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ORIGINAL ARTICLE

Effect of garden and park waste hydrochar and biochar in soil application: a comparative study

Eneko Suarez[1] · Montserrat Tobajas[1] · Angel F. Mohedano[1] · Maria Reguera[2] · Elvira Esteban[3] · Angeles de la Rubia[1]

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Abstract
Thethermochemical treatments of biowaste yield alternative renewable carbon-rich materials, while achieving waste valorization. These technologies allow to reduce amount of biowaste and enhance its life span avoiding the landfill disposal. The agronomic application as a soil amendment strategy using fresh hydrochar (obtained via the hydrothermal treatment of garden and park waste (GPW) at 180 °C for 1 h), post-treated hydrochar (washed, aged, and thermally treated), and biochar (obtained via the pyrolysis of GPW at 900 °C for 90 min) has been studied and compared in order to stablish the best approach for its valorization. We evaluated the effects of mixing fresh hydrochar (1–5% on dry weight) with different peat-based growth substrates on the seed germination index as well as the fresh and dry weights of *Arabidopsis thaliana*, *Chenopodium quinoa*, and *Solanum lycopersicum* (tomato). We also performed a germination assay with marginal agricultural soil mixed with fresh and post-treated chars as well as biochar using the same doses. All carbonaceous materials complied with the European legal framework being categorized as a class A amendment and present a favorable chemical composition for their agronomic use, carbon sources with a low heavy-metal content, and a high mineral and organic matter content. Application of post-treated hydrochar to the agricultural marginal soil improved the germination index of tomato seeds (by 10–20%) at low dosages (< 3% on dry weight) when washed and thermally treated hydrochar was used. However, fresh hydrochar negatively affected seed germination and plant growth when applied to marginal soil and peat-based growth media, particularly sandy substrates. Washing improved the germination index (by approximately 18%), reduced 90% of the total volatile fatty acid content, and effectively removed furans, amines, amides, pyridines, pyrazines, benzoic compounds, and organic acids that can affect seed germination and plant growth. Because the use of hydrochar in soils for agricultural purposes requires post-treatment to alleviate germination and plant growth inhibition, washing is the most suitable option considering the energy and technological requirements.

Keywords Biochar · Germination index · Plant growth · Post-treated hydrochar · Soil amendment

1 Introduction

The depletion of fossil resources and intensifying environmental issues related to biowaste management have become a major global concern according to the bioeconomy strategy of the European Commission and European Union members [1]. In this framework, the sustainable exploitation of natural resources, such as peatlands (one of the most used growing media in horticulture), has gained special relevance to ensure a valuable carbon store and preserve natural habitat. Therefore, biodiversity maintenance is related to the need to use alternative renewable carbon-rich materials [2]. Thermal treatment, such as pyrolysis, is a well-implemented method for biochar production from biowaste. The solid product obtained that is rich in carbon has been used for diverse applications, such as biofuel, contaminant remediation, and soil amending [3]. However, with the aim of producing value-added materials by waste valorization, a circular economic development must be constructed for proven and

✉ Angeles de la Rubia
angeles.delarubia@uam.es

[1] Chemical Engineering Department, Universidad Autonoma de Madrid, 28049 Madrid, Spain

[2] Department of Biology, Universidad Autonoma de Madrid, 28049 Madrid, Spain

[3] Department of Agricultural Chemistry and Food Sciences, Universidad Autonoma de Madrid, 28049 Madrid, Spain

emerging technologies. Hydrothermal treatment (HTT) is a promising alternative to produce a solid material, referred to as hydrochar, which is generated at lower temperatures (180–250 °C) than those used in pyrolysis [4]. Biowastes, such as sewage sludge, food waste, livestock manure, and agricultural waste are common feedstocks for biochar and hydrochar production [5–8]. Pruning residues from fruit orchards, olive groves, and urban green waste are the main agricultural biomass residues in Mediterranean countries [9]. Typically, urban green waste includes biodegradable garden and park wastes (GPW), which is a highly available resource in municipalities [10]. It is currently being processed through composting, resulting in a low-value product (i.e., compost), or through anaerobic digestion, leading to low biogas production due to its structural complexity [11]. Therefore, a large volume of GPW is still landfilled or incinerated, posing a great challenge to the management of urban environments [12].

Although hydrochar functionality is similar to biochar, its application must be evaluated and compared owing to its different physicochemical properties related to the operating conditions involved in its production. Hydrochar exhibits higher O/C and H/C ratios, thus lower aromaticity resulting in poorer stability when added to soil [4]. Hydrothermal treatment of biowaste performed in the temperature range of 150–200 °C results in filamentous structures with porous surfaces [13] and a high proportion of oxygen-containing functional groups that can enhance the soil water holding capacity (WHC), nutrient retention capacity, and cation exchange capacity (CEC) [14, 15]. Pyrolysis at temperatures between 500 and 900 °C yields low amounts of O-containing functional groups in biochar compared to hydrochar which results in a higher stability toward microbial and chemical degradation [16] and triggers the difference in degradation rate between both chars.

Poor physical properties such as low porosity and high bulk density are typically found in marginal agricultural soils. However, soil amended with biochar and hydrochar may effectively increase its porosity [17, 18] and decrease bulk density [19, 20]. In addition, the application of hydrochar from urban biowaste on soil may raise crop productivity and nutrient availability [21]. These benefits have been described in soils with a wide range of textures, such as clay, loamy, and sandy soils [17, 19, 22]. However, direct application of biochar and hydrochar can also have an adverse impact on seed germination and plant growth since, depending on the source and operating conditions, hazardous chemicals may be present on their surfaces. These substances include furfural, polycyclic aromatic hydrocarbons (PAHs), organic acids and phenols, polychlorinated dibenzodioxins, and dibenzofurans, which potentially place plant and soil health at risk [20, 23, 24]. Kalderis et al. [25] and Bargmann et al. [26] observed a decrease in plant growth of barley and maize when hydrochar from a wide variety of biomass such as sewage sludge, wood chips, spent brewer's grains, and orange peel was applied in soil. However, the responses of the soil chemical and biological properties to the application of hydrochar are still unclear and further research should be developed to determine the effects on carbon storage, nutrient bioavailability, and specially toxicity of contaminants.

Thus, strategies involving cost-effective physical, chemical, or biological post-treatment to eliminate organic phytotoxic substances are gaining attention among scientific community. A decrease on soluble and volatile biodegradable compounds and phytotoxic effects on both germination and growth have been reported after hydrochar washing [26]; also, aging post-treatment produced microbial degradation of some hydrochar components and their mineralization [4, 27]. Hitzl et al. [28] also observed a reduction of phytotoxic effect using hydrochar thermally treated at 200–600 °C. Moreover, addition of compost to the hydrochar diminished the phytotoxicity on cress seeds and Chinese cabbage germination [2, 29]. Nevertheless, further research is needed to understand the relationship between the characteristics of hydrochar and the responses of different crops linked to soil type and dosage [30, 31], time of stabilization [2], and the effect of post-treatments prior to soil application.

The aim of this work was to evaluate the potential application of fresh hydrochar, post-treated hydrochar, and biochar obtained from GPW as a component of horticultural peat-based growing media or as a soil amending agent. For this purpose, the effects of fresh hydrochar (2–10% on dry weight, d.w.) on the germination index (GI) and plant growth (i.e., fresh and dry weights of *Arabidopsis thaliana*, *Chenopodium quinoa*, and *Solanum lycopersicum* (tomato)) were determined using peat- and sand-based growing media. The evaluation of its application as a soil amending agent was conducted by analyzing the potential phytotoxic effect of adding 1–5% d.w. of fresh, washed, aged, and thermally treated hydrochar, as well as biochar, to a marginal agricultural soil on *Solanum lycopersicum* seed germination.

