (iv) 60% MB and no fertilizers, to promote plant growth and nutrient uptake and to minimise leaching losses. MB had high mesoporosity (average pore width of 4.46 nm), moderate surface area (75 m² g⁻¹), elevated nutrient content and low heavy metal concentrations as compared to other biosolids biochars reported in literatures. Substitution of peat with 60% MB on volume basis reduced leaching loss of nitrate and phosphate from the media but increased leaching loss of ammonium. Addition of MB in conjunction with fertilizer increased plant growth and plant nitrogen and phosphorus use efficiency. Our study has shown microwave pyrolysis as a promising technology for pyrolyzing biosolids and also has demonstrated the synergistic interaction of MB and fertilizer which results in greater plant growth and nutrient uptake and use efficiency.

1. Introduction

Globally, approximately 3.3×10^{12} dry tonnes biosolids (i.e. treated sewage sludge) is produced each year [1], and the count is increasing [2]. Biosolids can be disposed for beneficial uses such as land application (e.g. land reclamation, agriculture, landscaping), industrial processes, resource and energy recovery (via bio-digestion and incineration)[1]. However biosolids landfill disposal is still among the most common uses in higher-income countries (e.g. 30% of biosolids placed in landfill stockpiles in USA, 42% in the European Union) [3]. Alternative sustainable uses of biosolids should be explored.

Pyrolysis of biosolids is one of the ways of managing biosolids with the primary aim of land application of resulting biochar and energy generation from the other pyrolysis products [4]. The process of pyrolysis of biosolids yields: syngas, bio-oil, and biochar [5–7]. Syngas is a combustible fuel. Bio-oil has several potential applications, depending on the chemical composition of the oil. Biochar has recently been used as a soil ameliorant in agricultural systems.

Experimental and theoretical evidence suggests that biochar has a larger impact on environmental outcomes than agricultural production [8] with experiments revealing a non-significant change in some key crop production measures [9,10]; however, there is still considerable interest in quantifying the effect of biochar on cropping systems.

Biochar has commonly been generated in furnaces, using conventional convective heating. However, the use of the conventional method to pyrolyze biosolids is deterred by the high moisture content of this material. This is because convective heat transfer propagates from the surface into the material, with the final temperature profile depending on the material's thermal diffusion properties [11] and the influence of moisture transport out of the heated material, which often hinders the convective heating process [12,13] due to surface cooling associated with evaporation of moisture.

Microwave energy offers a better alternative to conventional convective heating for pyrolysis of biosolids because of its speed. The origin of this speed is the volumetric interactions between the microwave's electric field and the material, and the simultaneous diffusion of heat

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and hot vapours from within the material [14], which reinforces heating rather than hindering it. Thus, microwave heating can generate biochar from biosolids more efficiently. Other advantages of microwave assisted pyrolysis of biomass include: the generation of a wider range of products, especially in the oil fraction of the pyrolysis products [15,16]; energy savings at significantly lower processing temperatures compared with conventional pyrolysis [16]; less feedstock pre-treatment needed [16]; the bio-oil produced from microwave assisted pyrolysis preserves some of the functional groups of the feedstock, which renders the oils less environmentally toxic [17]; and the possibility of continuous processing [16]. Some disadvantages of microwave assisted pyrolysis include: the need to add some form of microwave susceptor to achieve good microwave heating of the dry feedstock; the challenge of scale-up [16]; and non-uniformity of microwave heating [16,18,19].

Although there is evidence that environmental outcomes are more likely than production increases from incorporation of biochar into plant growth media, the potential of the microwave generated biochar as a growing media amendment has not been explored. This study characterizes the physical and chemical properties of microwave biochar using modern analytical techniques and fills the gap in crop production knowledge by investigating the effect of microwave generated biochar on: growing media properties; nitrogen and phosphorus use efficiency; and plant growth.

2. Materials and methods

2.1. Feedstock and preliminary microwave pyrolysis

Biosolids, acquired from the Euroa municipal waste water (sewage) treatment plant was used as feedstock to produce biochar by using an experimental microwave chamber, hence the biochar tested in our study is called microwave biochar (MB).

A preliminary experiment, where 80 g samples of biosolids were subjected to pyrolysis using a 1.17 L, single mode microwave chamber fed from a variable power 1200 W microwave source, was used to determine the yield of pyrolysis products when processed at different temperatures [20]. The pyrolysis chamber was sealed at the top end with a flanged lid that had connections to allow nitrogen gas inflow and a connection for pyrolysis gas removal. The bottom of the chamber was sealed with a fused quartz window to allow the microwave energy to enter the chamber, but create a barrier to prevent product escape. The lid and fused quartz window were sealed with silicone rubber O-rings to create an air tight treatment chamber.

