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Characterization of biochar obtained from olive and hazelnut prunings and comparison with the standards of European Biochar Certificate (E.B.C.)

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Abstract

Concerns about climate change and food productivity have spurred interest in biochar, a form of charred organic material, typically applied in agriculture to improve soil productivity and as mean of carbon sequestration. An innovative approach in agriculture is the use of agro-industrial waste for the production of soil fertilizers for agricultural purposes and as a source of energy. A common agricultural practice is to burn crop residues in the field to produce ashes that can be used as soil fertilizers. This approach is able to supply plants with certain nutrients such as Ca, Na, K, Mg, B, S and Mo. However the lack of N and P in the ashes, together with the occasional presence of heavy metals (Ni, Pb, Cd, Se, Al, etc.), has a negative effect on soil and therefore crop productivity. This work describes the opportunity to create an innovative supply chain from agricultural waste biomass. Olive (*Olea europaea* L.) and hazelnut (*Corylus avellana* L.) pruning residues represent a major component of biomass waste in the area of Viterbo (Italy). In this study, we evaluated the production of biochar from these residues. Furthermore, a physico-chemical characterization of the produced biochar was performed to assess the quality of the two biochars according to the standards of the European Biochar Certificate (EBC). The results of this study indicate the cost-effective production of high-quality biochars from olive and hazelnuts biomass residues.

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1. Introduction

Biochar is a carbon-rich material produced by thermal decomposition of biomass under oxygen-limited conditions. According to the International Biochar Initiative (IBI), biochar is primarily used for soil applications for both agricultural and environmental gains (Gaunt & Lehmann 2008). The International Biochar Institute definition distinguishes biochar from charcoal, which is used as a fuel for heating, as an adsorbent material, or as a reducing agent in metallurgical processes (Sohi, Lopez-capel & Krull 2009). Thermo-chemical processes include (i) slow pyrolysis (conventional carbonization), (ii) fast pyrolysis, (iii) flash carbonization and (iv) gasification. Slow pyrolysis has the advantage that can retain up to 50% of the feedstock C in stable biochar (Steinbeiss, Gleixner, & Antonietti, 2009), which makes it suitable as soil fertilizer. Pyrolysis process and its parameters such as final temperature, heating rate, pressure, and residence time greatly condition the quality of biochar. High-temperature pyrolysis (>550°C) produces biochar with high aromatic content and therefore recalcitrant to decomposition (Blasi, Di Branca, Lombardi, Ciappa, Giacomo& Di Chimica, 2013).Low-temperature processes Biochar from low temperature pyrolysis (<550°) typically have produce biochar with a less-condensed C structure and are expected to have a better contribution to soil fertility. The nature of the biomass feedstock also influences the properties of the produced biochar. The relationship between biochar properties and its potential to enhance agricultural soils is a nascent focus area and the appropriate pyrolysis conditions are still unclear (Lehmann, 2007). A number of recent studies focused on characterization methodologies of biochar, other studies investigated the intrinsic potential of biochar as soil amendments, but further efforts are needed to perform soil tests in order to establish an appropriate formulation of desired biochar properties. One of the characteristics of biochar that makes it attractive as a soil amendment is its porous structure, which is responsible for improved water retention and increased soil surface area (Gaunt & Lehmann, 2008). Moreover, the addition of biochar to soil has been associated with an increase of the nutrient use efficiency, either through nutrients contained in biochar or through physicochemical processes that allow a better utilization of soil-inherent or fertilizer-derived nutrients (Lehmann, 2007). Application of biochar in soil increases its physical and chemical qualities, resulting in greater productivity of the agro-ecosystem. Biochar, due to its biological and chemical stability, can also act as C sink. The recalcitrance of biochar to microbial degradation enables the long-term sequestration of C in soil(Brewer, Schmidt-Roht, Satrio & Brown, 2009).