2 Material and methods

2.1 Hydrothermal treatment, pyrolysis, and post-treatments

GPW was collected from municipal parks and gardens of *Comunidad de Madrid* (Spain) and was composed mainly by leaves and tree branches that were ground and sieved to reduce and homogenize the particle size (< 3 mm). Subsequently, the GPW was dried at 100 °C for 48 h in a convection oven and stored in airtight containers until use. Biochar (BC) was produced by pyrolyzing 200 g of raw GPW in a rotatory reactor tube furnace (CARBOLITE

HTR 11/150, England) equipped with a quartz tube (15 cm × 21 cm) at 900 °C for 90 min, using a heating rate of 3 °C/min and N_2 flow rate of 1 mL/min to ensure an oxygen-free atmosphere. Wet GPW (20% GPW/80% deionized water (w/v), 1 kg) was subjected to HTT in a 4-L ZipperClave (USA) 316 stainless steel pressure vessel at 180 °C for 1 h and autogenerated pressure. The working temperature was reached at a 3 °C/min heating rate. The reaction was stopped by cooling with an internal heat exchanger using tap water [7].The obtained slurry was separated into liquid and solid (hydrochar) fractions via filtration (0.45 µm). The obtained char was dried at 105 °C for 48 h in a convection oven and labelled as fresh hydrochar (FHC).

Fresh hydrochar was subjected to different post-treatments: (i) Aging: Bulk samples of fresh hydrochar were placed on trays with a maximum height of 4 cm for 4 months at room temperature (20–25 °C), and periodic turning was performed to allow their maturation through air exchange to obtain aged hydrochar labelled as AHC, following the method described by Puccini et al. [27]. (ii) Washing: Fresh hydrochar was washed with deionized water in a 1:10 (w:v) ratio. The resulting suspensions were shaken at 120 rpm for 1 h, centrifuged, and filtered. According to Al-Wabel et al. [32], this procedure was repeated three times to obtain washed hydrochar, referred to as WaHC. Other washing procedures can be found in the literature [27, 33], varying length or number of cycles. (iii) Thermal post-treatment: The fresh hydrochar was thermally treated at 650 °C for 90 min in a rotatory tube furnace as previously described (pyrolysis method) to produce thermally treated hydrochar labelled as THC following the procedure of Hitzl et al. [28]. Temperature of 650 °C, slightly higher than used by Bahcivanji et al. [34], was selected to assure the removal of the volatile compounds responsible of the phytotoxicity.

All the carbonaceous materials were dried at 105 °C for 24 h, ground to a particle size of 3 mm, and stored in ziplock bags until further characterization. Characterization of the feedstock, all hydrochar, and biochar (moisture, ash, volatile matter (VM), and fixed carbon (FC)) was performed via thermogravimetric analysis according to ASTM-D7582 [35] in a Discovery SDT 650 thermogravimetric analyzer. The elemental compositions (C, H, N, and S) were determined using a CHNS analyzer (LECO CHNS-932) and mineral elements were quantified using inductively coupled plasma-atomic emission spectroscopy (ICP-AES) on an Elan 6000 Sciex instrument (Perkin Elmer) following the standard manufacturer's procedure. The pH and electrical conductivity (EC) were measured as described by Manzano et al. [36]. Moisture and organic matter (OM) were determined following standardized methods [37, 38]. The mass yield of the fresh hydrochar/biochar was calculated using Eq. 1. The char yield ($Y_{FHC,\ BC,\ THC}$) was defined as the ratio of recovered solid fraction ($W_{FHC,\ BC,\ THC}$) to feedstock (W_{GPW}) on a dry weight basis.

$$Y_{FHC,BC,THC} = \frac{W_{FHC,BC,THC}}{W_{GPW}} \times 100 \quad (1)$$

Individual VFAs (C2–C7, including iso-forms) in the effluents of the washing procedure were identified using gas chromatography (GC–FID) (Varian 430-GC instrument) as described by De la Rubia et al. [39]. Gas chromatography-ion-trap mass spectrometry (GC–MS; CP-3800/Saturn 2200) was used to identify the chemical species in process water from HTT [7] as well as in the effluents from hydrochar washing [40]. The compounds were identified using the 2008 National Institute of Standards and Technology library.

2.2 Peat-based growing media

Fresh hydrochar was mixed with several peat-based substrates to obtain 4 mixtures with different compositions (Table 1). The components of the substrates are peat TS3 fine, which is a moderately decomposed white peat derived from putrefaction and incomplete carbonization of vegetation in acidic wetlands and marshes; vermiculite N° 2, which is a clay mineral that contributes to soil aeration and water retention; and river sand, which is an inert fraction (mm) of soil minerals that improves substrate drainage.

Substrate S1 was fully peat-based, whereas S2 was prepared by mixing peat and sand (4:1) (d.w./d.w.). Substrate S3 was based on a peat and vermiculite mixture (3:1), whereas in S4, the three components described were mixed in the proportions indicated in Table 1. A wide range of fresh hydrochar dosages (2–10% d.w.) were tested in each substrate using the bare growth media as a control.

The substrates were characterized following the UNE standard methods for soil amendments and growing media, including pH, EC, CEC, OM, and moisture [37, 38, 41–43]. The total Kjeldahl nitrogen (TKN) was determined using standard analytical methods [44], oxidizable organic matter was determined using the Walkley–Black method [45], and total P content was quantified using ICP–AES on an Elan

Table 1 Substrate composition including FHC concentration and sterilization conditions

Substrate name	Composition (% d.w.)			Concentration of FHC (% d.w.)
	Peat	Vermiculite	River sand	
S1	100	-	-	Control, 2.5, 5, and 10
S2	80	-	20	
S3	75	25	-	Control, 2, 2.5, 5, and 10
S4	60	20	20	

6000 Sciex instrument (Perkin Elmer), following the manufacturer's procedure.

Three different plant species were used *Arabidopsis thaliana* ecotype Columbia (Col 0), *Chenopodium quinoa* (commonly known as quinoa) variety F16 provided by ALGOSUR S.L. (Seville, Spain), and *Solanum lycopersicum* (tomato) variety Marmande RAF purchased from Semillas Batlle S.A. (Spain).

Arabidopsis thaliana tests were conducted in 0.5-L square thermoformed pots (Projar, Spain), while *Chenopodium quinoa* and *Solanum lycopersicum* were grown in 1-L thermoformed pots (Projar, Spain). Thirty to forty *Arabidopsis* seeds, eight quinoa seeds, and ten tomato seeds were sown per pot. Arabidopsis was evaluated using the four different substrates, S1–S4, adding FHC at different proportions of 2.5, 5, and 10% d.w. on S1 and S2, and 2, 2.5, and 5% d.w. on S3 and S4. Quinoa and tomatoes were evaluated only with S3 and S4 substrates using 2.5, 5, and 10% of FHC. All assays were performed in triplicate. The substrate-char mixtures were autoclaved at 120 °C for 40 min (Presoclave 75, J.P. Selecta, Spain) before sowing. Vernalization occurred at 4 °C for 72 h in the dark prior to each experiment to ensure seed stratification, and the pots were placed on trays and covered with plastic film during germination to avoid total nutrient depletion via lixiviation and water evaporation. Thereafter, the pots were transferred to a controlled plant growth chamber set at 24 °C/18 °C with a 12 h/12 h light/dark photoperiod (with a light intensity of 120 µmol/m^2/s). The plastic film was removed 5 days after sowing, and irrigation with distilled water was performed every 2–3 days when the trays dried up.

