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Exhausted fire-extinguishing powders: A potential source of mineral nutrients for reuse and valorisation in compost enrichment for soilless cultivation

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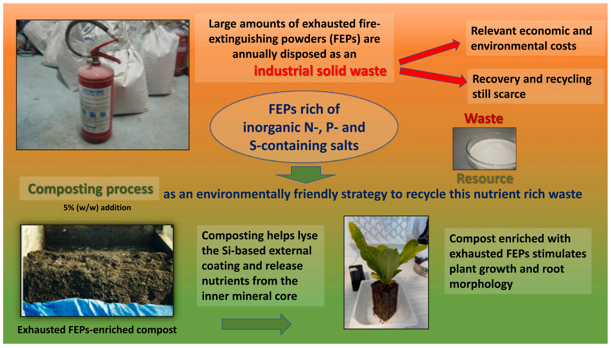
HIGHLIGHTS

- Exhausted fire-extinguishing powders (FEPs) contain inorganic N, P and S salts.
- Composting helps lyse Si-based FEPs coating and release inner mineral nutrients.
- Compost enriched with exhausted FEPs stimulates plant growth and root morphology.
- Low dosage of compost enriched with exhausted FEPs is no phytotoxic to lettuce.
- K derived from compost mitigates excess Na released from exhausted FEPs.

GRAPHICAL ABSTRACT



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ABSTRACT

Fire-extinguishing powders (FEPs) are constituted by an inner mineral core of $(NH_4)H_2PO_4$ and $(NH_4)_2SO_4$ salts (>95 %, by weight) externally coated with Si-based additives, which make problematic reuse after their service life has expired (36 months). This study aimed to assess the feasibility of using the composting process as an environmentally friendly strategy to lyse the external coating and recycle this nutrient-rich solid waste for replacement of inorganic fertilization in soilless cultivation of horticultural crops. A microcosm-scale experiment with lettuce plants grown into a soil/quartz sand mixture under controlled conditions for 28 days was used to investigate plant responses (fresh and dry biomass, chlorophyll fluorescence parameters, root morphology, ash and nutrients content) to amendment with increasing dosages (equivalent at 0, 10, 20 and 30 t ha^{-1}) of an exhausted FEPs-enriched compost. Chemical properties (pH, EC, TOC, TN) and content of soluble nutrients (Na^+, K^+, Mg^{2+}, Ca^{2+}, NH_4^+, $H_2PO_4^-$, SO_4^{2-}, NO_3^-, Cl^-) released into the growing substrate were also monitored. Non-amended microcosms and non-enriched compost treatments were taken as controls. Results showed, beside the expected rise of phosphate, sulphate and ammonium ions, exhausted FEPs contributed Ca^{2+}, Mg^{2+} and Na^+ content. Whereas compost determined a dose-dependent release of K^+, which was particularly useful in maintaining the K/Na ratio in a range not harmful to plant physiology. It was also found that the compost enriched with 5 % (*w*/w) exhausted FEPs was no phytotoxic to lettuce. On the contrary, it stimulated the plant growth, increased the photosynthetic efficiency and the shoot biomass accumulation, thus incrementing the shoot/root

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ratio. Moreover, it oriented the root morphology development and promoted the plant uptake of both water and solutes. To sum up, composting represents a suitable alternative to chemical treatment to recover readily available nutrients contained in exhausted FEPs and produce an enriched compost for use in soilless cultivation.

1. Introduction

Most common multi-purpose ABC-E fire-extinguishing dry powders (FEP) are constituted by a finely divided mixture of mineral salts composed of mono-ammonium phosphate ($(NH_4)H_2PO_4$) and ammonium sulphate ($(NH_4)_2SO_4$) as main ingredients (>95 %, by weight). Ammonium phosphate is industrially manufactured by reacting ammonia and phosphoric acid, the latter being derived from mined phosphate raw materials (apatite and phosphorite) of either sedimentary (87 %) or igneous (13 %) origin (Geissler et al., 2018). It must be noted that in their commercial formulations, FEPs are externally coated with colouring additives, fluidifying agents and silicone-based additives to improve their rheological properties and increase flowability and water-repellence (Kondrashova et al., 2017). These external coating agents, such as water-proofing and organosilicon-based liquids, make problematic the environmentally friendly disposal of the exhausted powders after their life expiration 36 months, under current international (UNI EN ISO 9994-1) and national (DM 13/3/1998) legislation), with little chance of reuse their inner mineral core (Kunin et al., 2016). According to recent estimates, an amount as high as 100.000 t of fire extinguisher materials are annually disposed as an industrial waste, with relevant economic and environmental costs (ESPP, 2017). Nonetheless, studies on recovery and recycling of raw materials (mined phosphates, sulphate) from exhausted FEPs are still scarce, and only a few authors have proposed aprotic solvent-based systems for the recovery of the inner mineral core (Michelotti, 2012; Dotelli and Viganò, 2020). Phosphate rock reserves, which are globally estimated as large as 60 Gt, will become exhausted as early as over the next 64–130 years (after reaching a peak in the mid-21st century), depending on the increasing trend of phosphate rocks mining (van Kauwenbergh, 2010; Koppelaar and Weikard, 2013). Thus, recognizing phosphate rock as a finite and non-renewable critical resource, recycling of P-rich waste products has become an urgent need in food and industrial production chain (Sarvajayakesavalu et al., 2018; Nenov et al., 2019).

Waste composting plays a key role in advanced circular economy models as an economically and environmentally friendly strategy for providing multiple ecosystem services such as reduction of landfilling and incineration, recovery of nutrients and energy, organic waste conversion in value-added industrial products, and also mitigation of the global greenhouse effect (Mikula et al., 2020; Vaverková et al., 2020; Palansooriya et al., 2023). In fact, metabolically active microbial communities operating during the composting process are responsible not only for the biochemical breakdown of organic compounds and *de-novo* synthesis of humic-like molecules (Guo et al., 2019), but also for C and N conservation (Huang et al., 2022), and the biodegradation and detoxification of organic waste components (Nozhevnikova et al., 2019). Nonetheless, within the context of sustainable management of agroecosystems, usage of composted materials constitutes a key measure to recycle and regulate nutrient release and thus to increase limited resources use efficiency (Schröder et al., 2018). Thus, in line with the urgent need to cope with "the transition to a more circular economy" the composting process can be proposed as a technically feasible method to biologically lyse the silicon-based external coating of exhausted fire-extinguishing particles and recover the inner mineral core to produce a nutrient-enriched compost for use in agriculture.

