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Range analysis of *Eucalyptus globulus* bark low-temperature hydrothermal treatment to produce a new component for growing media industry

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ABSTRACT

The use of industrial *Eucalyptus globulus* bark residues for organic growing media formulation was studied. Hydrothermal treatments were tested using Response Surface Methodology approach. Model design consisted of twelve combinations of temperature (T: 60–140 °C) and residential time (t: 20–60′) to evaluate the effect on bark properties. Temperature had a significant effect in C mineralization and N immobilization rates, where the lowest responses (111.8 mmol CO_2 kg^{-1} d^{-1} and NIR = 4.1 mmol N kg^{-1} d^{-1}, respectively) compared to IEB (214.6 mmol CO_2 kg^{-1} d^{-1} and 8.9 N kg^{-1} d^{-1}, respectively) were suggested after modeling at 40 °C during 70′. Industrial bark was phytotoxic and treatments were effective for phytotoxicity removal. Industrial bark presented high air content but low water availability; treatments had no effect on bark physical properties and the use of demineralized water may have leached nutrient content. Results from pot experiment recommend the use of 25% (v v^{-1}) of treated barks in future growing media formulations.

1. Introduction

In European horticultural industry, the peat presents almost 80% of total plant and seedling growing media (Gruda, 2012a). It is available in Northern Europe as a cheap resource widely used either pure or as the main constituent of growing media (Gruda, 2012a, 2012b; Barrett et al., 2016). No other natural raw-material offers that many advantages as growing media: regular consistency, lightweight, good air and water-holding capacities, low pH and nutrient content (easy to control) and a biological stable structure. However, peat is a limited resource with a great demand, and the extraction of peat bogs causes negative impacts on environment, decreasing the carbon sink in peat bogs and releasing greenhouse gases into the atmosphere through degradation and oxidation of the unsaturated peat layer, which produces an estimated annual emission equivalent of 15 million tons of carbon (Gruda, 2012a, 2012b; Barrett et al., 2016).

The increased environmental awareness of peatland conservation has stimulated intensive research aiming to reduce the use of peat in growing media (peat-reduced growing media), replacing it, totally or in part, by new materials. Recently, organic materials derived from agricultural and municipal waste streams, as well as industrial by-products have become common (Ribeiro et al., 2009; Gruda, 2012a; Barrett et al., 2016). Their characterization and performance interpretation have been supported by multiple approaches, making them difficult to compare and dependent on crop culture used (Barrett et al., 2016; Depardieu et al., 2016).

Organic materials alternative to peat that are already commercialized include composted biowastes, coir, bark and wood fiber (Gruda, 2012a). For instance, from coconut husk, different products are available based on particle size, including chips of various sizes, fiber of various lengths or coir dust material. However, the raw material used in these products is produced out of Europe, for example in India, Sri Lanka, Vietnam, Philippines and Ivory Coast (Maher et al., 2008). The long transportation distance makes this alternative less attractive and less “environmentally friendly” when compared to locally sourced materials. For this reason, European countries are focused in pine, spruce and other softwoods as the main source of raw materials for plant growth substrates, regarding the availability of residues from forest harvest or from wood processing industries (Ribeiro et al., 2009; Jackson et al., 2010; Gruda, 2012b; Barrett et al., 2016).

In Portugal, *Eucalyptus globulus* is the predominant species for pulp and paper production (Domingues et al., 2010; Neiva et al.,

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2016) and around 500,000 tons of *E. globulus* bark (CELPA, 2015) are generated as industrial residue used mainly as a solid biofuel in power plants inside of pulp and paper industries (Gruda, 2012a; Domingues et al., 2013; Neiva et al., 2016). Other actual or potential uses to valorize bark residues were investigated by several researchers such incorporation in the pulping process (Neiva et al., 2016), source of chemical compounds with biological and pharmacological activities (Domingues et al., 2010, 2013), or incorporation into the soil to improve its structure and fertility (Murphy et al., 2010).

E. globulus bark as raw-material for substrate production alternative to peat was investigated in this paper. In literature, there is no information or data on this potential valorization of *E. globulus* bark.

Phytotoxicity is a common problem to solve when the forest residual biomass is used as a component of growing media, since the presence of phenolic compounds, terpenes and tannins are typical in the chemical composition (Caron et al., 2010; Gruda, 2012b). Previous studies demonstrated that *E. globulus* bark is rich in phenolic, triterpenic and other inhibitory compounds (Domingues et al., 2010; Neiva et al., 2016). A different approach has been suggested to eliminate the phytotoxicity from the forested materials and to create a biological stable environment for plant growth (Gruda, 2012b). Buamscha et al. (2008) studied the differences in plant growth between fresh and aged Douglas fir (*Pseudotsuga menziesii*) bark used as growing media and found out that plants were smaller in fresh bark than in the aged one. Gruda et al. (2009) reported improvements in germination rate and radicle growth after washing/leaching pine tree bark based substrates. Cunha-Queda et al. (2007) and Jackson et al. (2010) proposed composting pine bark to remove phytotoxicity and to increase low electrical conductivity. From the techno-economical point of view, the more raw-material transformation required the higher associated cost, potentiating low-temperature hydrothermal treatment as an attractive process due to its simplicity and rapid implementation, while using water as the main reagent (Barrett et al., 2016).