The germination index (GI) was calculated using Eq. 2 and the leaf area was determined by analyzing leaf images using the ImageJ software (https://imagej.nih.gov). Both parameters were recorded every 2 days. To evaluate plant biomass at the end of the experiment, tomato and quinoa plants were cut at stem level 21 and 43 days after sowing (DAS), respectively, and the fresh weight (FW) was determined. Thereafter, the tissue was dried at 65 °C for 72 h and weighed again to determine the dry weight (DW).

$$Germination\ index\ (\%) = \frac{grown\ cotyledons}{total\ seeds\ in\ pots} \times 100 \qquad (2)$$

2.3 Soil germination media

A marginal agricultural sandy loam soil (Burgos, Spain), with 1% of organic matter, a low clay concentration (8%), and a pH of 8.4, was mixed with 1, 3, and 5% (on a dry weight basis) fresh hydrochar, post-treated hydrochar (obtained after washing, aging, and thermal treatment, as mentioned above), and biochar. Prior to mixing, the pH, EC, moisture, and OM of the soil and char were determined, as previously mentioned [37, 38]. For the germination assay, Petri dishes (9 cm diameter) were filled with 45 g of each mixture. Bare soil was used as a control. They were watered until a 75% WHC was reached and allowed to stabilize for 1 week in darkness at 28 °C. Five replicates of each condition were prepared; one was used for pH and EC determination after stabilization using the methodology previously mentioned [41, 42] and the other four were used for seed germination. Tomato (Marmande RAF) seeds were surface-sterilized using a sodium hypochlorite standard washing procedure (1:10 bleach/water (v/v)) and sown (10 seeds per plate). Plates were placed in the dark at 28 °C for 3 days and transferred to a growth chamber set at 26 °C/20 °C with a 13 h/11 h light/dark photoperiod. The GI was determined after 5 days.

2.4 Statistical analysis

The effects of hydrochar concentration, type of substrate, and their reciprocal interactions on germination, fresh and dry weight, and leaf area were analyzed using two-way completely randomized analysis of variance on the FHC-substrate assays. Tukey's test at $p \leq 0.05$ was carried out to establish significant differences between means (***: $p < 0.001$, the most significant difference; **: $p < 0.05$, significant difference; *: $p = 0.05$, slight difference). The Minitab statistical program (version 19) was used.

3 Results and discussion

3.1 Proximal and elemental analysis of feedstock, hydrochar, and biochar

Remarkable differences in terms of solid fraction yield of GPW were found for hydrothermal treatment (87%) and pyrolysis (30%). The higher solid yield achieved in FHC is related to the operating temperature due to the filamentous structural complexity of the feedstock and the stability of lignin at higher temperatures. The yield obtained in THC agrees with Fu et al. [46] at similar operating temperatures resulting in a mass loss close to 50% due to polymerization/condensation reactions and the volatilization of light compounds occurring during pyrolysis. Moreover, the removal of soluble organic matter by washing procedure leads to an extra 10% of mass loss while aging post-treatment does not show a remarkable effect on the yield.

Table 2 lists the main characteristics of the raw material and chars. Fresh, aged, and washed hydrochars presented a slightly acidic pH, in accordance with findings by Al-Wabel et al. [32], with the potential to be used as an amending agent for moderately basic soils such as the one used in this study

Table 2 Main characteristics of feedstock and carbonaceous materials

	GPW	FHC	WaHC	AHC	THC	Biochar
Yield (%)	-	87 ± 0.1	-	-	45 ± 0.1	30 ± 0.1
pH	6.8 ± 0.1	5.5 ± 0.1	5.2 ± 0.1	5.5 ± 0.1	11.2 ± 0.1	12.5 ± 0.1
EC (mS/m)	1872 ± 6.2	385 ± 6.4	378 ± 7.4	90 ± 3.1	1152 ± 9.6	993 ± 8.4
OM (%)	96 ± 0.5	97 ± 0.9	94.6 ± 0.1	97 ± 0.9	85.4 ± 0.4	84.5 ± 0.6
Moisture (%)	4.0 ± 0.1	3.8 ± 0.1	3.2 ± 0.1	4.3 ± 0.1	3.0 ± 0.1	3.9 ± 0.1
VM (% d.w.)	76.5 ± 0.1	67.1 ± 0.1	68.2 ± 0.1	73.3 ± 0.1	22.5 ± 0.1	6.3 ± 0.1
Ash (% d.w.)	5.1 ± 0.1	3.3 ± 0.1	12.5 ± 0.1	6.8 ± 0.1	21.8 ± 0.1	22.6 ± 0.1
FC (% d.w.)	18.4 ± 0.1	29.6 ± 0.1	19.4 ± 0.1	19.9 ± 0.1	55.7 ± 0.1	71.1 ± 0.1
C (% d.w.)	46.9 ± 0.1	49.8 ± 0.1	50.4 ± 0.1	49.2 ± 0.1	69.3 ± 0.1	70.5 ± 0.1
H (% d.w.)	6.1 ± 0.1	5.3 ± 0.1	5.5 ± 0.1	5.4 ± 0.1	1.8 ± 0.1	0.8 ± 0.1
N (% d.w.)	0.9 ± 0.1	1.3 ± 0.1	1.5 ± 0.1	1.3 ± 0.1	1.7 ± 0.1	1.7 ± 0.1
S (% d.w.)	0.4 ± 0.1	0.2 ± 0.1	0.1 ± 0.1	0.1 ± 0.1	0.0 ± 0.1	0.1 ± 0.1
O (% d.w.)*	40.6 ± 0.1	40.1 ± 0.1	30.0 ± 0.1	37.2 ± 0.1	5.4 ± 0.1	4.3 ± 0.1
C/N	60.8	44.7	39.2	44.2	47.6	48.4
H/C	1.6	1.3	1.3	1.3	0.1	0.0
O/C	0.6	0.6	0.4	0.6	0.3	0.1
(O + N)/C	0.7	0.6	0.5	0.7	0.1	0.1
N/P/K	0.9/0.1/4.9	1.3/0.1/3.4	1.5/0.2/0.1	1.3/0.1/0.5	1.7/0.5/0.1	1.7/0.5/2.0

*O = 100 − C − H − N − S–ash (% d.w.)

(pH 8.4), contributing to improve soil pH and thus increasing nutrient availability [47]. The higher temperatures used to obtain THC and biochar induce a strongly basic pH, in agreement with Taskin et al. [9]. This increase in pH could be due to the higher ash content of alkali and alkaline earth metals found in chars produced at higher temperatures. The differences in pH for BC and THC respect to hydrochar can be related not only to the surface chemistry and the ash content but also to H/C and O/C ratios [21]. Biochar and THC show the highest EC values among the processed wastes. AHC shows values close to that of the soil (62.3 mS/m) due to the mineralization process while WaHC maintains a value similar than the obtained for FHC (385 mS/m), indicating that washing has a slight lowering effect on CE, in a similar way to the small effect on pH. Regarding OM, values are related to operating temperatures; a decrease is observed when the pyrolytic temperature varies (biochar and THC). As mentioned above, the soil presents a low OM content (0.6–1%) for its texture [48]. All materials show similar low moisture contents (< 4%). The ash content of FHC is lower than that of the feedstock, as also reported by Melo et al. [30] for sewage sludge hydrochar because inorganic components would be solubilized reducing the ash content of hydrochars [49]. Moreover, all the post-treatments that have been tested increases the ash content compared to FHC. The higher ash content in the pyrolyzed materials (THC and BC) could be related to the formation and condensation of mineral constituents and elements under pyrolytic conditions [50]. The increase in ash content reported in WaHC is attributed to variations in labile and fixed carbon content due to the OM removal (≈10% mass loss) during washing, whereas inorganic compounds were not solubilized and remained in the char. In the aging post-treatment, only oxidable organic compounds reacted to the air oxidation during the mineralization process, causing a slight increase in the ash content.

