



# Evaluation of biodegradable polymers as encapsulating agents for the development of a urea controlled-release fertilizer using biochar as support material



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## HIGHLIGHTS

- Polymeric coating materials retard N leaching from biochar based fertilizers.
- N-species concentration in leachates depends on polymeric materials nature.
- Biochar did influence N release in the tested fertilizers.

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## ABSTRACT

Biochar constitutes a promising support material for the formulation of controlled-release fertilizers (CRFs). In this study we evaluated the effect of different polymeric materials as encapsulating agents to control nitrogen (N) leaching from biochar based CRFs. Nitrogen impregnation onto biochar was performed in a batch reactor using urea as N source. The resulting product was encapsulated by using sodium alginate (SA), cellulose acetate (CA) and ethyl cellulose (EC). Leaching potential was studied in planted and unplanted soil columns, monitoring nitrate, nitrite, ammonium and urea concentrations. After 90 days, plants were removed from the soil columns and plant yield was evaluated. It was observed that the ammonium concentration in leachates presented a maximum concentration for all treatments at day 22. The highest concentration of N in the leachates was the nitrate form. The crop yield was negatively affected by all developed CRFs using biochar compared with the traditional fertilization.

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## 1. Introduction

Nutrient fertilization is a key factor in crop production. From an economic viewpoint, N-fertilizer is the most important chemical input in crop production. Crop N use efficiency from urea or other N-fertilizer sources is about 30–40%, depending on the culture conditions. Losses of N-fertilizer from cultivated soils via ammonia (NH<sub>3</sub>) volatilization, nitrate (NO<sub>3</sub><sup>-</sup>) leaching and nitrous oxide (N<sub>2</sub>O) emissions have a high economic and environmental impact (Ramírez et al., 1997; Zhou et al., 2003).

One way to improve grain quality and N uptake efficiency while reducing the environmental impacts is through the use of controlled-release fertilizers (CRFs) (Wu et al., 2008). The CRF products are developed to release nutrients gradually trying to coincide with the nutrient demand during crop growth. These fertilizers are prepared by coating the active soluble nutrient component with a membrane that serves as a diffusion barrier. Several polymer materials have been proposed for CRF encapsulation. The most important of these include – among others – wax and sulfur, as well as organic polymers such as Kraft pine lignin (García et al., 1996), cellulose acetate (Jarosiewicz and Tomaszewska, 2003), and sodium alginate (Liang et al., 2007).

Biochar is a carbon-rich material obtained from the thermal pyrolysis of agricultural residues or other lignocellulosic biomass at moderately high temperature (Lehmann and Joseph, 2009; Lee et al., 2013). Biochar is a value-added product, which can be used

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for several purposes such as a support material for delivering plant nutrients (González et al., 2012). It is well known that the biochar is used as a soil amendment due to positive effects, including agricultural profitability, management of pollution and eutrophication risk to the environment, restoration of degraded land and sequestration of C from the atmosphere (Blackwell et al., 2009).

Published studies suggest the modification of biochar before their incorporation into the soil as a bio-fertilizer. These modifications include the addition of one or more nutrients either by a direct mixing process, encapsulation or pelletization, among other techniques (Radlein et al., 1997; Kotaka, 2005; Magrini-Bair et al., 2009).

The use of polymeric materials has not been reported yet in the formulation of a CRF using biochar as support material. Thus, it is important to evaluate nutrient release rates and associated mechanisms in order to develop proper CRFs for crop production.

The main objective of this research was to evaluate the influence of biodegradable polymers on the release and leaching of N from CRF as well as the yield of wheat (*Triticum aestivum*) under greenhouse conditions. Sodium alginate (SA), cellulose acetate (CA) and ethyl cellulose (EC) were used as encapsulating agents for the development of CRFs using biochar as support material.

## 2. Material and methods

### 2.1. Biochar

Biochar was generated by slow pyrolysis of oat hull at 300 °C in a reactor with a capacity to process about 5 kg of biomass per batch; the operational parameters were described in a previous work (González et al., 2013). When loaded with biomass, the reactor was purged with N<sub>2</sub> at a flow rate of 5 L min<sup>-1</sup> to remove the oxygen present in the chamber. After the pyrolysis process, the obtained biochar was crushed using a high-speed rotary cutting mill and sieved to obtain the desired particle size range ( $\leq 500 \mu\text{m}$ ). The physicochemical properties of the obtained biochar were previously reported by González et al. (2013).