Given this premise, this paper examines the potential of using a mature compost enriched with exhausted FEPs during the composting process to entirely replace the supply of inorganic fertilizers in soilless cultivation of a horticultural crop with no detrimental effects on plant growth. Lettuce was selected in this study because it is the first salad crop cultivated and commercialized internationally (~27 million tonnes per year globally; FAOSTAT, 2021). Moreover, lettuce is widely used in ecotoxicological assays to test compost-based growing media for its sensitivity to inorganic contaminants, organic pollutants, and high salt concentrations (Emino and Warman, 2004; Gattullo et al., 2017). Plant responses were evaluated in terms of fresh and dry biomass, chlorophyll fluorescence parameters, root morphology and inorganic nutrient content. In addition, chemical properties (pH, EC, total organic C and total N content) and readily available nutrient (sodium, potassium, magnesium, calcium, ammonium, phosphate, sulphate, nitrate, chloride) content of the growing substrate were measured before and after the 28-day observation period.

2. Materials and methods

2.1. Exhausted fire-extinguishing powder (E-FEP)

The fire-extinguishing powder (ABCE class fire, ProPHOS Chemicals S.r.l.) was industrially produced by ProPHOS Chemicals S.r.l. (CR, I) and kindly supplied by a national fire extinguisher dealer as a mixture of fine particles (99 % under 0.250 mm and at least 44 % below 0.040 mm) after its expiring date. Principal ingredients of the powder are: $NH_4H_2PO_4$ (40 ± 2 %) (Reach registration 01–2,119,488,166-29), $(NH_4)_2SO_4$ (52 ± 2 %) (Reach registration 01–2,119,455,044-46) and coating additives (~5 %). Heavy metal content: As <0.5 ppm, Cd < 0.2 ppm, total Cr 7.1 ppm, Hg < 0.2 ppm, Ni 2.38 ppm, Pb < 0.5 ppm, Cu 12.6 ppm, Se < 0.5 ppm.

2.2. Plant material

Seeds of lettuce (*Lactuca sativa* L. cv. "Romana") were purchased from Fratelli Ingegnoli (MI, Italy), germinated and grown for four weeks before transplanting.

2.3. Soil and substrates

The soil was a clay loam: sand 34.0 %, silt 32.0 %, clay 34.0 %, bulk density 1.23 ± 0.04 kg dm^{-3}, pH_{H2O} 7.2 ± 0.2, pH_{KCl} 6.4 ± 0.1, total organic C 14.9 ± 1.5 g kg^{-1} dw (dry weight soil), total N 1.7 ± 0.1 g kg^{-1} dw soil, C/N ratio 8.8, $EC_{1:2}$ at 25 °C 0.271 ± 0.018 dS m^{-1}, CEC 20.9 ± 1.3 $cmol_{(+)}$ kg^{-1} dw soil, total $CaCO_3$ 10.0 ± 0.5 g kg^{-1} dw soil, active $CaCO_3$ 3.9 ± 0.6 g kg^{-1} dw soil. The soil was collected from the top 15-cm layer of an agricultural field located at the Agricultural Experimental Station of the University of Reggio Calabria, and freshly sieved at 4 mm opening mesh. Soil properties were determined according to standard methods (Sparks et al., 1996).

Quartz sand (particle size 2.0 mm, SiO_2 96.75 %, Al_2O_3 2.30 %, K_2O 0.75 %, Fe_2O_3 0.15 %, TiO_2 0.05 %; purchased from Croci Trading Company s.r.l., VA, I) was thoroughly washed with deionized water and oven-dried at 60 °C until complete dryness before use.

Mature compost enriched with the exhausted fire-extinguishing powder (95:5 biomass:powder, *w*/w) was obtained after a 3-month composting process of mixed livestock and crop solid wastes. Before use, it was sieved through a 30-mm opening sieve. Compost major properties (Table 1) were determined according to standard methods (UNI, 1998; ANPA, 2001; Tambone et al., 2015).

2.4. Experimental set up

Soil microcosms consisted of square plastic pots (12 cm height x 7 cm

Table 1
Main properties of the composted material from mixed livestock and crop solid wastes enriched with the exhausted fire-extinguishing powder (FEP) (95:5 compost:powder, *w*/w)[a]. Mandatory limits for use as a soil conditioner for organic fertilization are according to the current Italian legislation (legislative decree no. 75/2010).

Parameter	Value	Mandatory limits
pH (25 °C)[b]	7.8 ± 0.3	6.0–8.8
EC (dS m^{-1}; 25 °C)[c]	4.4 ± 0.5	–
Dry matter (% fresh weight)	16 ± 1	≤ 50
Total organic C (%)	26 ± 2	≥ 20
Total N (%)	2.2 ± 0.3	–
Organic N (% total N)	90 ± 1	≥ 80
C/N	11.5 ± 0.5	≤ 25
Humic and fulvic C (%)	11.0 ± 1.9	≥ 7
Cr_{VI} (mg kg^{-1})	< 0.1	< 0.5
Cd (mg kg^{-1})	0.53 ± 11	< 1.5
Cu (mg kg^{-1})	74 ± 14	< 230
Hg (mg kg^{-1})	< 0.2	< 1.5
Ni (mg kg^{-1})	15 ± 3	< 100
Pb (mg kg^{-1})	23 ± 4	< 140
Zn (mg kg^{-1})	244 ± 36	< 500
Salmonellae spp. (CFU 25 g^{-1})	Absent	Absent
Escherichia coli (CFU g^{-1})	< 100	≤ 100

[a] Values (means ± SD, $n = 2$) are expressed on a dry matter basis.
[b] Biomass/H_2O, 3:50, *w/v*.
[c] Biomass/H_2O, 1:10, w/v (1 dS m^{-1} = 1000 µS cm^{-1}).

diameter, ~588 cm^3 volume) closed at the bottom by a thin layer of nylon stocking material and filled with 580 g of a soil/quartz sand (50:50, w/w) mixture. Before transplanting, either *E*-FEP-enriched (C + FEP) or non-enriched (C) compost was added at three doses: 1, 2 and 3 kg m^{-2} (C1, C2, C3), corresponding to 10, 20 and 30 t ha^{-1}. These rates were established by considering the dosage commonly applied to maintain the C budget in Mediterranean soils where TOC is often below the critical threshold of 2 % (Zdruli, 2012), and following the suggested amendment rates outlined by Raviv (2013) when using compost as a component in potting mixtures. Control pots filled with the potting mixture alone (0 g compost) (C0) were also included in the experiment. Soil microcosms were brought to 70 % water holding capacity with deionized water and placed in a growth chamber under controlled conditions (temperature 20 ± 1 °C, relative humidity 70 %, 10-h daylight photoperiod, PAR 400 W m^{-2} at plant height) for 15 days until the tillage effect had declined (Tortorella and Gelsomino, 2011). Then, young seedlings of lettuce (4-week-old and with 8–10 leaves) were transplanted into the soil microcosms (1 seedling per microcosm) and left growing under the same growth chamber conditions previously reported for additional 28 days, a time frame needed for transplanted lettuce to reach its peak in vegetative development (Fig. S3). Unplanted (bare) microcosms were also included in the experiment. Thus, treatments were the following: unplanted soil (S + C0, S + C1, S + C2, S + C3, S + C1 + FEP, S + C2 + FEP, S + C3 + FEP) and planted soil (SP + C0, SP + C1, SP + C2, SP + C3, SP + C1 + FEP, SP + C2 + FEP, SP + C3 + FEP).