The hydrothermal treatment reduces the VM content of hydrochar by the volatilization of light compounds [7]. With increasing temperature (THC and BC), the VM content decreases due to further volatilization. All carbonaceous materials show a higher fixed carbon content than raw waste. Thus, the pyrolytic chars (THC and BC) show the highest contents (56 and 71%), which increase as the operational temperature increases, as also reported by Afolabi et al. [51]. This result also agrees with the findings of Yargicoglu et al. [52], who reported a carbon content ranging from 50 to 78% for biochar obtained from pinewood pellets. According to elemental analysis, all carbonaceous materials show higher C and N contents as well as lower H, S, and O contents than the raw GPW. Farru et al. [53] and Cervera-Mata et al. [54] also observed high C content in a wide variety of chars. H and O content decreases as the temperature increases, which could be attributed to the loss of H- and O-containing functional groups during the thermal treatment of wastes [55]; slight differences are observed between washing and aging compared to FHC due to the further removal of O-functional groups of the surface during washing process [32].

The C/N ratio is lower in all carbonaceous materials than in the raw GPW. The BC and THC exhibit the highest ratios. Aging has no effect and shows a similar value to FHC; however, washing lowers the ratio closer to the optimal value (of

21) for microbial activity and proper plant growth [56]. H/C and O/C ratios as well as the polarity index ((O + N)/C) are negatively correlated with the temperature, because aromatization and O-functional groups are reduced as operational temperature increases [57] yielding hydrophobic materials. The lower H/C ratio obtained for chars suggests a non-condensed and less aromatic structure [58] than the raw material with a large amount of carboxylic and hydroxyl groups. Hydrochars result in more aromatic structures compared to BC. Aged hydrochar shows similar ratios to FHC. However, the decrease in O content leads to the decrease of the O/C ratio and makes the WaHC more hydrophobic, as can be observed on the polarity index compared to FHC. Thermal treatment and pyrolytic temperatures enriches the elementary content and lowers even more H/C and O/C ratios as determined Huff et al. [59]; however, higher concentrations of heavy metals are detected (Table 3) in agreement with findings of Taskin et al. and Lang et al. which can have a negative effect on plant growth [9, 60]. Both pyrolytic chars comply the quality indicators for stability in soil that assures slow OM release (H/C < 0.6 and O/C < 0.4) established by Schimmelpfennig and Glaser [61].

The concentration of potentially toxic metals (Table 3), such as Cr or Zn, is under the maximum value allowed for organic amendments in Europe (Cr ≤ 70 mg/kg, and Zn ≤ 200 mg/kg) in raw GPW, FHC, and BC [55, 56], being categorized in Spain as a class A amendment suitable for use on any vegetable crop according to RD 506/2013 [62], even though the Cr values are at the upper limit allowed and should be monitored. They also comply with the guidelines of the European Biochar Certificate and International Biochar Certificate categorized as premium carbonaceous materials [63]. Hydrochar is rich in Ca, K, and P, which are important macronutrients for plant growth, indicating its potential use in soil amendment, especially in degraded soils with poor OM contents [21], except for K and Mg, which decrease slightly. Regarding the mineral content, the high operational temperatures used to obtain THC and biochar lead to a richer composition than that of hydrochar and raw GPW. However, other potentially toxic metals, such as As, Cr, Fe, and Al, are also present in higher proportions. The thermal treatment increases Ca, Al, and Fe content according to Hitzl et al. [28]. WaHC shows a higher relative content of Ca, Cu, Fe, and Mn as these inorganic compounds were not solubilized and remained in the char, while OM removal occurred during the washing process in concordance with the findings of Cervera-Mata et al. [54] and aging increases the Al, Ca, Fe, and Mg content compared to fresh hydrochar due to the mineralization process.

3.2 Characterization of peat-based substrates and soil-char mixtures

Table 4 lists the physicochemical properties of the four peat-based growth substrates. The pH is related to nutrient availability/solubility and affects microbial activity [58]. All substrates show slightly acidic pHs in the adequate range of 5.7–7.0 for most plant growth [64, 65]. EC indicates

Table 3 ICP analysis (mg/kg) of raw and carbonaceous materials

	Raw GPW	FHC	WaHC	AHC	THC	Biochar
Al	123.0	366.7	1466.8	746.7	1867.5	2299.9
As	< 0.01	< 0.01	0.2	< 0.01	1.9	0.5
Ca	10,100	16,700	34,059	24,250	63,238	49,110
Cd	0.0	0.1	0.7	0.4	0.1	0.0
Co	< 0.01	0.1	0.7	< 0.01	0.6	< 0.01
Cr	1.4	3.7	10.5	9.7	14.5	50.8
Cu	2.5	7.0	12.1	7.4	22.5	13.4
Fe	96.1	226.7	888.3	418.8	1227.7	1815.7
K	48,550	34,300	751.2	4900	11,453	20,300
Mg	770.0	650.0	606.5	900.0	2264.2	4100.0
Mn	12.8	12.7	38.9	22.5	77.0	79.0
Mo	< 0.01	< 0.01	1.2	< 0.01	2.0	< 0.01
Na	30.0	50.0	40.6	70.0	276.9	330.0
Ni	0.3	10.9	3.4	2.0	4.2	12.2
Pb	0.4	1.3	6.4	1.8	3.2	1.0
P	932.0	1162.0	2261.7	1440.0	4924.2	5260.0
Si	332.7	463.0	329.4	687.8	512.3	581.0
S	460.0	780.0	1172.0	950.0	878.4	990.0
Zn	20.1	29.4	96.3	51.4	100.3	9.8

*Each data point shows a standard deviation ≤ 0.1

Table 4 Main characteristics of peat-based substrates

	S1	S2	S3	S4
pH	5.19 ± 0.01	5.93 ± 0.01	5.48 ± 0.01	6.53 ± 0.01
EC (mS/m)	40.2 ± 0.1	15.7 ± 0.1	27.8 ± 0.1	31.4 ± 0.1
OM (%)	98 ± 1	15 ± 1	59 ± 1	15 ± 2
Moisture (%)	61 ± 1	22 ± 1	48 ± 1	24 ± 2
CEC ($cmol^+/kg$)	866.0 ± 0.2	298.8 ± 0.2	900.8 ± 0.6	306.9 ± 0.2
TKN (g/kg)	0.37 ± 0.02	0.23 ± 0.03	0.32 ± 0.02	0.25 ± 0.02
P total (g/kg)	0.6 ± 0.1	0.5 ± 0.1	1.5 ± 0.1	1.2 ± 0.1

