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Revalorization of spent coffee residues by a direct agronomic approach

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ABSTRACT

Spent coffee grounds (SCG) represent a high-volume food waste worldwide, and several reuse approaches have been attempted. Herein, a greenhouse field experiment was carried out by cultivating Batavia lettuce with 5%, 10%, 15%, 20% and 30% (v/v) espresso SCG directly composted in the soil. Healthy vegetables were obtained for all treatments, without yield loss for up to 10% SCG. A progressive increment of green color intensity with increasing SCG content was observed, corroborated by the increase of their photosynthetic pigments (chlorophylls and carotenoids). Furthermore, total ascorbic acid and tocopherols showed statistical significant increases ($p < 0.001$) between control and all tested groups. Marked variations of nutritionally relevant minerals, particularly potassium, phosphorous and sodium were also revealed at higher percentage treatments (20% and 30%). This approach constitutes a clean, direct, simple and cost-effective measure to produce value-added vegetables, while reducing food waste disposal.

1. Introduction

Modern food consumption habits are leading to severe environmental problems due to uncontrolled waste discharge. Coffee industry, for instance, releases a huge variety and volume of residues, resulting either from its production (e.g., coffee pulp, cherry husks, defective beans, and parchment skin), from roasting industries (e.g., coffee silverskin), from soluble coffee industry (industrial spent coffee), and also directly by daily coffee consumers after beverage preparation (spent coffee grounds, SCG). All these coffee wastes set up a global ecotoxicological concern due to their high content of organic matter and bioactive components as caffeine, free phenols and tannins (polyphenols), with recognized toxicity (Cruz et al., 2012).

While several applications have been proposed for many of the aforementioned coffee by-products (Murthy & Naidu, 2012), the compositional worth of SCG, resulting from beverage preparation, only recently has received proper attention. Hence, distinct applications for this particular residue have been highlighted, such as CO_2/mineral adsorbents, as fuel pellets, as substrate for the extraction of compounds with huge potential applications in the industrial or pharmaceutical fields (Folmer, 2014). The potential application of espresso SCG in agriculture was already been tested in previous works, with positive outcomes on plenty micronutrients in pot experiments using SCG directly and after classical composting (Cruz, Gomes et al., 2014; Cruz, Morais et al., 2014). However, no field experiments on horticultural production simulation in an industrial scale have been performed yet on this subject.

Therefore, aiming to support a cost-effective, simple, direct and sustained reuse of this coffee by-product, the current work was developed using direct composting of espresso SCG on the soil (pit or trench composting), for lettuce cultivation, being the overall performance evaluated through vegetables physical and nutritional quality, including yield, color, pigments, vitamins, total phenols and minerals.

2. Materials and methods

2.1. Reagents

L-Ascorbic acid, caffeine, gallic acid, D-(+)-glucose and tocopherols (α-, and γ-) were purchased from Sigma-Aldrich (Germany). Tocol, used as internal standard for tocopherol quantification, was from Matreya Inc. (USA).

Methanol and n-hexane, both HPLC grade, were from Sigma-Aldrich. All the remaining reagents were analytical grade from several suppliers and included: acetone, anhydrous sodium sulfate, butylated hydroxytoluene (BHT), caesium chloride, chloroform, 1,4-dioxane, Folin and Ciocalteu's reagent (FCR), glacial acetic acid, magnesium oxide, metaphosphoric acid, methanol, nitric acid, sodium acetate, sodium dichromate dihydrated, sodium carbonate, sulfuric acid, tris(2-carboxyethyl)phosphine (TCEP), this last used for ascorbic acid extraction as a 2.5 mM aqueous solution with 3% (w/v) metaphosphoric acid and 8% (v/v) glacial acetic acid.

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2.2. Experimental design

The experimental setup for this study was a randomized greenhouse field design performed at the School of Agriculture, Polytechnic Institute of Bragança (NE Portugal), under controlled conditions (day/night thermal regime of 23/18 ± 2 °C, 70 ± 10% relative humidity, and natural sunlight).

A total of approximately 100 kg of espresso SCG were collected from several coffee shops in January of 2013. The greenhouse ground was prepared to receive 5 × 5 random distributions of 40 L holes (100 cm length × 20 cm width), covered with plastic film. Five SCG mixtures with plain vegetable soil were prepared, with 0%, 5%, 10%, 20%, and 30%, all on a volume basis, in a total of 200 L each. On a mass basis, it corresponds approximately to 9–70% of fresh SCG. Soil samples of the initial soil mixtures were collected for analysis, being preserved in plastic bags at −18 °C ("pre-composting"). The holes were filled with the mixtures, watered, and left to rest for a 4 month period. After this direct composting period, the soil was revolved and homogenized, and soil samples were collected again ("planting"). Batavia lettuce plantlets (*Lactuca sativa* L. var. *capitata* cv. "Rolina") were planted, five on each plot with a 50 × 20 cm compass, in a total of 125 plants.