2. Materials and methods

2.1. Biomass for olive and hazelnut's pruning

In the area of Viterbo, pruning residues from olive and hazelnut are rarely utilized as a source of energy in burning stoves or boilers, they are instead burned in situ, therefore reducing the formation of soil organic carbon. During summer, besides pruning residues, suckers are removed before the harvest, representing another significant loss of biomass. Recent studies (Boubaker, De Franchi, Colantoni, Monarca, Cecchini, Longo, Allegrini, Di Giacinto, Biondi & Menghini, 2014; Speranza, Bucini & Paparatti,2009)have investigated the possibility of enhancing olive and hazelnut residues waste management a mean to produce soil fertilizers and energy, therefore reducing the environmental impact of such residual organic waste. Biomass from pruning crop operations (Fig.1)(Abenavoli& Marcianò, 2013; Abenavoli &Proto, 2015; Proto & Zimbalatti, 2015) represent an attractive resource that could be exploited for (i) fuel production (combustion and/or gasification) and (ii) pyrolysis to produce biochar that can be used as soil fertilizer.

Fig. 1. Pruning residues from (a) olive and (b) hazelnut after crops operations. (c) bio-shredding.

A pellettization procedure was developed and applied on bio-shredding obtained from olive and hazelnut residues (Fig.1c). Pruning residues were collected on site and immediately transferred in the laboratory for sifting and exsiccation (Fig.2 a, b, c) until a water content of 15% was achieved. Final water content as low as 15% is necessary for further refining of the product and pellet production.

Fig. 2. Schematic of the pellettization process showing (a) pellet mill,(b) Olive and Hazelntu's pellet and (c) packaging.

2.2. Pyrolysis process

Pyrolysis is carried out under complete or partial exclusion of oxygen and relies on capturing the off-gases from thermal decomposition of the organic materials (Moneti, Delfanti, Marucci, Bedini, Gambella, Proto & Gallucci, 2015).The biochar's physical and chemical characteristics are determined by feedstock type and pyrolysis temperature. For example, higher salt and ash contents are expected in wheat straw than in wood derived biochar, and C content and N content are greater in pine chips than in poultry litter-derived biochar. A higher pyrolysis temperature results in lower biochar recovery, greater surface area, elevated pH, higher ash content, minimal total surface charge (Lehmann, 2007; Sánchez, Lindao, Margaleff, Martínez & Morán, 2009), and lower cation exchange capacity (Emer, Grigolato, Lubello & Cavalli, 2011). Removal of volatile compounds at higher pyrolysis temperatures also cause biochar to have higher C content and lower hydrogen (H) and O content (Monarca, Cecchini, Colantoni & Marucci, 2011; Boubaker, De Franchi, Colantoni, Monarca, Cecchini, Longo, Allegrini, Di Giacinto, Biondi& Menghini, 2014).Pyrolysis of agro-forestry residues is typically carried out with temperatures between 400 and 800 °C. With these conditions, the feedstock is converted to liquid products (so-called tar or pyrolysis oil) and/or gas (syngas), which can be used as fuels or raw materials for subsequent chemical transformation. The residual solid carbonaceous material obtained (biochar) could be further refined to products such as activated carbon. The carbonization system Elsa Research (Blucomb Ltd) was used to produce the biochar from olive and hazelnut pellets; biomass conversion was achieved by pyrolytic micro-gasification (Fig 3).

Elsa Research works with natural ventilation and does not require to be powered by electricity or batteries. A chimney is typically used to increase the air draft for fuels that have difficulty to ignite.