soil salinity and with all the substrates used classified as non-saline as EC is less than 400 mS/m [66, 67]; therefore, plant growth would not be compromised. EC is the measure of total dissolved salts in a solution. Too low levels indicate low soluble nutrients and are often found in sandy soils with low OM levels compromising plant health and reducing yield. On the other hand, excessive EC obstructs nutrient uptake by increasing the osmotic pressure leading to nutrient leaching and associated environmental hazards [68]. The OM content is also a key attribute in assessing soil health and fertility, generally positively correlating with crop yield [65] and favoring the WHC. Substrates S1 and S3 show higher OM contents owing to the absence of sand in the mixture. Low EC together with a poor OM content as presented for sandy mediums can be related to a potential nutrient deficiency [25]. The higher OM contents in S1 and S3 lead to higher moisture contents (61% and 48%, respectively, when compared with 22–24% in S2 and S4). The presence of sand also has an impact on moisture; S2 and S4 show similar low moisture levels when compared with the rest of the growing media. The combination of peat and vermiculite in S3 provides a similar CEC in both treatments. CEC measures the ability of the soil to retain nutrients; therefore, the high CEC values obtained for S1 and S3 (approximately 900 $cmol^+/kg$) will contribute to nutrient availability promoting plant growth, while S2 and S4, with lower OM contents, exhibit lower values (approximately 300 $cmol^+/kg$), with the only advantage of slightly increasing the substrate pH. Regarding nutrient content, TKN reaches a large N content in the S1 and S3 substrates due to the greater peat content, which is fertilized by the manufacturing company. Regarding the P content, S3 and S4 are the most enriched substrates due to the presence of vermiculite [69]. In summary, all substrates present good characteristics as growing media, but the presence of sand increases pH and decreases the EC, OM, moisture, and TKN; thus, S1 and S3 are better substrates.

As can be seen in Fig. 1, the EC in all stabilized soil-char mixtures (1–5% d.w.) ranges from 149 to 224 mS/m, showing an increase when the dosage of char increases, suggesting that the OM supply by char addition could positively contribute to providing nutrients to plants without compromising germination by excess salt [70]. Soil-char mixtures show no remarkable effect on EC at low dosages (1% d.w.), whereas at high dosages, the EC increases when compared with bare soil. Biochar and thermally treated hydrochar show similar ECs to that of the bare soil (163 mS/m) at low dosages, with slightly higher values for biochar at each dosage, as higher temperatures were involved in its production. Washed hydrochar shows the highest EC value at low dosages (184 mS/m), similar to the values reported for the other soil-char mixtures at 3% d.w., whereas at high dosages, the highest EC is reported by the aging post-treatment (224 mS/m). Regarding pH, fresh and aged hydrochars show no effect upon this parameter while washing reduces the pH from 7.6 to 6.8 and pyrolytic chars increase pH as the dosage increases. The higher pH of BC with respect to THC is correlated to the characterization of carbonaceous materials and induced by high operating temperatures.

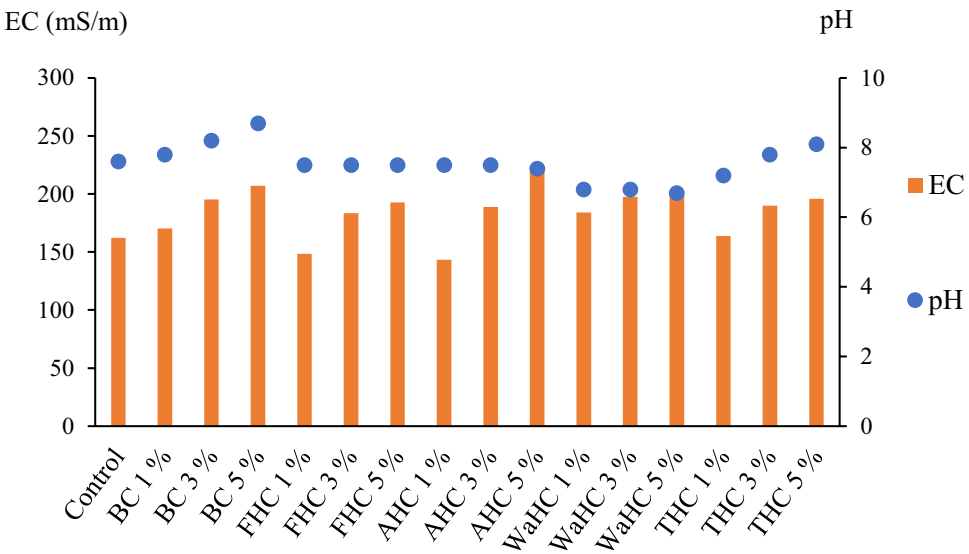

Fig. 1 Electrical conductivity and pH variations of soil-char stabilized mixtures after 1 week using biochar, fresh hydrochar, and post-treated hydrochars

3.3 Germination and growth tests using peat-based substrates with fresh hydrochar

Figure 2 shows the time course of the germination index of *Arabidopsis thaliana* on the four substrates studied (S1–S4). The type of substrate and FHC concentration used cause significant differences in the GI ($p < 0.001$). As previously observed by Busch et al. [23, 71] using beetroot chip hydrochar on barley and salad seeds, the increasing concentration of FHC negatively affects the GI, which indicates a germination inhibition with all substrates, especially on S2, at FHC dosages above 2.5%. Therefore, the partial substitution of peat with fresh hydrochar does not seem to be an alternative for *Arabidopsis* germination. S4 and more markedly S3 reach lower GI when compared with S1 and S2, even in the control assay, suggesting that regardless of the presence of char, mixtures containing vermiculite do not have a positive effect on *Arabidopsis* germination.

The time courses of tomato and quinoa seed germination were also evaluated (Figs. 3 and 4, respectively). As can be observed, tomato sown on S3 shows a slight increase in GI at higher FHC concentration, which is not detected for quinoa. Nonetheless, the survival rate of both species is higher than that of *Arabidopsis*, showing that *Arabidopsis* is more sensitive to hydrochar application than tomato or quinoa. However, no germination is observed for 10% S4 owing to the presence of mold. The FHC application has a different effect on the evaluated growth medium (Fig. 3) ($p < 0.01$). While the GI slightly increases with the highest dose of FHC in substrate S3, a marked decrease in the GI is caused by a 5% FHC concentration in substrate S4. This could be because of the presence of sand in this substrate, resulting in poor water retention in combination with a high concentration of hydrophobic hydrochar that leads to excessive water drainage and, subsequently, poor nutrient availability [17]. The difference in the water holding capacity of substrates could also affect the solubility of toxic compounds that are trapped in the peat vermiculite complex and become more available in sandy growing media [72].