### 2.2. Urea impregnation onto biochar

Impregnation experiments were carried out in a batch reactor according to the partially modified methodology of Bimer et al. (1998), lower temperatures and reaction time, and different reagent ratios were used. Biochar was mixed with urea in aqueous media in a metallic pressure cooker of 5 L. The ratio of biochar:urea-N:deionized water was 1:0.5:5 to give an urea concentration of 3.66 M. The impregnation with urea was carried out at constant temperature of 150 ± 5 °C for 10 min, and at 1.0 bar. The nitrogen content in the biochar impregnated with urea was determined by the Kjeldahl method

(Sadzawka et al., 2004). The amount of urea retained onto biochar was 50% of the initial concentration.

### 2.3. Evaluation of polymers for CRF formulations

The polymers used for the preparation of encapsulates were cellulose acetate (CA) from Acros, ethyl cellulose (EC) from Sigma and sodium alginate (SA) from Sigma. Formamide (F) from Merck was used as modifying agent for the preparation of CA and EC solutions. The addition of F as a modifier agent is essential for the preparation of encapsulates by the phase inversion technique. Without the addition of this compound, the result is the dissociation of the mixture, forming an asymmetric colloidal membrane. On the other hand, the presence of formamide increases the solvent loss (acetone) until the miscibility of the polymeric solution in water is no longer possible.

The polymer solutions were prepared by dissolution of the solid polymer in an adequate solvent. Acetone was used for CA and EC, whereas distilled water was used for SA. The proportions of solvent and modifying agent to polymeric materials are shown in Table 1. The densities (gravimetric method) and viscosities (digital viscometer VIS-79) of the resulting polymer solutions were measured at room temperature (Table 1).

Encapsulates of CRFs were developed from the mixture of the polymeric solution and impregnated biochar through the phase inversion technique for the polymers CA and EC (Jarosiewicz and Tomaszewska, 2003). Impregnated biochar with urea was gradually added to the polymeric solutions in different proportions (see Table 1) and dropped into the precipitation bath (distilled water), where the solvent–nonsolvent exchange proceeded, resulting in the formation of encapsulates (gelation process). The temperature of the precipitation bath was 25 °C. The encapsulated biochar beads were left in the distilled water for 5 min to ensure complete gelling; then, they were separated and dried under a stream of air.

For the formulation of encapsulates using SA, cross-linking technique was used. The mixture between polymer and biochar in different proportions (see Table 1) was dropped into the precipitation bath of CaCl<sub>2</sub>. The semi-spherical beads formed were left in the CaCl<sub>2</sub> solution for 5 min to ensure complete gelling and then separated and dried under a stream of air. The average diameters of beads were approximately between 2 and 4 mm.

Nitrogen content of the encapsulate samples were determined by Kjeldahl method (Sadzawka et al., 2004). The morphology of selected samples was analyzed using a scanning electron microscope (SEM Quanta 600 FEI from, FEI Inc., Hillsboro, OR, USA) at 20 kV.

Table 1 shows the physicochemical properties of the polymer solutions and nitrogen content of the CRFs used for leaching test evaluation.

**Table 1**

Composition of the biochar encapsulates used in the leaching test evaluation. C: control; U: urea; ESN: commercial N-CRF; BU: biochar impregnated with non-encapsulated nitrogen; SA 1: biochar + urea + SA 1% (R:1/1); SA 2: biochar + urea + SA 2.5% (R:1/5); EC 1: biochar + urea + EC 10% 10% F (R:1/4); EC 2: biochar + urea + EC 10% 15% F (R:1/4); CA 1: biochar + urea + CA 10% 10% F (R:1/2) and CA 2: Biochar + urea + CA 10% 15% F (R:1/6).

Sample	Polymer concentration (wt.%)	Solvent concentration (wt/v)	Modifying agent concentration (F) (wt/v)	Polymer viscosity <sup>a</sup> (cP)	Polymer density <sup>b</sup> (kg m <sup>-3</sup> )	Proportion biochar/polymer (wt/wt) (R)	Nitrogen content (%)	Biochar content (%)
CA 1	10	80	10	127	877	1/2	15.6 ± 0.5	23.4
CA 2	10	75	15	150	908	1/6	8.9 ± 0.4	10.0
EC 1	10	80	10	321	870	1/4	14.2 ± 0.3	14.0
EC 2	10	75	15	379	888	1/4	14.5 ± 0.4	14.0
SA 1	1	–	–	202	1006	1/1	15.0 ± 1.1	35.1
SA 2	2.5	–	–	43226	1119	1/5	14.7 ± 1.3	11.7
BU	–	–	–	–	–	–	25.7 ± 0.4	–
U	–	–	–	–	–	–	46	–
ESN	–	–	–	–	–	–	44	–

<sup>a</sup> Viscosities were measured by a digital viscometer VIS-79 at room temperature.

<sup>b</sup> Densities were obtained by gravimetric method at room temperature.

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