Microcosms were randomly arranged in a randomized complete block design, with three replications in order to compare: 4 compost dosages (0, 1, 2 and 3 kg m^{-2}), 2 compost treatments (enriched or non-enriched with FEP) and 2 plant treatments (with or without lettuce plants). Microcosms mixtures were kept at 70 % water holding capacity by periodical (every 2 or 3 days) addition of distilled water.

2.5. *Plant analysis: chlorophyll fluorescence parameters*

Chlorophyll fluorescence parameters were estimated weekly at 7, 14, 21 and 28 days after transplanting. The imaging technique was performed by using an IMAGING-PAM chlorophyll fluorometer (Imaging-PAM ver. MAXI, Walz, Germany), as described by Genty et al. (1989). These measurements allow to estimate the photochemistry of the photosynthetic system II (PSII) from the fluorescence decline, termed "quenching". Precisely, a decrease in fluorescence due to photochemistry is called *photochemical quenching.* The basal fluorescence (F0) was determined on dark-adapted samples using non-actinic measuring flashes modulated at 1 Hz, this pulse was required to reduce the plastoquinone pool. The maximum fluorescence yield F_m was determined with a saturating pulse of 8000 µmol m^{-2} s^{-1} PPFD with a 1–2-s duration. The F_0 and F_m values were subtracted and divided $[(F_m - F_0)/Fm]$ to calculate the maximum quantum efficiency of PSII photochemistry: F_v/Fm. Other detected parameters related to photochemical quenching were: the electron transport rate (ETR, μmol electrons m^{-2} s^{-1}) and the effective quantum yield of the PSII photochemistry (Y(II)).While the non-photochemical quenching (NPQ) was the fluorescence emitted but not due to photochemical activity (Bilger and Björkman, 1990; Kramer et al., 2004). NPQ represents a valuable feature for the study of photosynthetic organisms' light utilization and dissipation (Malnoë, 2023).

2.6. *Plant analysis: total biomass, root morphology, and inorganic nutrient content*

At the end of the growing period, microcosms were destructively sampled. Lettuce plants were gently removed from the pot mixtures, separated into shoot and root, rinsed with de-ionized water and their fresh weight determined. The shoot dry weight (WS, g) was measured after oven-drying at 70 °C for 72 h. The root system was stained with 0.1 % (*w/v*) toluidine blue O for 5 min, thoroughly washed with water and then scanned (WinRhizo STD 1600, Instruments Régent Inc., Canada) at a resolution of 600 dpi for morphological analyses. Scanned images were processed using the WinRhizo® root analysis software (Régent Instruments) to measure total root length, surface area, average diameter, and volume. Then, the root dry weight was determined after oven-drying at 70 °C for 72 h. Total plant dry weight was obtained by summing root and shoot dry weights. Based on the measurements above, the following morphological ratios were calculated: specific root length (root length per unit of root dry weight, SRL), and root tissue density (root dry mass per unit root volume, RTD) which represent functional parameters.

Total inorganic cations (sodium, potassium, magnesium, calcium) and anions (chloride, nitrate, phosphate, sulphate) together with malate were extracted from lyophilised root and shoot samples according to Conversa et al. (2020), filtered through a 0.22 µm Millipore™ filter and stored at −20 °C before chromatographic analysis.

2.7. *Growing medium: chemical properties and water-extractable inorganic nutrients*

Aliquots (50 g) of the growing medium were sampled immediately before transplanting (time 0 d) and at the end of the observation period (time 28 d). Standard methods (Sparks et al., 1996) were used to measure pH, EC, total organic C (TOC) and total N (TN) content. Briefly, soil acidity was potentiometrically measured in a 1:2.5 (*w/v*) soil-to-0.01 M $CaCl_2$ solution mixture (pH_{CaCl2}); electrical conductivity was measured at 25 °C in a 1:2 (w/v) soil-to-water ratio slurry ($EC_{1:2}$ 25 °C). Total organic C and N were analysed by an elemental analyser LECO CN628 (LECO Corporation, MI, USA). Readily available nutrients (nitrate, chloride, phosphate, sulphate, ammonium, sodium, potassium, magnesium, calcium) were water-extracted (substrate:water 1:10, w/v) under horizontal shaking at room temperature for 2 h, centrifuged at 5000*g* for 5 min and filtered through Whatman™ n. 42 filter paper. Filtrates were passed through a 0.22 µm Millipore™ filter and stored at −20 °C before chromatographic analysis.

2.8. *Ion chromatography*

Inorganic cations and anions were determined by ion chromatography using a Dionex™ ICS 1100 (Dionex Corp., Sunnyvale, CA, USA) equipped with an isocratic pump, a conductivity detector, and an autosampler (Dionex™ AS-DV, ThermoFisher Scientific, Waltham, MA, USA). Cations were determined using a self-generating CDRS-600 suppressor (4 mm) (ThermoFisher Scientific), an analytical column (4 mm ID × 250 mm) Dionex™ IonPack™ CS12A RFIC™ (Dionex Corp.), and an eluent solution (20 mM methane sulfonic acid) at a flow rate of 1.2 mL min^{-1}. Anions were determined using a self-generating AERS-500 suppressor (4 mm) (ThermoFisher Scientific), an analytical column (4 mm ID × 250 mm) Dionex™ Ion-Pac™ AS22 RFIC™ (Dionex Corp.), and an eluent solution (3.5 mM sodium-carbonate/1.0 mM sodium-bicarbonate) at a flow rate of 1.2 mL min^{-1}. Instrumental control and chromatographic data processing was done by the Thermo Scientific Dionex Chromeleon Chromatography Data System 7.2 (ThermoFisher Scientific).

2.9. Statistics

Soil and plant variables were firstly checked for deviations from normality (Kolmogorov-Smirnov test) and homogeneity of within-group variances (Levene's test). Since the block effect was not significant ($P > 0.05$), the data were subjected to further statistical analysis. Soil-related data (shown in Table 2 and in Figs. 1 and 2, and in Figs. S1 and S2) were statistically processed by a three-way analysis of variance (compost dose × FEP enrichment × lettuce plant) with repeated measures; it was run in order to highlight the main effect of the compost dosage, compost enrichment with FEP, the presence of the plant and their interactions, alongside the variability given over time. Plant-related data (shown in Tables 3-6 and in Figs. 3 and 4) were analysed by two-way analysis of variance (compost dose × FEP enrichment). Tukey's *post hoc* test comparison (at $P < 0.05$) was applied for multiple pairwise comparison of means. Statistical analyses were run using the Systat 13.0 software (SYSTAT Software Inc., Erkrath, D). Graphs were drawn by using the SigmaPlot 10.0 software (SYSTAT Software Inc.).