Lettuce plants were harvested after 5 weeks, carefully washed with deionized water to remove soil contamination, and the edible part was separated and weighted (Fig. 1). Afterwards, 15 plants from each test group were assembled into five composites of three plants, named hereafter as samples and transported immediately to the lab under refrigeration. After color evaluation, whole leaves were immediately frozen at −80 °C in polyethylene bags and then freeze-dried (Telstar Cryodos-80, Spain). Dried samples were carefully homogenized by grinding in a blender (Grindomix, Retsch GmbH, Germany) and further sieved (150 μm mesh size). Pre-freezing and post-lyophilization moisture was evaluated by oven drying at 103 ± 2 °C (WTC Binder, Germany). "Post harvest" soil samples were also collected at this stage.

All soil samples from the three sampling dates were spread on plastic trays (~10 mm layer) and dried in a forced-air oven (WTC-Binder, Germany) at 40 °C, for a 24 h period (ISO, 11464:2006). Later, samples were crushed and sieved (2 mm mesh size).

All samples were stored at 4 ± 2 °C, protected from light, and all analyses were performed at least in duplicate.

2.3. Chemical analysis

2.3.1. Soil samples

2.3.1.1. Physicochemical characteristics. Soil pH (H_2O) was determined in the upper layer of 1:5 (w/v) prepared with deionized water, according to ISO 10390:2005. Specific electric conductivity was performed as described in ISO 11265:1994. Total organic carbon (TOC) content was estimated by the oxidation of organic matter in a mixture of sodium dichromate (0.17 M) solution and sulfuric acid (98%, v/v) at a temperature of 135 °C (Skjemstad & Baldock, 2008).

2.3.1.2. Caffeine. For caffeine analysis in soil, the extracts previously obtained for pH (H_2O) were filtered through a Whatman® filter No. 42 and 10 ml were freeze-dried. The extracts were further recovered with 1 ml of deionized water for HPLC analysis. Chromatographic conditions were previously described in Cruz et al. (2012). The quantification limit was 20 μg 100 g^{-1}, which was defined as the lowest caffeine concentration that resulted in an average signal-to-noise ratio at 276 nm greater than 10.

2.3.2. Lettuce samples

2.3.2.1. Color evaluation. Instrumental color was measured across the surface of fresh whole green lettuce leaves using a Minolta CR-400 colorimeter (Konica Minolta Optics Inc., Japan). The color coordinates were computed in the CIELAB scale in a CIE C/2° illuminant/observer condition and a 2.5 cm port/viewing area. Color results were expressed as tristimulus parameters: lightness (L^*), redness (a^*), yellowness (b^*), hue angle (h_{ab}), chroma (C^*_{ab}), and color index (CI^*), determined as $CI^* = (a^* \times 1000) \div (L^* \times b^*)$ in accordance with Goñi, Agüero, Moreira, Ponce, and Roura (2010). Color evaluation was performed in 15 leaves from each composite sample, with the final results being the average of 75 measurements per treatment.

2.3.2.2. Chlorophylls and carotenoids. Analytes extraction was performed according to the method reported by Lichtenthaler and Buschmann (2001a), with minor adjustments. Duplicate amounts of freeze-dried lettuce samples (20 mg) were extracted with methanol (1 ml), after addition of MgO (50 mg), and the mixture was homogenized (5 min), centrifuged (13,000 rpm, 0 °C, 5 min) and the clear upper layer was transferred to an amber flask. The extractive procedure was repeated twice with methanol (500 μl). The combined extracts were diluted (1:5) with methanol and dehydrated with anhydrous Na_2SO_4. After new centrifugation (13,000 rpm, 0 °C, 2 min), absorbance was recorded at 470, 520, 652.4, 665.2 and 750 nm for chlorophyll *a*, chlorophyll *b* and total carotenoids quantification, as described by Lichtenthaler and Buschmann (2001b).

Pigments preservation was ensured during extraction by working in the absence of light, with low temperature operating conditions and in the presence of an acid neutralizing (agent) MgO, to avoid conversion into pheophytins (Lichtenthaler & Buschmann, 2001b).

2.3.2.3. Total phenolic content. The Folin–Ciocalteau method, which actually gives an estimation of total reducing capacity rather than a true phenolic content, is the most commonly reported method in lettuce antioxidant activity evaluation, being therefore chosen. Based on the guidelines proposed by Pérez-Jiménez et al. (2008), duplicate amounts of freeze-dried sample (500 mg) were macerated with 20 ml methanol/water (50:50, v/v, pH = 2), under stirring for 1 h. After centrifugation (5000 rpm, 4 °C, 10 min), the residue was re-extracted with 20 ml acetone/water (70:30, v/v), under stirring for 1 h. After a second centrifugation (5000 rpm, 4 °C, 10 min), the supernatants were combined and filtered (nylon filter, 0.22 μm) for subsequent analysis.

Fig. 1. Example of plants collected at harvest.

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