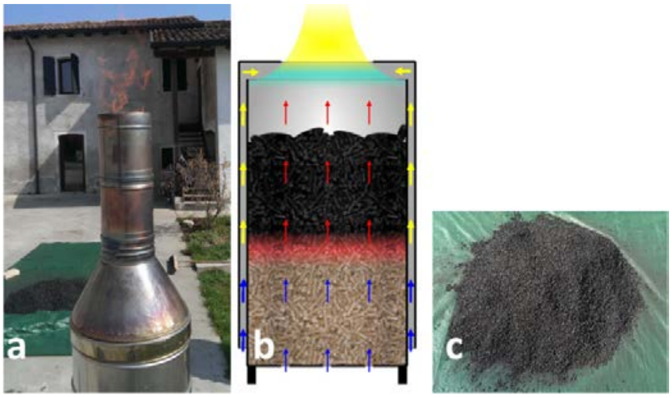

Fig. 3. Biochar production from pellets showing (a) the Elsa Research carbonisation system, (b) a schematic representation of the conversion process and (c) final product (biochar).

3. Results

3.1. From biomass to biochar: conversion rates analyses

Autothermic conversion of biomass was carried out under natural ventilation. Quantitative analyses of pyrolysis biomass and produced biochar, as well as the conversion rates are reported for 10 and 4 sessions of pyrolysis, respectively for olive and hazelnut pellets (Table 1).

Table 1. Conversion rates of biomass obtained from each pyrolysis session.

Olive			
Session	Biomass [kg]	Biochar [kg]	Conversion rate
1	38,35	8,11	0,209
2	39,07	8,21	0,210
3	38,88	8,19	0,211
4	38,96	8,16	0,209
5	34,09	7,10	0,208
6	39,02	8,23	0,211
7	38,89	8,19	0,211
8	38,93	8,19	0,210
9	38,97	8,20	0,210
10	38,81	8,13	0,209
Total	384,47	80,71	Mean 0,210
		Standard deviation	0,00088

Hazelnut			
Session	Biomass [kg]	Biochar [kg]	Conversion rate
1	37,69	8,11	0,215
2	36,25	7,96	0,220
3	37,03	8,09	0,218
4	37,11	8,09	0,218
Total	148,08	32,25	Mean 0,218
		Standard deviation	0,00188

Further analyses were carried out to investigate the calorific power of the two biochar produced. The calorific values calculated in this work were compared with those provided by the producers in order to make energy considerations on the process(Proto, Zimbalatti, Abenavoli, Bernardi & Benalia, 2014). The results obtained in this study are consistent with other pyrolysis processes. Such processes lead to volatilisation of a fraction of biomass with a calorific value ranging between 75 and 85% of the starting biomass.

Table 2. Analysis of the calorific power of pyrolysis reaction for the two biochar produced in this study.

Olive wood	Units	Pellet	Biochar
Higher calorific value	MJ/kg	19,47	31,71
Lower calorific value	MJ/kg	16,17	30,48
Calorific value from pyrolysis	MJ/kg	12,37	
Percentage of calorific value from pyrolysis	%	0,76	
Hazelnut wood	**Units**	**Pellet**	**Biochar**
Higher calorific value	MJ/kg	19,02	26,62
Lower calorific value	MJ/kg	16,71	25,66
Calorific value from pyrolysis	MJ/kg	14,21	
Percentage of calorific value from pyrolysis	%	0,85	

3.2. Elemental analysis

Biochars produced by Blucomb Ltd were analyzed by Eurofins laboratories, accredited for the certification of the European Biochar Certificate (EBC). The EBC has been developed by international biochar scientists to become the voluntary European industrial standard. The EBC ensures a sustainable biochar production and low hazard use in agronomic systems. Biochar produced in accordance with the standards of the EBC fulfils all the requirements of sustainable production and environmental impact by certifying (i) sustainable provision and production of biomass feedstock; (ii) energy efficient, low emission pyrolysis technique; (iii) low contaminant level in biochar and (iv) low hazard use and application of biochar. These standards are in compliance with current environmental European regulations

Table 3. Elemental analyses from EBC (Method DIN 51732).