3. Results

3.1. Growing medium: chemical properties

The three-way ANOVA evidenced that compost dosage and dosage x plant interaction significantly affected both TOC and TN values (Fig. S1). Not unexpectedly, the variability of TN values was influenced also by the FEP enrichment and its interaction with compost dosage (Fig. S1). In details, TOC resulted markedly increased (up to a 2.3-fold increase) with increasing compost dosage at the beginning of the trial. However, at the end of the 28-day observation period a similar trend of significantly different TOC contents among treatments was observed in unplanted (bare) microcosms; whereas in soil microcosms with the lettuce plant final TOC values resulted not statistically different, broadly ranging from 17.5 to 23.3 mg g^{-1} (Fig. S1). On the other side, TN evidenced a marked compost dose related increase (from 0.3 to 2.0 mg g^{-1}), which was further enhanced by FEP addition (up to 2.8 mg g^{-1} on average) at time zero (Fig. S1). A similar trend as observed for TOC was found after 28-day growing period: statistically higher values in compost amended bare microcosms, and statistically similar readings in planted microcosms (Fig. S1).

Soil pH showed a compost dose related increasing trend (from 6.6 to 6.9 at the highest dose, on average) at the start of the trial. However, after 28 days a similar trend was found in compost-treated microcosms, whereas it declined to control values when using the FEP-enriched compost, irrespective of the plant occurrence (Fig. S2). Expectedly, compost addition produced an immediate, strong dose-dependent increase in EC, which was further enhanced by the FEP enrichment. Moreover, contrasting responses were observed at the end of the experimental period depending on the plant absence/presence: EC further raised in all unplanted microcosms, whereas it declined in all planted microcosms (Fig. S2).

3.2. Growing medium: water-extractable inorganic nutrients

The compost dosage brought about a steadily increasing and persistent concentration of NO_3^- in the growing substrate (a 4.0-fold increase at the highest dosage on average, Fig. 1), as also confirmed by the three-way ANOVA (Table 2), with no significant variations between initial and final values.

A similar compost-driven effect was observed for Cl^-, but unlike nitrate, chloride concentration was also affected by FEP-enrichment (Table 2), which determined a further and persistent 30 % increase still noticeable at the end of the observation period (Fig. 1). The three-way ANOVA evidenced that both phosphate and sulphate were highly significantly ($P < 0.001$) affected by the compost dosage, FEP enrichment and their interaction (Table 2). In detail, both nutrients markedly increased (precisely from 7 to 200 μg $H_2PO_4^-$ g^{-1} and from 40 to 180 μg SO_4^{2-} g^{-1}, on average) with increasing compost dosage, becoming even more evident when applying FEP-enriched compost (precisely from 167 to 328 μg $H_2PO_4^-$ g^{-1} and from 365 to 1050 μg SO_4^{2-} g^{-1}, on average) at the highest compost rate (Fig. 1). In both cases, only slight time-dependent fluctuations were noticed between initial and final samplings since there were neither plant nor time effects (Table 2).

The three-way ANOVA evidenced that water-extractable cationic nutrients were all significantly affected by the compost dosage, FEP enrichment, lettuce plant and, in almost all cases, by their interactions (Table 2). In brief, increasingly larger ammonium amounts were detected in microcosms treated with increasing dosages of compost or FEP-enriched compost (Fig. 2). However, the largest NH_4^+ amount was recovered after applying FEP-enriched compost at rates as high as 20 and 30 t ha^{-1} in planted units (Fig. 2). Moreover, a time-dependent declining trend was also noticed between initial and final NH_4^+ values in most treatments. Same as NH_4^+, cations such as Na^+, Mg^{2+} and Ca^{2+} showed a similar pattern, being strongly increased in soil microcosms amended with FEP-enriched compost at rates as high as 20 and 30 t ha^{-1} (160 and 360-fold increase for Na^+, 16 and 11-increase for Mg^{2+}, and 260 and 142 -fold increase for Ca^{2+}, respectively) (Fig. 2). No statistic

Table 2

Significant effects due to compost dosage (D), exhausted FEP enrichment (FEP), plant (P) and their interactions on the variability of soil concentration of water-soluble inorganic anions and cations shown in Figs. 1 and 2, respectively. Statistics is presented as *F*-values and level of significance (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$; *ns* $P > 0.05$) estimated by three-way ANOVA (compost dose × FEP enrichment × lettuce plant) with repeated measures.

Main effect		Anions				Cations				
	df[a]	NO_3^-	Cl^-	$H_2PO_4^-$	SO_4^{2-}	NH_4^+	Na^+	K^+	Mg^{2+}	Ca^{2+}
Dosage	3	164.12***	299.26***	141.69***	93.65***	210.53***	98.52***	638.68***	248.70***	641.63***
FEP	1	2.81^{ns}	74.64***	83.58***	284.74***	326.87***	242.28***	76.79***	511.00***	1018.52***
Plant	1	1.30^{ns}	0.98^{ns}	0.58^{ns}	1.13^{ns}	168.41***	43.53***	8.80**	61.11***	73.71***
D x FEP	3	0.40^{ns}	28.13***	9.60***	55.03***	82.19***	50.52***	12.81***	157.50***	359.67***
D x P	3	0.50^{ns}	3.88*	0.14^{ns}	0.90^{ns}	42.86***	13.49***	8.64***	28.87***	37.55***
FEP x P	1	0.04^{ns}	1.00^{ns}	0.01^{ns}	4.40*	187.37***	30.57***	3.06^{ns}	47.01***	66.21***
D x FEP x P	3	0.66^{ns}	2.49 ^{ns}	0.56^{ns}	3.61*	55.74***	5.65**	2.97*	25.32***	32.09***

[a] Degrees of freedom.