The aim of this study was to investigate the effect of hydrothermal treatment in the phytotoxic compounds removal from the industrial *E. globulus* bark. A range analysis of theoretical treatment residence time and temperature was carried out in order to improve *E. globulus* bark physical, chemical and biological properties. Based on the initial treatments, was evaluated the viability of adding up to 25% of *E. globulus* bark for structural improvement of commercial peat-based growing media.

2. Material and methods

2.1. Raw-material

Industrial *E. globulus* bark (IEB) was collected from The Navigator Company pulp mill (Setúbal, Portugal). Raw-material was gridded in a knife mill (Ø ≤ 6 mm). Granulometry revealed more than half (64% of total weight) bark fibers distributed within particle sizes of 5–1 mm, around 11% of fines (<1 mm), and 25% of coarse particles (>5 mm). Peat moss (PM) slightly decomposed (H2-H5 on Von post scale) from Floragard Co. (Germany), amended with 4 g L^{-1} of calcitic lime and 4 g L^{-1} of dolomitic lime to adjust pH (to 5.6–5.8), was used as a commercial standard.

2.2. Low-temperature hydrothermal treatment

Hydrothermal treatment of industrial *E. globulus* bark (HTEB) was performed using Response Surface Methodology (RSM) and Central Composite Design (CCD), which consisted in modeling the simultaneous effect of two process variables, temperature (T) and retention time (t).

Table 1 summarizes the experimental design matrix with natural (T and t), and the correspondent coded (X_1 and X_2) factor values for three-factor levels (−1, +1 and α). Based on preliminary tests of independent variables, the input parameters were minimum and maximum T = 60 and 140 °C (X_1 = -1 and + 1), and t = 20 and 60 min (X_2 = −1 and +1). To maintain rotatability in full factorial design, when the number of factors (k) is equal to 2, α value was calculated by Eq. (1):

$$\alpha = 2k^{[1/4]} = 1.414 \quad (1)$$

Determination of experiment number (runs) is essential to achieve desired responses with reliable measurements. Physical, chemical, and biological properties were the responses of the system, and the number of model runs required that guarantees the feasibility of CCD was found to be 12 (four factorial, four star and four central).

The HTEBs were performed in autoclave reactor/conditions placing 5 individual hermetic vessels (1 L) containing 90 g (10% moisture content) of bark and 900 ml of water. Under the present experimental condition, the autoclave pressures ranged between 0 (run V; T = 43 °C) to 0.6178 MPa (run VI; T = 157 °C). Treatment liquor fractions were collected and frizzed for further analysis. Water excess from the solid treated material was removed by centrifugation. All experiments were performed in randomized order to minimize uncontrolled factors.

2.3. Physical and chemical analysis

Water-air relationships, as defined by Wallach (2008), were determined according to the European Standard (CEN, 2011a). The physical properties were obtained: total porosity (TP); bulk density (BD), air-filled porosity (AFP) as the amount of air at a suction of 1 kPa, easy available water (EAW) as the difference between the water content at suctions of 1 and 5 kPa, and water buffer capacity (WBC) as the difference between the water content at 5 and 10 kPa. Electrical conductivity, pH, and water-soluble K, P, Ca, Mg, Na and mineral N (NH_4^+-N and NO_3^--N) were measured in water extract 1:5 by volume, according to the European Standards (CEN, 1999a, 1999b, 2001).

The dry mass (DM) content was assessed by oven-drying bark at 105 °C for 24 h and the ash content was determined by combustion of the oven-dried sample at 550 °C for 5 h in a muffle furnace. Afterward, the difference between DM and ash was considered the organic matter (OM) content.

Table 1
Experimental design matrix.

	Runs	Independent variables			
		Coded		Natural	
		X_1	X_2	T (°C)	t (min)
Factorial k^2	I	−1	−1	60	20
	II	−1	1	60	60
	III	1	−1	140	20
	IV	1	1	140	60
Star	V	−α	0	43	40
	VI	α	0	157	40
	VII	0	−α	100	12
	VIII	0	α	100	68
Central	IX	0	0	100	40
	X	0	0	100	40
	XI	0	0	100	40
	XII	0	0	100	40