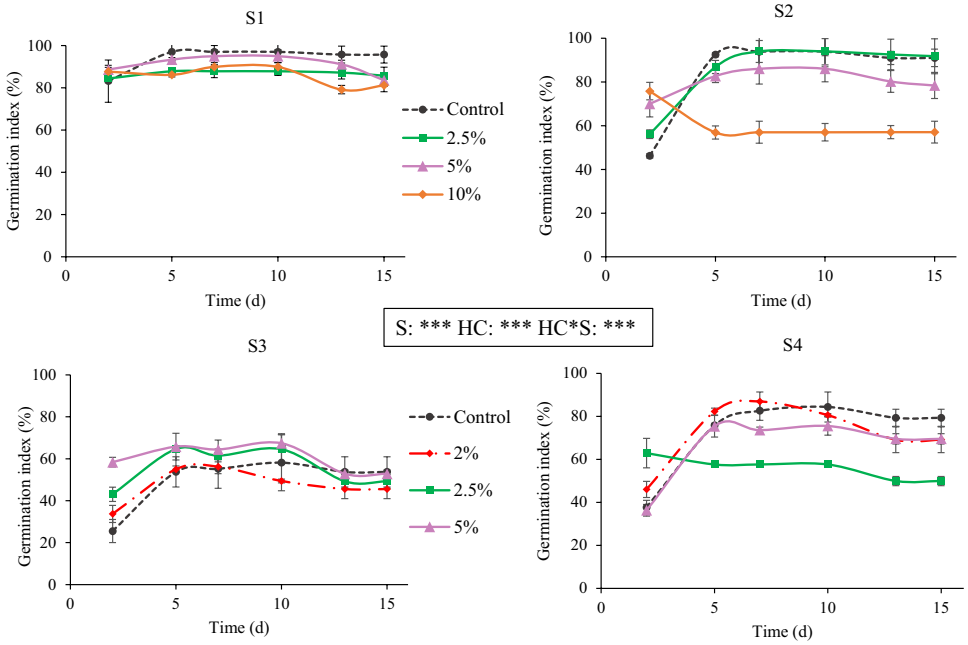

Fig. 2 Germination index of *Arabidopsis thaliana* on different substrate-FHC mixtures. ***: $p < 0.001$

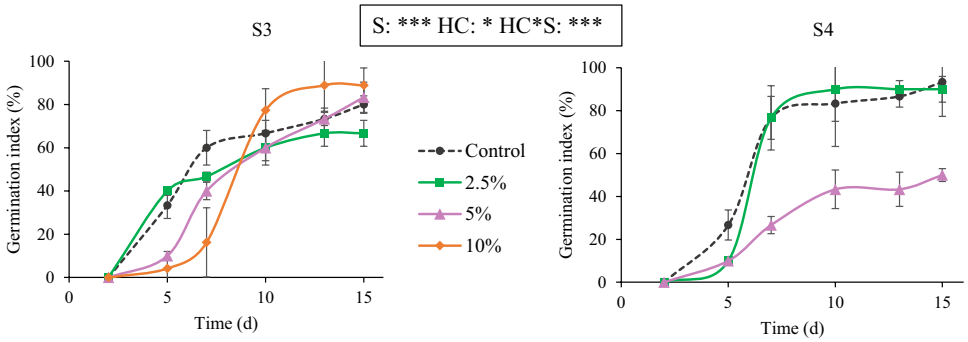

Fig. 3 Germination index of tomato on different substrates-FHC mixtures. ***: $p < 0.001$; *: $p = 0.05$

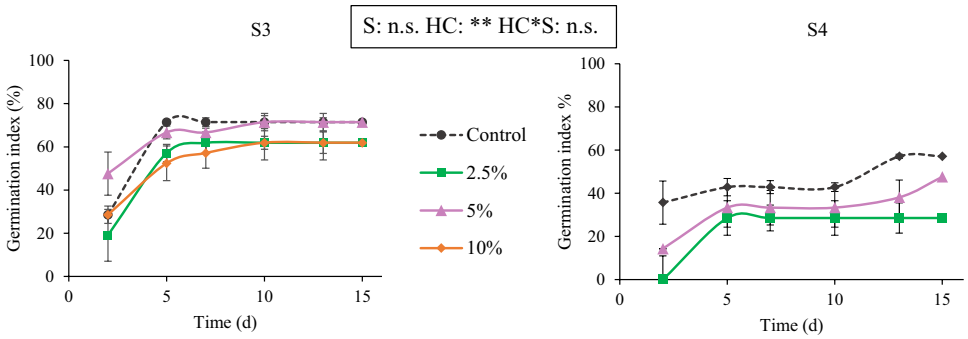

Fig. 4 Germination index of quinoa on different substrates-FHC mixtures. **: $p = 0.01$; n.s., not significant

Different behavior between the substrates ($p < 0.001$) (with S4 showing a 20–40% drop) and FHC concentrations ($p < 0.05$) was found for quinoa GI (Fig. 4). While no remarkable difference using S3 substrate among the dosages evaluated is observed, the FHC application in the sandy substrate (S4) produces a decrease in the GI. The presence of phytotoxic compounds like phenols or PAHs and the adsorption of dissolved organic compounds such as volatile fatty acids may also led to a lower GI [26, 27]. As mentioned above, the water holding capacity variation of substrates can affect the solubility of toxic compounds with a higher availability in sandy growing media, which explains the differences in char addition on the evaluated substrates [72].

The effect of FHC concentration on *Arabidopsis* growth was assessed by evaluating the changes in leaf area (Fig. 5). The presence of FHC negatively affects the leaf

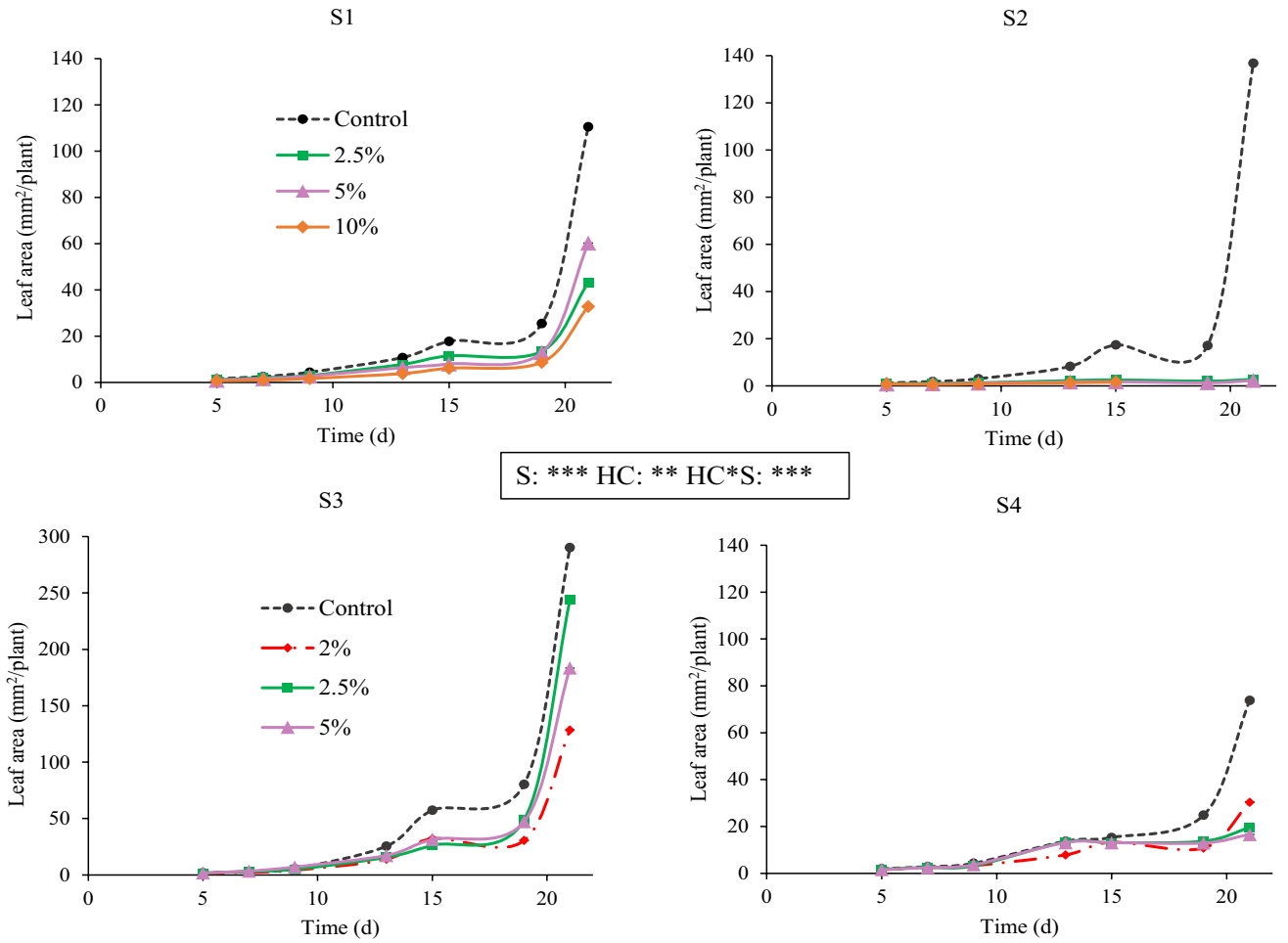

Fig. 5 Leaf area *of Arabidopsis thaliana* plants growing in different substrate-FHC mixtures as a function of time. ***: $p < 0.001$; **: $p = 0.01$