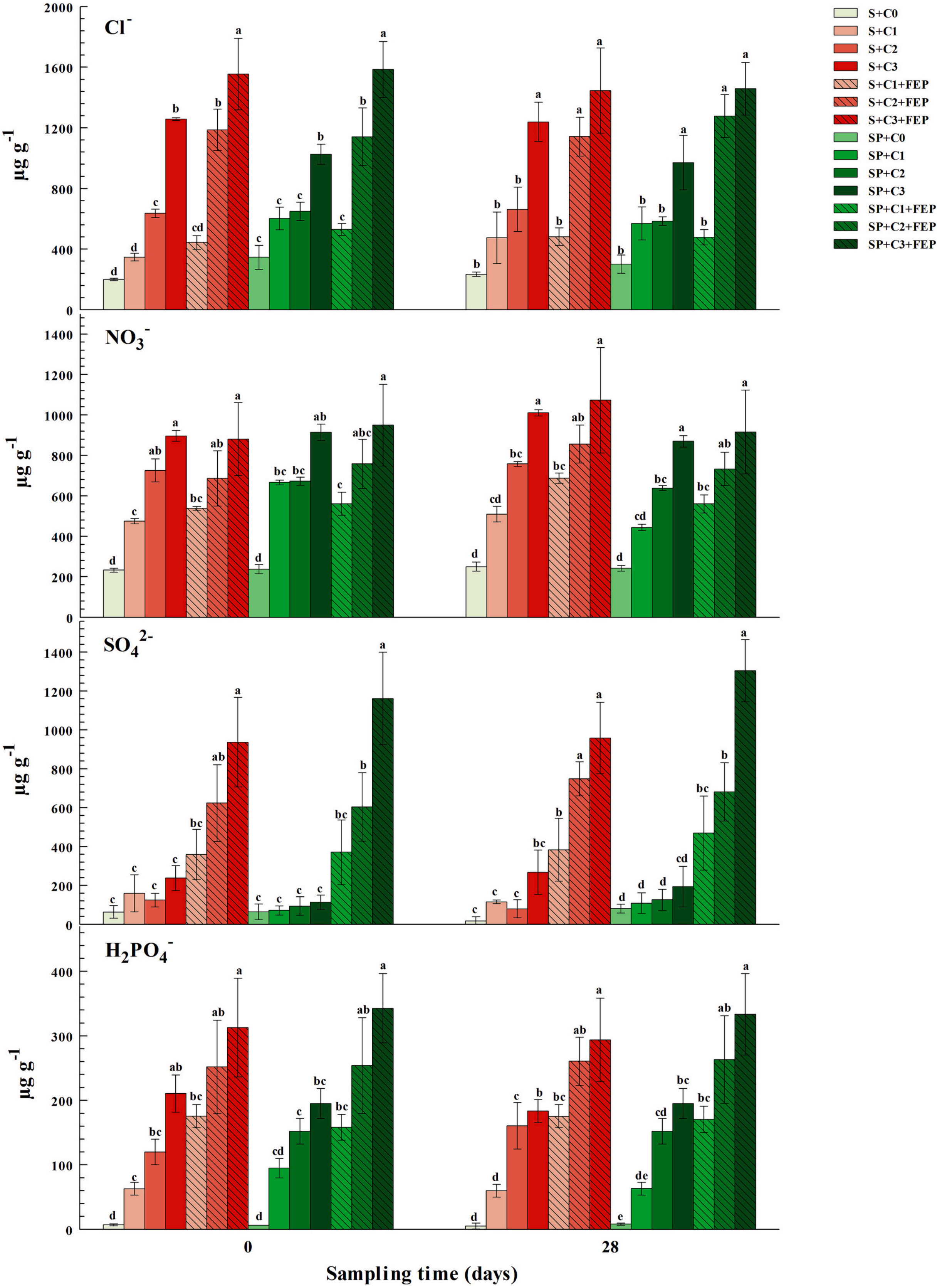

Fig. 1. Changes in water-soluble chloride, nitrate, phosphate, and sulphate (mean ± SD, $n = 3$) in soil microcosms under different treatments before and after the 28-day observation period. Within each sampling period and soil/soil+plant treatment, different letters indicate significant differences among soil treatments (Tukey's HSD test at $P < 0.05$). Statistics from three-way ANOVA with repeated measures (compost dose × FEP × plant) is shown in Table 2.

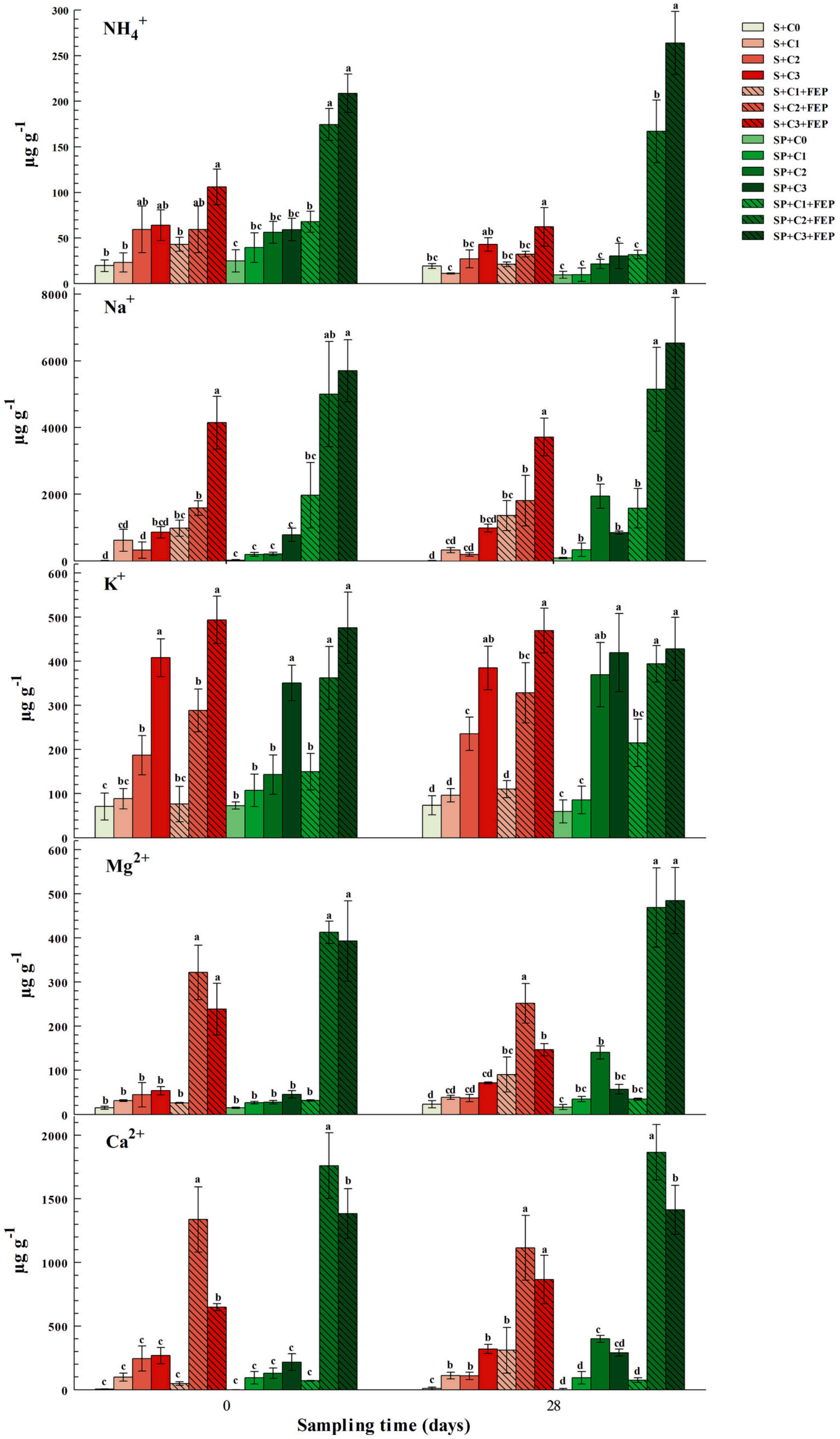

Fig. 2. Changes in water-soluble ammonium, sodium, potassium, magnesium, and calcium (mean ± SD, *n* = 3) in soil microcosms under different treatments before and after the 28-day observation period. Within each sampling period and soil/soil+plant treatment, different letters indicate significant differences among soil treatments (Tukey's HSD test at $P < 0.05$). Statistics from three-way ANOVA with repeated measures (compost dose × FEP × plant) is shown in Table 2.

