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An investigation of drum granulation of biochar powder

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ABSTRACT

Biochar is a fine powder that is difficult to handle and would be blown away if applied to soils without modifications. Wet drum granulation of biochar using binder solutions of hydroxypropyl methylcellulose was investigated to create a form that would be easier to handle and apply effectively to the soil. The biochar granules were free flowing and approximately spherical with a significant yield between a size range of 1 to 4 mm. The binder concentration, total binder solution volume and drum rotational speed all affected the yield of granules within this size range. The drum rotational speed and binder concentration affected the granule strength; biochar granules were resistant to attrition and showed a crushing strength of 0.15 to 0.50 MPa. The results demonstrated that biochar can be granulated using wet drum granulation. Further research is required to further refine the granulation parameters to produce granules with comparable characteristics to fertilizers to ensure that the granules could be effectively applied to soils using existing infrastructure and techniques.

1. Introduction

Biochar, solids produced from the pyrolysis process, can be used agriculturally as a soil amendment. When cultivated into soils, biochar has been shown to reduce nutrient leaching, immobilize heavy metals, and increase water retention and microbial activity [1–5]. As biochar properties can be adjusted, it has also been proposed that biochar can be fine tuned for specific soil needs. For example, biochar derived from a glucose source stimulated Gram-negative bacteria growth, while yeast derived biochar promoted fungi growth [6].

The major problem with biochar is that it is a fine powder with particle sizes ranging from approximately 1–600 μm and therefore would be easily blown away when used as a soil amendment without any modifications. When biochar particles become airborne, they can negatively impact the health of exposed occupants causing respiratory irritation and lung damage [7]. In addition, very fine powders make packaging difficult and cultivation into soil troublesome. Techniques that have been proposed for application of biochar to soils that minimize dust hazards include surface spreading followed by immediate plowing into the soil or spraying with water, mixing with liquids and applying using liquid spreading techniques, or pelletizing the biochar and applying as for solid fertilizers [7]. Dumrose et al. pelletized biochar after blending with wood flour, polylactic acid and starch [1]. The pellets were combined with *Sphagnum* peat and the combinations were assessed for potential use in container nurseries. The pellets improved the hydraulic conductivity of the mixture, but expansion at high pellet additions caused potential problems in filling the containers. Recommendations included modifications of pellet composition and porosity to reduce pellet expansion and also improve the carbon to nitrogen ratio of the mixture.

Wet granulation is a process that uses a liquid binder to agglomerate particles into granules. There are three main wet granulation processes: high shear, fluidized bed, and drum. Drum granulation would be the best choice for biochar granulation due to its lower capital and operating costs combined with easier scale-up. In drum granulation, powder is loaded into the drum and agitated by rotating the drum while a liquid binder is sprayed onto the powder surface using spargers. The liquid binder wets the particles allowing granule nuclei to form which then develop into larger granules followed by consolidation into the final product.

The objectives of the current research were to demonstrate that biochar could be granulated and then to investigate process parameters that affect biochar granule properties.

2. Materials and methods

2.1. Biochar formation

Biochar was created by the pyrolysis of cornstalk. The cornstalk was treated before pyrolysis by drying at 105 °C and then grinding to 1–2 mm in size using an IKA Werke model MF10 grinder at 4500 rpm. Pyrolysis was performed in a custom manufactured reactor (University Machine Services, London, Canada) with a diameter of 25 cm and an effective bed height of 60 cm. The reactor temperature was 500 °C and pressure was 100 kPa. The bed was fixed, but then fluidized at intervals to ensure constant temperature and uniform bed composition. The

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cornstalk biochar was recovered from the reactor and then ground using the IKA Werke model MF10 grinder. This step only reduced any large clumps of biochar that may have been recovered from the reactor. It was important to eliminate these large clumps at this stage to accurately determine the size enlargement of granulation.

2.2. Biochar characterization

Images of the biochar powder were taken using a Hitachi S-4500 field emission scanning electron microscope. The powder was mounted on a plate using a carbon adhesive and a thin layer of gold was deposited on the sample surface to minimize sample charging. The images allowed the shape and morphology of the biochar powder to be examined. The particle size distribution of the biochar powder was measured using a Malvern Mastersizer 2000.

Flowability of the biochar powder was examined using a Mercury Scientific Revolution Powder Analyzer. A powder sample size of 118 cm^3 was loaded into a drum with a diameter of 11 cm and width of 3.5 cm. The drum was rotated at 0.3 rpm until 128 avalanches had occurred with an avalanche defined as being a rearrangement of at least 0.65 vol.% of the sample in the drum. Optical measurements with a resolution of 648 × 488 at 60 frames per second allowed the powder surface to be measured as the sample was rotated. Software calculated various flowability indicators. Samples were measured in triplicate.

Drop penetration tests were conducted to examine the interaction between the liquid binder solutions and the biochar powder. The drop penetration measurements were conducted using a procedure similar to the one outlined by Hapgood et al. [8]. The biochar powder was sieved through a 1.4 mm sieve into a petri dish. A spatula removed excess powder to create a loosely packed bed of biochar powder with a level surface. A 25 gauge needle syringe was mounted 1 cm above the powder bed. A liquid binder droplet of 0.0048 ml was gently dropped on the powder bed and a video of the drop penetration was filmed. The procedure was repeated in triplicate. The drop penetration time was determined as the time at which the liquid droplet was no longer visible at the powder bed surface.