area regardless of the concentration used in S2 and S4, as reported by Yin et al. [62], who determined a decrease in lettuce shoot biomass using reed straw hydrochar. This effect might be attributed to the physicochemical properties of the substrate, such as the low water retention of sand (preventing nutrient absorption by the plant) and/or its lower sorption ability to complex low organic phytotoxic compounds coming from FHC. The S3 substrate exhibits the highest plant growth, probably because of the high OM content of peat, which, in combination with vermiculite, provides higher moisture retention and aeration. These results highlight the importance of choosing an adequate type of char material for each crop, plant, and soil [31]. Regarding the FHC concentration, we found that plants growing in S3 suffer a soft growth inhibition as the dose increases, while in the other substrates, a significant decrease in leaf area is observed in comparison with the control.

Although tomato and quinoa GI is quite similar to the control when using S3 substrate, the addition of FHC to the soil produces plant growth inhibition in both growing media. These results are consistent with those obtained by Yin et al. [70], who found that the addition of reed straw pristine hydrochar on soil reduced the fresh biomass of lettuce by about 30%. As can be seen in Figs. 6 and 7, great differences in plant FW for both substrates are obtained. This effect on plant growth worsens with increasing FHC dosage. Quinoa plants are more affected than tomato ones, especially when using sandy soil (S4). This can be due to the presence of sand in the S4-FHC mixture, which enhances water drainage, thereby reducing the soil water retention and therefore affecting water availability for plants. In fact, sandy media shows a poor OM content compared to peat-based substrates which might affect plant growth by nutrient deficiency [73].

3.4 Germination assay using soil with fresh hydrochar, post-treated hydrochar, and biochar.

Figure 8 shows the germination index of tomato, 5 days after germination, using agricultural marginal soil mixed with fresh and post-treated hydrochar and biochar. This is compared with the germination of bare soil (100%). All post-treated hydrochars have no negative effects on tomato germination, which alleviates the inhibition shown by FHC. Indeed, at low concentrations, FHC gives values that are lower than those of the control, but being exacerbated at higher dosages, in line with the findings of the meta-analysis

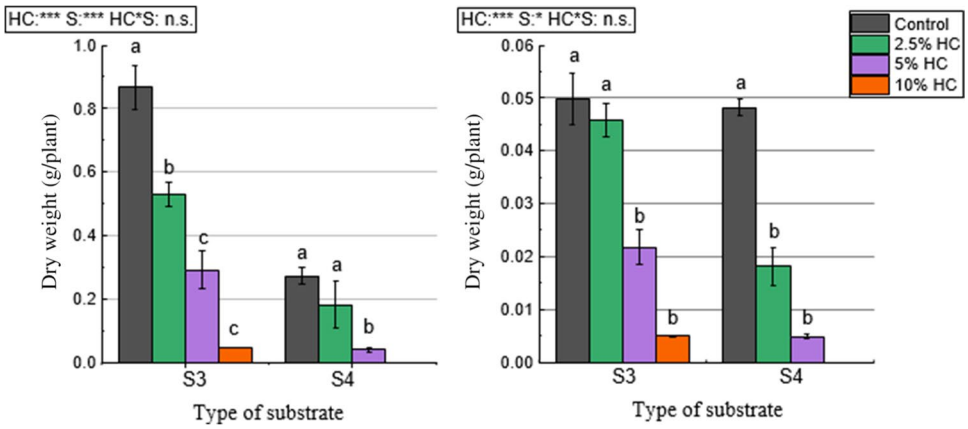

Fig. 6 Fresh and dry weight of tomato after 21 DAS. Bars with a different letter indicate significant differences. ***: $p<0.001$; *: $p=0.05$; n.s., not significant

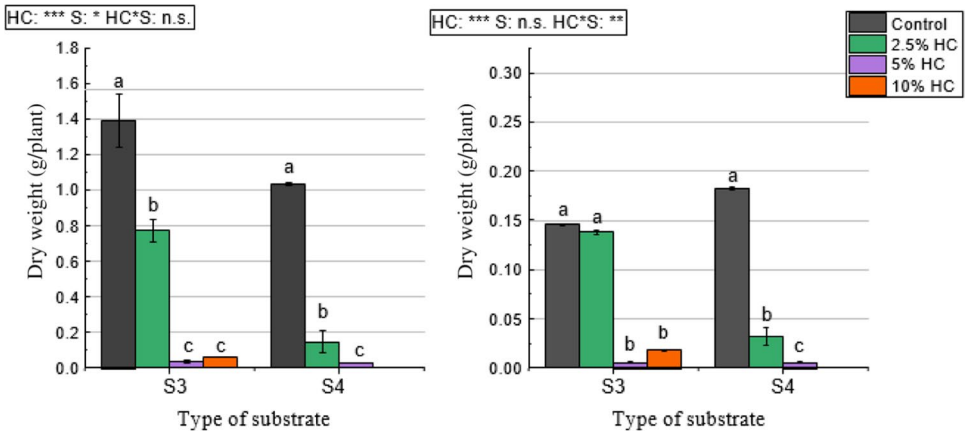

Fig. 7 Fresh and dry weight (FW and DW, respectively) of quinoa plants after 43 DAS. Bars with a different letter indicate significant differences. ***: $p<0.001$; **: $p=0.01$; *: $p=0.05$; n.s., not significant

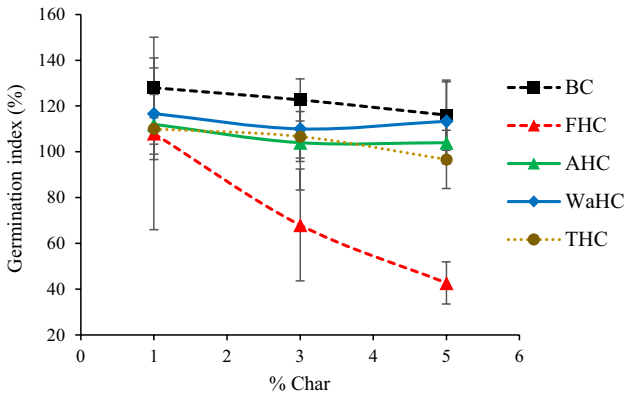

Fig. 8 Germination index of tomato on marginal soil using fresh hydrochar, post-treated hydrochar, and biochar

performed by Luutu et al. [74], who also reported severe germination inhibition above an average of 2.5% (w/v) when using GPW fresh hydrochar. EC and pH values of the mixtures (Fig. 1) do not reach values that could impact negatively on seed germination, and each char shows heavy metals concentrations below regulation limits. No other toxic effects are expected from essential elements values reported in Table 3, although K concentration is significantly reduced by all post-treatments (washing, aging, and thermal treatment) and Ca levels enhanced, although to a lower extent. The N/P/K ratio (Table 2) also reflects that main differences between fresh and post-treated hydrochars are related to K content. Hence, the presence of toxic organic species such as furfural, furans, aromatic organic acids, and phenolic compounds may be the main sources of germination inhibition [75–77].