Table 3

Significant effect due to compost dosage (D), exhausted FEP enrichment (FEP) and their interaction on the variability of ETR, Y(II) and NPQ of lettuce (*Lactuca sativa* L.) plants grown in soil microcosms under different treatments in a growth chamber for 28 days and monitored at four observation periods. Statistics is presented as *F*-values and level of significance (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$; *ns* $P > 0.05$) estimated by two-way ANOVA (compost dose × FEP).

Chlorophyll fluorescence parameter	Observation period (day)			
	7	14	21	28
ETR				
Dosage	*ns*	*ns*	15.20***	16.82***
FEP	*ns*	*ns*	121.82***	40.61***
D × FEP	*ns*	*ns*	20.36***	18.27***
Y(II)				
Dosage	43.86***	7.02**	*ns*	31.18***
FEP	*ns*	*ns*	16.05**	38.74***
D × FEP	*ns*	*ns*	*ns*	6.46**
NPQ				
Dosage	31.87***	3.46*	7.73**	21.90***
FEP	5.19**	*ns*	35.07***	52.76***
D × FEP	17.84***	12.80***	*ns*	9.32**

difference was appreciated among compost amended and control treatments, with or without lettuce plant. Moreover, no time-dependent variation was found between initial and final samplings. On the contrary, release of water-soluble K^+ was always directly related to the compost dosage (up to a 4.6-increase at the largest dose, on average), notwithstanding FEP-enrichment or plant presence (Fig. 2). Once again, no time-dependent variation was found between initial and final samplings.

3.3. Plant responses: chlorophyll fluorescence parameters

The F_v/Fm ratio is considered a parameter indicating the potential photochemical activity of the PSII. In lettuce plants, after 28 days, in all treatments, the F_v/Fm values remained close to the typical values for healthy plants (higher than 0.70) and similar to control plants (*data not shown*). The electron transport rate (ETR), a light-adapted parameter, remained unchanged among treatments during the first two weeks; then it increased significantly in plants treated with the two highest doses of FEP-enriched compost (Fig. 3, Table 3). YII is a parameter directly related to ETR, as it gives an indication on the linear transport of electrons, taking into account the quantity of electron acceptors available in the PSI. An initial fluctuating trend in YII was mainly due to the different compost concentrations (Table 3). After 21 days the positive effect induced by the addition of powders in soil is further confirmed by a significant increase in the YII values of plants, pointing out an increase in the overall photosynthetic process (Fig. 3, Table 3). On illumination, there is a rapid increase in the rate constant for heat dissipation of chlorophyll excitation energy, monitored using a parameter termed non-photochemical quenching (NPQ). NPQ is a photo-protective process removing excess excitation energy by conversion to heat. During the 28 days NPQ values remained significantly high both in controls and in plants grown with the sole compost addition; on the contrary, with the addition of FEP, the leaf NPQ values of plants treated with FEP-enriched compost declined at later stages of the experimental period (Fig. 3, Table 3).

3.4. Plant responses: aboveground and belowground biomass

The two-way ANOVA evidenced that plant biomass-related variables were all significantly affected by the compost dosage, FEP enrichment and their interaction (Table 4). In detail, fresh and dry weight of both lettuce shoot and roots markedly increased (up to a 2-fold increase) with increasing compost dosage, becoming more evident at the highest rate in the shoot than in the root fraction (+134 and + 100 % *vs* + 108 and 48 %, fresh and dry weight, respectively) (Table 4; Fig. S4). Moreover, when applying FEP-enriched compost the plant growth promoting effect observed in lettuce shoot and root fractions was confirmed in trend, but with a greater magnitude (as also corroborated by the significant D x FEP interaction): 10-fold and 6-fold increase *vs* 5-fold and 4-fold increase, fresh and dry weight, respectively (Table 4). Interestingly, lettuce plants treated with FEP-enriched compost showed opposite trends in their shoot (increasing) and root (declining) mass ratios (Table 4).

3.5. Plant responses: ash content

An apparently contrasting trend in the ash content was observed under the differing treatments. In brief, when considering the relative

Table 4

Fresh and dry weight, root (RMR) and shoot (SMR) mass ratios, relative and total ash content of lettuce (*Lactuca sativa* L.) plants grown in soil microcosms under different treatments in a growth chamber for 28 days[a]. Lowercase different letters in a column indicate significant differences among treatments (Tukey's HSD at $P <$ 0.05). Significant effects due to compost dosage (D), exhausted FEP addition (FEP) and their interaction are presented as *F*-values and level of significance (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$; *ns* $P > 0.05$) estimated by two-way ANOVA (compost dose × FEP).