The biosolids were broken up for 30 s, using a food blender, thoroughly mixed with about 10% by mass of activated carbon, obtained from Sigma Aldrich (Ref. 242276) [20]. Pyrolysis temperatures were measured using a shielded type K thermocouple connected to an Arduino board and recorded every 500 ms. The target temperatures in these small scale experiments were achieved by varying the input microwave power to maintain the set-point temperatures of the system based on measurements made by the thermocouple.

The pyrolysis gases were removed using a vacuum pump, and the oil fraction was condensed using a cold-water trap. The vacuum pump was connected to the chamber via a clear silicone tube. The pyrolysis gases were visible through the tube. Pyrolysis was assumed to be completed when there was no longer any visible gas in the extraction tube. The experimental arrangement for this preliminary experiment is thoroughly described in [20]. The amount of non-condensable syngas was calculated as the difference between dry biosolids and the combination of the biochar and oil. The resulting breakdown of products was: biochar 59.93%; biogas 37.70%; and bio-oil 2.37%, as percentages by mass of dry biosolids. The biosolids, biochar and bio-oil were subjected to chemical analyses to determine nitrogen, carbon, hydrogen, sulphur, oxygen and ash content for use in energy recovery calculations. The

chemical composition of the syngas was determined by mass balance calculations, assuming conservation of these elements during the pyrolysis process. The biochar produced during these small scale experiments, which was not subjected to chemical analyses, was conserved for use in larger scale experiments described later. It was assumed that the initial 10% concentration of the activated carbon in these experiments would be significantly diluted in the final biochar samples used for the plant growth experiments described later. The following equation was used to calculate the anticipated high heating value (HHV) of the biosolids and biochar [21]:

$$HHV, \text{ kJ/gDrySludge} = 0.3515[C\%] + 1.617[H\%] + 0.1232[S\%] - 0.1198[O\% + N\%] - 0.0153[A\%] \quad (1)$$

The calorific values of the bio-oil and syngas were determined from the Boie equation [22]:

$$HHV, \text{ kJ/g} = 0.3516[C\%] + 1.16225[H\%] - 0.11090[O\%] + 0.06280[N\%] + 0.10465[S\%] \quad (2)$$

2.2. Microwave pyrolysis of larger biosolids samples

Biosolids were treated in a 6000 W, 1 m³, multi-mode microwave chamber, operating at 2.45 GHz [23]. The experimental chamber was located at Dookie campus of the University of Melbourne. The mass of these samples was approximately 2340 g. The samples were broken up for 30 s, using a food blender, thoroughly mixed with about 10% by mass of biochar that was produced from earlier experiments, and placed into a 4 L fused quartz crucible with a close fitting lid. The previously generated biochar, from the small chamber experiments, acts as a microwave susceptor. The susceptor material was necessary because the dielectric properties of dry biosolids is very low [24] and therefore microwave heating of the samples would be negligible. Mixing a small amount of a material like carbon or biochar, which has much higher dielectric properties than biosolids, can initiate the microwave heating process by absorbing microwave energy and transferring the generated heat into the surrounding biosolids. As the pyrolysis reaction initiates, more biochar is created and therefore more microwave energy is absorbed by the sample, creating a thermal runaway effect [25,26]. The quartz crucible limited the amount of oxygen available to the samples during the treatment, therefore ensuring that the biosolids underwent pyrolysis rather than combustion. Samples were placed into the multi-mode chamber with the input microwave power set to maximum (6000 W).

The experimental microwave chamber has been thoroughly described elsewhere [23]. Because this experiment focused on the production of biochar, the syngas and oil fractions, produced from this main experiment, were vented to the atmosphere. Pyrolysis was assumed to have commenced when volatiles, which condensed quickly and appeared as 'smoke' in the air-steam exhausting from the microwave chamber, were first observed. Pyrolysis of the samples was assumed to be completed when the smoke in the air stream venting from the microwave chamber reduced to a negligible amount. Treatment was allowed to continue for a further 10 min to ensure the pyrolysis reaction for the whole sample was completed. Because of the design of this large microwave chamber, thermocouple measurement of the pyrolysis temperature was not possible; however, a thermal camera (FLIR T640) was used to record the temperature of the system and the samples after treatment. Our microwave pyrolysis process took less than 30 min which is considerably faster than conventional slow pyrolysis which generally takes few hours to complete [4]. Because the biochar susceptor, which was used in these larger scale experiments, was derived from the same biosolids feedstock, it was assumed that the susceptor material's contribution to the final biochar product's properties would be comparable to that of the fresh feedstock.

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