Hydroxypropyl methylcellulose (HPMC) (Pharmacoat® 603, Shin-Etsu Chemical Co.) was used as the binder. HPMC is a commonly used binder for granulation of pharmaceutical powders [9]. Binder solutions were prepared by heating distilled water to approximately 85 °C and then gradually adding the required amount of HPMC powder. The solutions were stirred until the powder fully dissolved and cooled to about 21 °C before conducting experimental trials. Viscosity measurements of the binder solutions were conducted using a Brookfield Viscometer at 20 rpm with a 61 spindle.

The hygroscopicity of the biochar powder was determined by measuring the change in moisture content of the powder after exposure to air at different humidity levels. The biochar powder was spread into a thin layer on trays and placed in a humidity chamber for 48 h. The humidity of the air within the chamber was adjusted by varying the flow ratio of dry air and air humidified by bubbling through water in a column. The temperature and the humidity of the air within the humidity chamber were measured using dry and wet bulb temperature sensors. After 48 h within the humidity chamber, the moisture content of the biochar powder was determined using a Mettler-Toledo HG63 moisture analyzer based on weight loss-on drying at 105 °C; triplicate samples of approximately 3 g were analyzed.

2.3. Granulation

Experimental design allows a series of experimental trials to be planned and conducted in a specific order that maximizes the information gained with a minimum number of trials. StatEase Design Expert 8 statistical software (Stat-Ease, Inc. Minnesota, USA) was used to generate a two-level factorial design of experiments (DoE) to determine the effect of concentration of binder in the liquid solution, volume of liquid binder solution added, and drum rotational speed. Based on preliminary trials, the binder concentration was varied from 3 wt.% to 9 wt.% with a centre value of 6 wt.%, the binder solution volume ranged from 17.9 to 20.5 to 23 ml to correspond to 70, 80 and 90 wt.% of binder solution to biochar mass, and the drum rotational speeds were 40, 50 and 60 rpm. An analysis of variance (ANOVA) was conducted with optimal granule size yield, flowability, attrition resistance and crushing strength as the response variables. P-values less than 0.05 were considered significant, indicating at least a 95% confidence level for a specific response variable.

A schematic diagram of the drum granulator is shown in Fig. 1. The drum had an inner diameter of 7.5 cm, an inner length of 12.0 cm and was made of transparent Plexiglas to allow visual observations. One end of the drum was connected to a motor which allowed for the drum to be rotated. The other end of the drum had an opening to allow a sparger to be inserted. The sparger spanned the length of the drum, had an inner diameter of 3.0 mm, and had four 1.0 mm holes about 3.0 cm apart axially. The sparger was attached to a peristaltic pump to deliver the liquid binder solution. The binder solution was added drop wise onto the powder bed surface with a mean droplet volume of 0.08 ml and at a rate of approximately 8.5 ml/min. The drum granulator and sparger system were custom manufactured by University Machine Services (London, Canada).

For the granulation trials, 25 g of the biochar powder was added to the granulation drum. The drum was rotated at the required speed for the specified trial. Liquid binder solution was added drop wise onto the cascading powder surface until the specified volume for a given trial. Liquid binder addition was then stopped. The drum was rotated for two more minutes to allow wet massing before the granulation was stopped. Granules were removed, spread onto trays and dried at 24 °C and a relative humidity of 3–5% for more than 24 h. The dried granules were then analyzed for various properties.

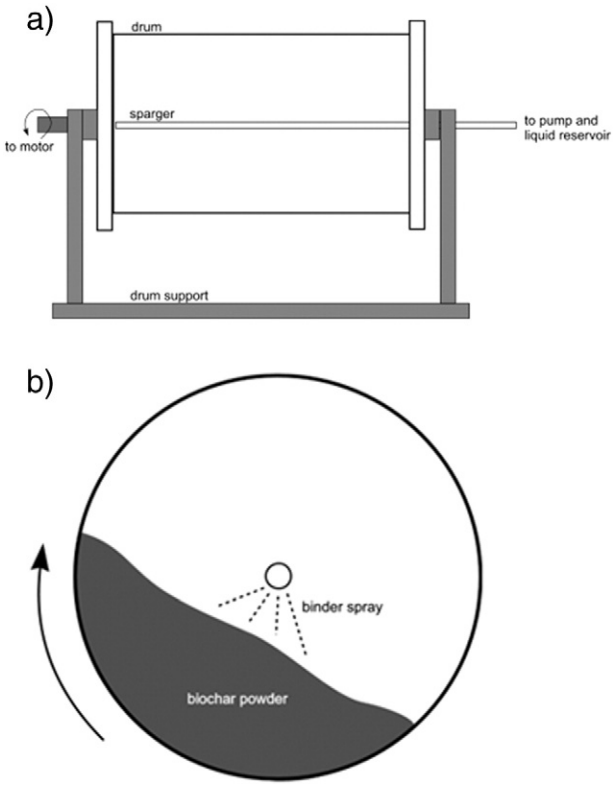

Fig. 1. Schematic diagram of the drum granulator.