Treatment	Fresh weight (g)		Dry weight (g)		RMR	SMR	Relative ash (mg g^{-1} dw)		Total ash (mg)	
	Root	*Shoot*	*Root*	*Shoot*			*Root*	*Shoot*	*Root*	*Shoot*
SP + C0	2.6 ± 0.5^{e}	5.8 ± 0.1^{e}	0.21 ± 0.01^{c}	0.52 ± 0.02^{c}	0.29 ± 0.01^{a}	0.71 ± 0.01^{c}	102 ± 2^{ab}	132 ± 4^{c}	21.4 ± 1.1^{d}	68.6 ± 3.3^{d}
SP + C1	3.4 ± 0.1^{e}	7.7 ± 1.9^{e}	0.28 ± 0.01^{c}	0.60 ± 0.03^{c}	0.32 ± 0.01^{a}	0.68 ± 0.01^{c}	129 ± 16^{ab}	155 ± 14^{bc}	36.1 ± 4.7^{c}	93 ± 13.0^{cd}
SP + C2	4.9 ± 0.5^{d}	13.6 ± 1.8^{de}	0.32 ± 0.05^{c}	0.86 ± 0.01^{c}	0.27 ± 0.03^{ab}	0.73 ± 0.03^{bc}	135 ± 4^{ab}	195 ± 3^{a}	43.2 ± 1.3^{c}	167.7 ± 3.2^{c}
SP + C3	5.4 ± 0.1^{d}	16.9 ± 0.9^{d}	0.31 ± 0.04^{c}	1.04 ± 0.03^{c}	0.23 ± 0.02^{b}	0.77 ± 0.02^{ab}	152 ± 38^{a}	179 ± 6^{ab}	47.1 ± 11.8^{bc}	186.2 ± 8.2^{c}
SP + C1 + FEP	8.3 ± 0.2^{c}	28.0 ± 4.7^{c}	0.61 ± 0.12^{b}	2.32 ± 0.34^{b}	0.21 ± 0.03^{bc}	0.79 ± 0.03^{ab}	93 ± 2^{ab}	138 ± 8^{c}	56.7 ± 11.2^{b}	320.2 ± 50.5^{c}
SP + C2 + FEP	10.5 ± 0.4^{b}	50.9 ± 4.4^{b}	0.83 ± 0.02^{a}	3.33 ± 0.34^{a}	0.20 ± 0.02^{c}	0.80 ± 0.02^{a}	88 ± 9^{b}	144 ± 3^{c}	73.0 ± 7.7^{a}	479.5 ± 18.7^{b}
SP + C3 + FEP	13.3 ± 0.6^{a}	60.6 ± 4.9^{a}	0.95 ± 0.13^{a}	3.89 ± 0.28^{a}	0.20 ± 0.03^{c}	0.80 ± 0.03^{a}	115 ± 18^{ab}	145 ± 7^{c}	109.2 ± 22.7^{a}	564 ± 48.9^{a}
Dosage	$F_{3.16}$ = 322.5***	$F_{3.16}$ = 143.5***	$F_{3.16}$ = 47.8****	$F_{3.16}$ = 113.8***	$F_{3.16}$ = 12.2***	$F_{3.16}$ = 12.2***	*ns*	$F_{3.16}$ = 23.7***	$F_{3.16}$ = 87.4***	$F_{3.16}$ = 55.6***
FEP	$F_{1.16}$ = 841.9***	$F_{1.16}$ = 421.1***	$F_{1.16}$ = 184.7***	$F_{1.16}$ = 483.2***	$F_{1.16}$ = 32.7***	$F_{1.16}$ = 32.7***	$F_{1.16}$ = 14.0**	$F_{1.16}$ = 56.0***	$F_{1.16}$ = 250.***	$F_{1.16}$ = 73.7***
D x FEP	$F_{3.16}$ = 109.1***	$F_{3.16}$ = 62.8***	$F_{3.16}$ = 26.0***	$F_{3.16}$ = 62.3***	$F_{3.16}$ = 6.4**	$F_{3.16}$ = 6.4**	*ns*	$F_{3.16}$ = 10.3***	$F_{3.16}$ = 33.1***	$F_{3.16}$ = 15.8**

[a] Values are mean ± SD (*n* = *3*).

Table 5

Root morphological parameters of lettuce (*Lactuca sativa* L.) plants grown in soil microcosms under different treatments in a growth chamber for 28 days[a]. Lowercase different letters in a column indicate significant differences among treatments (Tukey's HSD at $P < 0.05$). Significant effects due to compost dosage (D), exhausted FEP addition (FEP) and their interaction are presented as *F*-values and level of significance (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$; *ns* $P > 0.05$) estimated by two-way ANOVA (compost dose × FEP).

Treatment	Root length (cm)	Surface area (cm^2)	Average diameter (mm)	Volume (cm^3)	SRL[b] (cm g^{-1} d.w.)	RTD[c] (g d.w. cm^{-3})
SP + C0	3074 ± 84^{f}	257 ± 10^{g}	0.267 ± 0.004^{c}	1.72 ± 0.15^{f}	$14{,}643 \pm 70^{b}$	0.128 ± 0.003^{b}
SP + C1	3434 ± 105^{e}	283 ± 18^{f}	0.264 ± 0.004^{c}	1.87 ± 0.20^{f}	$12{,}264 \pm 54^{d}$	0.152 ± 0.010^{a}
SP + C2	4320 ± 120^{d}	368 ± 20^{e}	0.271 ± 0.003^{c}	2.50 ± 0.21^{e}	$14{,}897 \pm 42^{b}$	0.116 ± 0.006^{c}
SP + C3	5131 ± 170^{c}	448 ± 35^{d}	0.279 ± 0.004^{b}	3.12 ± 0.31^{d}	$17{,}103 \pm 76^{a}$	0.108 ± 0.004^{c}
SP + C1 + FEP	8046 ± 150^{b}	674 ± 42^{c}	0.270 ± 0.003^{c}	4.54 ± 0.35^{c}	$13{,}872 \pm 85^{c}$	0.128 ± 0.004^{b}
SP + C2 + FEP	9789 ± 165^{a}	859 ± 38^{b}	0.281 ± 0.004^{a}	6.00 ± 0.10^{b}	$11{,}794 \pm 38^{e}$	0.137 ± 0.002^{a}
SP + C3 + FEP	9990 ± 140^{a}	950 ± 35^{a}	0.288 ± 0.003^{a}	6.40 ± 0.20^{a}	$11{,}100 \pm 44^{f}$	0.141 ± 0.005^{a}
Dosage	$F_{3.16} = 1404.7$***	$F_{3.16} = 270.8$***	$F_{3.16} = 28.5$***	$F_{3.16} = 216.7$***	$F_{3.16} = 801.9$***	$F_{3.16} = 12.1$**
FEP	$F_{1.16} = 4860.6$***	$F_{1.16} = 873.0$***	$F_{1.16} = 17.5$**	$F_{1.16} = 676.7$***	$F_{1.16} = 5464.1$***	$F_{1.16} = 14.8$**
D x FEP	$F_{3.16} = 551.4$***	$F_{3.16} = 101.6$***	$F_{3.16} = 1.7^{ns}$	$F_{3.16} = 78.9$***	$F_{3.16} = 4431.3$***	$F_{3.16} = 38.2$***

[a] Values are mean ± SD (*n* = *3*).

[b] Specific root length (root length per unit of root dry weight).

[c] Root tissue density (root dry mass per unit root volume).

Table 6

Significant effects due to compost dosage (D), exhausted FEP enrichment (FEP) and their interaction on the variability of root and shoot concentration of major inorganic anions (plus malate) and cations shown in Fig. 4. Statistics is presented as *F*-values and level of significance (* $P < 0.05$; ** $P < 0.01$; *** $P < 0.001$; *ns* $P > 0.05$) estimated by two-way ANOVA (compost dose × FEP).