Elements	Units	Hazelnut biochar	Olive biochar	EBC Biochar base	European Biochar Certificate (standard) Biochar Premium
H (hydrogen)	% w/w	1,21	1,58	-	-
C (Carbon, total)	% w/w	78,1	90,1	>50	>50
N(nitrogen, total)	% w/w	0,64	0,42	-	-
O (oxygen)	% w/w	1,2	1,7	-	-
Carbonate as CO2	% w/w	2,62	1,17	-	-
Carbonate organic		75,5	89,8		
H/C ratio (molar)		0,18	0,21	<0,6	<0,6
O/C rate (molar)		0,012	0,014	<0,4	<0,4
Sulfur total	% w/w	0,07	<0,03		

Table 4. Determination from microwave digestion (method: DIN 22022-1).

Elements	Units	Methods	Hazelnut biochar	Olive biochar	EBC Biochar base	EBC Biochar Premium
P (phosphours)	mg/kg	ISO 11885	590	330	-	-
Mg (magneslum)	mg/kg	ISO 11885	2.900	1.400	-	-
Ca (calcium)	mg/kg	ISO 11885	38.000	11.000	-	-
K (potassium)	mg/kg	ISO 11885	5.500	3.500	-	-

Na (sodium)	mg/kg	ISO 11885	2.100	260	-	-
Fe (iron)	mg/kg	ISO 11885	6.500	1.500	-	-
Si (silicon)	mg/kg	ISO 11885	25.000	9.700	-	-
S (sulfur)	mg/kg	ISO 11885	910	200	-	-
Pb (lead)	mg/kg	ISO 17294-2	66	20	<150	<120
Cd (cadmium)	mg/kg	ISO 17294-2	<0,2	<0,2	<1,5	<1
Cu (copper)	mg/kg	ISO 17294-2	100	6	<100	<100
Ni (nikel)	mg/kg	ISO 17294-2	9	8	<50	<30
Hg (mercury)	mg/kg	DIN EN 1483	<0,07	<0,07	<1	<1
Zn (Zinc)	mg/kg	ISO 17294-2	340	84	<400	<400
Cr (chromium total)	mg/kg	ISO 17294-2	22	15	<90	<80
B (boron)	mg/kg	ISO 17294-2	32	10	-	-
Mn (manganese)	mg/kg	ISO 17294-2	350	380	-	-

Table 5. PAHs determination from toluene extract. *(GW 1 = quality level basic related dry bases, GW 2 = quality level premium related dry bases).

Elements	Units	Methods	Limits		Hazelnut biochar	Olive biochar
			GW 1*	GW 2*		
Naphtalene	mg/kg	DIN EN 15527	-	-	0,9	1,1
Acenaphthylene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Acenaphthene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Fluorene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Phenanthrene	mg/kg	DIN EN 15527	-	-	0,3	0,3
Anthracene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Fluoranthene	mg/kg	DIN EN 15527	-	-	0,1	0,1
Pyrene	mg/kg	DIN EN 15527	-	-	0,1	0,1
Benz(a)anthracene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Chrysene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Benzo(b)fluoranthene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Benzo(k)fluoranthene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Benzo(a)pyrene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Indeno(1,2,3-cd)pyrene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Dibenz(a,h)anthracene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
Benzo(g,h,i)perylene	mg/kg	DIN EN 15527	-	-	<0,1	<0,1
SUM PAHs (EPA)	mg/kg	calculated	<12	<4	1,20	1,60

Table 6. pH, electrical conductivity (EC) and density determination.

Elements	Units	Hazelnut biochar	Olive biochar
pH values (Cacl2)	-	9,9	8,4
Electrical conducivity	µS/cm	332	217
Salt content	g/kg	0,655	1,18
Salt content cal. with bulkdensity	g/l	0,287	0,527