The thermal treatment is an effective method to remove those harmful substances [28] producing a THC that avoids the negative effects of FHC on GI. Furthermore, the energy requirements for this post-treatment is similar than the needed for pyrolysis and higher than hydrothermal processing, washing, or aging. BC and THC show both higher nitrogen and phosphorus content but worse C/N ratio than the rest of post-treated hydrochars, being more hydrophobic materials which could lead to poorer water holding and leaching of nutrients when used in sandy media. AHC shows a similar trend compared to THC specially at low dosages and does not have a negative effect on the GI in agreement with Puccini et al. [27], who reported that aging garden and pruning waste FHC reduced lettuce germination inhibition, suggesting that it could be mainly attributed to the presence of polyphenols (tannins) and VFAs, which were effectively removed during aging post-treatment.

Since most of organic acids, VFA, and several potentially toxic compounds have also been removed with the washing procedure, while phenolic compounds remain at low levels, GI in WaHC should present less toxicity than the fresh hydrochar as is observed in our results. Thus, a low toxicity should be addressed to phenolic compounds. WaHC also presents the highest Ca/K ratio (Table 3) and the lowest C/N ratio of all the post-treatments tested in this experiment. WaHC and BC mixtures show the greatest GI enhancement of approximately 15% and 25%, respectively, at low dosages (1% d.w.). However, at the highest dosage (5% d.w.), a similar improvement (of approximately 15%) is achieved by washing treatment and the pyrolytic char. As expected, high temperatures involved on biochar production remove toxic substances; however, the remarkably higher energetic requirements and low yield of pyrolysis as well as the simplicity of washing procedure make the WaHC the best option for the use as soil amendment.

3.5 GC–MS analysis of the washing cycles leachates of fresh hydrochar to obtain washed hydrochar

The leachates obtained from the washing procedure applied to FHC contain high molecular weight species with low biodegradability (Figure S1 and Table S1), such as acids, cyclic, nitrogenous, aromatic, and phenolic compounds from the degradation of lignin during the HTT treatment, as well as amino acids, such as D-allotreonine, or long-chain alkyl compounds produced by the degradation of proteins, sugars, and Maillard reactions, according to the findings of Simangunsong et al. [78]. Furans, amines, amides, pyridines, pyrazines, benzoic compounds, and organic acids may affect seed germination and plant growth. Most of these species are removed during the washing procedure, as shown in Table S1. These results are in agreement with those found by Karatas et al. [20], who reported a reduction in the range of 75–96% of alkyls, furans, and polyaromatic compounds after FHC washing. Figure S1 shows the partial removal of most of the phenolic compounds and 2,4-dimethoxytoluene. Additionally, other refractory species (propylamine and 2,3-dietilpyrazine) remain unaltered after the washing cycles, suggesting that they can be found in similar amounts in FHC and WaHCs. The presence of these refractory species may still affect the microbiome, nutrient uptake, or have toxic effects on plant growth at high concentrations.

Figure 9 shows the individual distribution (C2–C7) and total volatile fatty acid content diminishing due to the washing procedure. Acetic (approximately 20%), propionic (approximately 50%), and (iso)butyric (approximately 5–10%) acids are predominant in the pool of TVFA. Most of the acetic (50%) and propionic (60%) acids are removed during the first washing cycle, while longer chain acids (C5–C7) remain in similar abundance after the washing procedure. A reduction of approximately 70% in the TVFA content is observed in the second cycle (WC2), reaching approximately 90% during the third wash (WC3) when compared with the

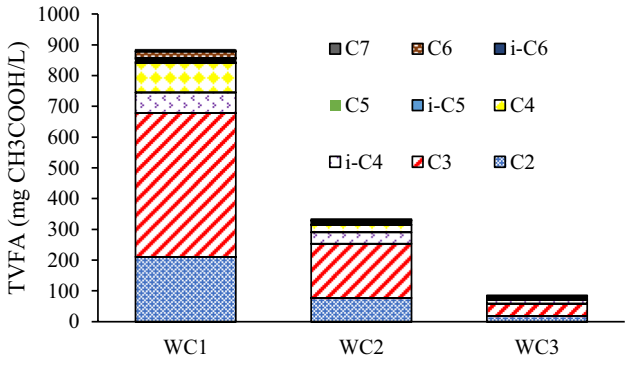

Fig. 9 Total volatile fatty acid content in the leachates of each washing cycle to obtain WaHC

initial effluent. Puccini et al. also found a drastic reduction in the TVFA content in washed and aged hydrochar of GPW [27].

Hydrochar upgrading by washing post-treatment removes soluble organic matter, including potential toxic substances, but also generates a side stream with similar composition to the diluted process water from hydrothermal treatment that could be managed through different approaches. Thus, its recirculation to the process (feedstock/water mixture) has been proposed, reducing the water requirement of the system [79, 80]. Moreover, it can be used for energy recovery by anaerobic digestion/co-digestion improving the overall energy balance of the process [7, 11]. Another way to valorize this by-product could be the nutrient recovery as phosphorus precipitation [80, 81]. Finally, once the ecotoxicity has been measured and minimized by dilution, it can also be used as source of water for irrigation [82].

4 Conclusions

Fresh and post-treated hydrochar, as well as biochar from lignocellulosic residues, presented suitable characteristics for use as soil conditioners, such as high C and nutrient (N, P) contents, and low toxic metal concentrations (below the regulated limits categorized as class A amendments). However, the application of fresh hydrochar to peat-based substrates, especially those containing sand, inhibited germination and showed a negative impact on plant growth in *Arabidopsis thaliana*, tomato, and quinoa crop plants. The same effect was observed on tomato seed germination in marginal agricultural soils amended with FHC. However, all hydrochar post-treatments alleviated the germination inhibition, which improved the GI relative to that of the control soil, especially at dosages above 1%. Washing was effective for VFAs, furans, amines, amides, pyridines, pyrazines, and benzoic compounds removal. At the highest dosage, WaHC enhanced germination compared to bare soil up to 15%, similar to the GI showed by biochar obtained at 900 °C. Considering techno-economical and energy aspects, as well as GI improvement, washing is the most feasible hydrochar post-treatment before application to soils.

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Author contribution *Eneko Suarez*: investigation, methodology, data curation, writing—original draft, formal analysis. *Montserrat Tobajas*: conceptualization, investigation, data curation, writing—review and editing, supervision, validation. *Angel F. Mohedano*: data curation, writing—review and editing, funding acquisition, validation. *Maria Reguera*: conceptualization, investigation, methodology, data curation, writing—review and editing. *Elvira Esteban*: conceptualization, investigation, methodology, data curation, writing—review and editing. *Angeles de la Rubia*: conceptualization, data curation, writing—review and editing, funding acquisition, supervision, validation.

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Data availability The authors confirm that the data supporting the findings of this study are available within the article [and/or] its supplementary materials. Derived data supporting the findings of this study are available from the corresponding author on request.

Declarations

Ethical approval No applicable.

Conflict of interest The authors declare no competing interests.

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