4. Conclusions

The results of elemental analyses of the two biochar analyzed in this study and reported in table 3 showed the both biochar are characterized by values well below the limits established by the E.B.C., in particular the olive and hazelnut biochar have high values of C and low H/C and O/C ratios. Low level of H/C ratio indicates that the produced biochar are also recalcitrant to microbial degradation. These results indicate that our production process yield high quality biochar with a level of carbonization that makes it suitable for C sequestration, as confirmed by the H/C ratios. These types of biochar have a lower total carbon (TC) content and cohesion than those obtained from wood-pruning biomass (table 4). The low C content, together with elevated concentrations of nutrients makes biochar from herbaceous material more readily biodegraded by microorganisms (Colantoni, Allegrini, Boubaker, Longo, Di Giacinto & Biondi, 2013; Cavalli & Grigolato, 2010). Therefore, the lower TC content, together high concentrations nutrients cause a more rapid degradation by microorganisms(Sohi, Lopez-capel & Krull, 2009; Blasi, Di Branca, Lombardi, Ciappa, Giacomo & Di Chimica, 2013). The concentration of phosphorus (P) and potassium (K) in the biochar is related to the initial content in the feedstock. Content of P and K are typically between 2.7 - 480 g kg^{-1} and 10 to 58 g kg^{-1} respectively.EBC biochar base and premium report the limits required by the Protocol of certification EBC. The total ash content ranged between 6.2 and 18.8% (w/w) for biochar from pellets of olive and hazelnut wood. The nutrients content is much greater in hazelnut biochar than olive, this was evident especially for Mg, Ca, Fe, S, Cu, and Zn. Biochar from hazelnut pellet, could bring a greater contribution of nutrients in soil and therefore be less resistant to microbial decomposition. Heavy metal content in both biochar was well below the EBC limits. Only Cu in the hazelnut biochar was close to the maximum value established by the EBC.PAHs are ubiquitous in the environment, being by-products of the incomplete combustion of organic material. The chemical structure of PAHs makes them highly resistant to biodegradation and oxidation. It is therefore critical to ensure PAHs concentration below the limits established by the EBC. The 16 priority US EPA PAHs are typically used to assess the total PAHs content; the limits established by the EBC are of <12 and <4 mg/kg for biochar standard and premium, respectively. The PAHs composition of the two biochar analyzed in this study (Table 5), shows that both biochar are well below the EBC limits, with values ranging from <0.1 to 1.1 mg/kg.Total PAHs content of the two biochar are 1.2 and 1.6 mg/kg for olive and hazelnut respectively. Therefore, both biochar can be considered suitable for soil applications, since well below the EBC threshold limit of 4 mg / kg for biochar premium. The two biochar have a pH of 8, 4 and 9.9 for olive and hazelnut, respectively (table 6). The EBC indicates a maximum limit of 10; therefore biochar produced from these types of wood residues is slightly below the limit established by the certification. The EC is of particular importance when adding biochar to soils with high EC and salinity. The two biochar had a EC of 217and 332mS/cm respectively for olive and hazelnut (Supporting Information 1 and 2). Both values are very low and do not represent a real risk for the addition to soil even under conditions of high EC. In general, biochar has a lower density than soil, with an average of 0.4 g cm^{-3} compared to a soil of medium texture, with average of 1.3 g cm^{-3}. When adding biochar to soils little ventilation, this property can help to reduce the density by mitigating issues related to compaction of soil. The olive and hazelnut biochar produced in this study have a density of 0.45 and 0.44 g cm^{-3} respectively. Finally, the two biochar analyzed in this study show excellent physic-chemical properties, which makes them suitable for agronomic applications.

Both biochar could be certified as Biochar Premium according to the regulations of the E.B.C.; this allows a potential commercialization of the biochar, with higher prices than Biochar Base, typically less expensive, but with a higher PAHs content. The benefits of using Biochar Premium as soil fertiliser include an improved productivity, increased water holding capacity of the soil and a better retention of nutrients and agrochemicals in soils, also allow a indirect integrated pest management of several pests (Speranza, Bucini & Paparatti, 2009; Pucci, Iannotta, Duro, Jaupi, Thomaj, Speranza & Paparatti, 2013), all of which should offset initial investment and provide added profits per application.

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