Main effect		Anions									
		Root					Shoot				
	df[a]	NO_3^-	Cl^-	$H_2PO_4^-$	SO_4^{2-}	Malate	NO_3^-	Cl^-	$H_2PO_4^-$	SO_4^{2-}	Malate
Dosage	3	0.06^{ns}	1.41^{ns}	12.28**	14.60**	11.88**	1.92^{ns}	22.39***	123.99***	0.32^{ns}	3.73^{ns}
FEP	1	1.07^{ns}	15.40**	0.27^{ns}	11.47**	45.25***	0.01^{ns}	75.59***	12.20**	0.82^{ns}	1.60^{ns}
D x FEP	3	1.40^{ns}	2.12^{ns}	3.42^{ns}	4.46*	9.21**	0.11^{ns}	14.50**	1.50^{ns}	0.59^{ns}	2.63^{ns}

		Cations							
		Root				Shoot			
	df	Na^+	K^+	Mg^{2+}	Ca^{2+}	Na^+	K^+	Mg^{2+}	Ca^{2+}
Dosage	3	29.12***	34.76***	3.88^{ns}	4.13*	2.06^{ns}	62.06***	0.77^{ns}	0.98^{ns}
FEP	1	2.08^{ns}	295.69***	0.73^{ns}	0.14^{ns}	1.34^{ns}	29.49**	0.33^{ns}	1.96^{ns}
D x FEP	3	0.42^{ns}	37.39***	0.31^{ns}	0.23^{ns}	0.45^{ns}	7.80**	1.02^{ns}	1.02^{ns}

[a] Degrees of freedom.

ash content, it was found to increase at increasing compost dosage especially in the shoot portion. Whereas in lettuce plant treated with FEP-enriched compost it was found to be lower than in the sole compost treatments, either in the root or in the shoot system, with no statistically difference respect to the non-amended control (Table 4). On the contrary, when considering the total ash content, calculated in relation to the total root or root dry weight biomass, it appeared a clear steadily increasing content both in the root and in the root systems, resulting even greater in the treatments with the FEP-enriched compost (Table 4).

3.6. Plant responses: root morphology

The three-way ANOVA evidenced that root morphological parameters were all significantly affected by the compost dosage, exhausted FEP enrichment and their interaction (Table 5). In details, total root length, surface area, volume and average diameter markedly went up with the compost dosage resulting in an ~70 % increase (on average) at the highest addition rate (Table 5; Fig. S4). Noteworthy, these morphological parameters were further increased (up to a 2.7-fold the non-amended control value) when lettuce plants were exposed to FEP-enriched compost. The SRL ratios increased in lettuce plants grown with compost and decreased with FEP-enriched compost. SRL parameter is also considered a root fineness index as it is inversely proportional to the square of the root diameter (Ryser, 2006). Values of the root tissue density (RTD), which is considered a structural parameter, were higher in treatments with exhausted FEP-enriched compost compared to those with the compost alone (Table 5).

3.7. Plant responses: anions content

Larger amounts of anionic compounds were found in the root rather than in the shoot system, with selective quantitative responses to the treatments depending on the chemical species (Fig. 4). To begin with, nitrate concentration in lettuce plant tissues remained practically unaffected by compost addition, either with or without FEP-enrichment, maintaining values similar to that of the control plants both in the shoot (ranging between 0.7 and 1.3 mg g^{-1} dw) and in the root system (3.7–5.6 mg g^{-1} dw range variation) (Fig. 4; Table 6). On the contrary, an increasing dosage of compost brought about a steadily increasing concentration of Cl^- in lettuce leaves (up to a 2.6-fold increase at the highest dosage), but not in the root system where it remained statistically unchanged. Moreover, in microcosms amended with exhausted FEP-enriched compost, a reduced amount of Cl^- was found in both shoot and root systems with no significant differences among treatments (Fig. 4; Table 6). Similarly, compost addition, but not the combined FEP-enrichment, promoted the plant uptake of $H_2PO_4^-$ which increased

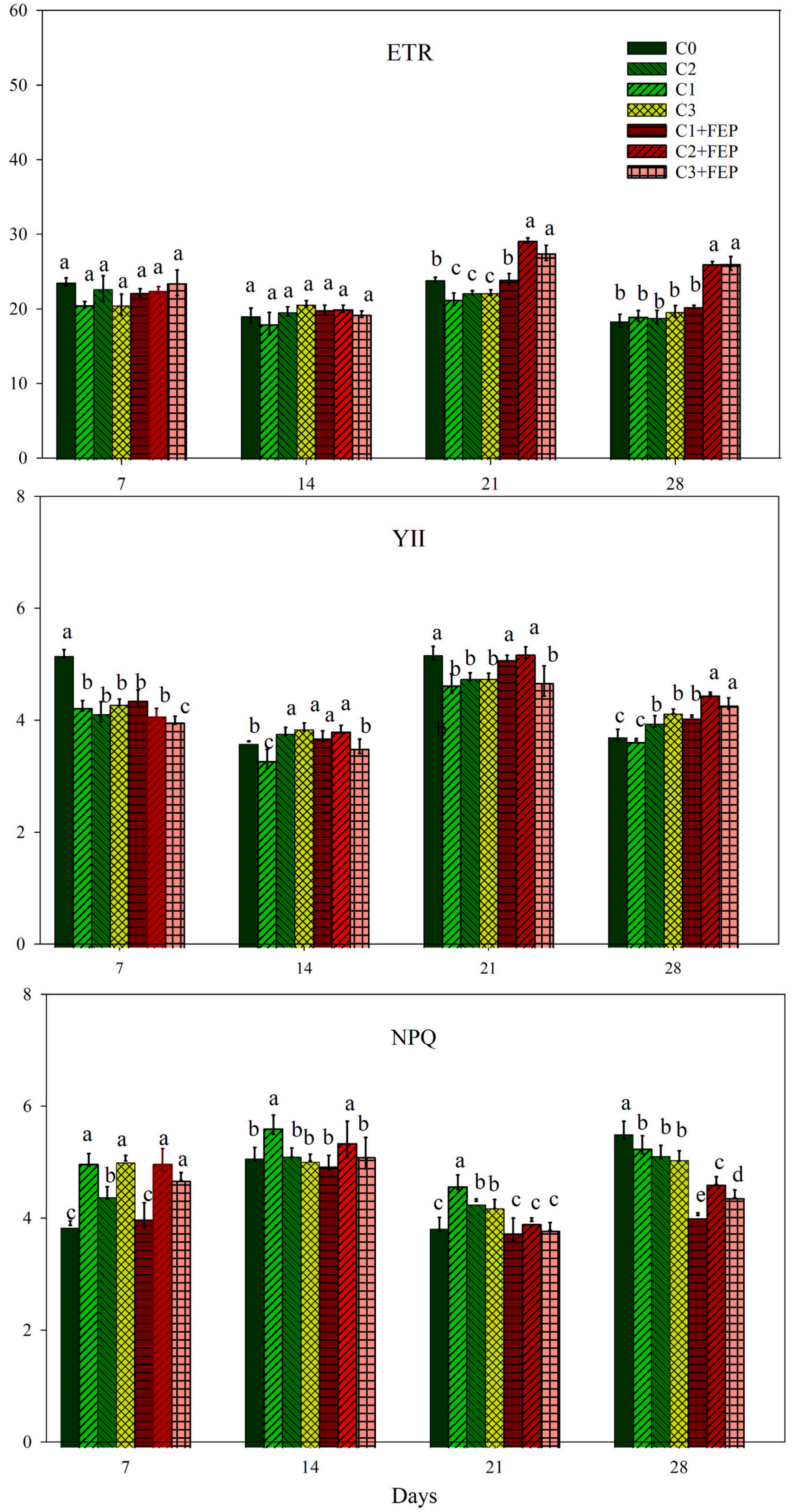

Fig. 3. Electron transport rate (ETR), effective quantum efficiency Y(II)), and quantum yield of light-induced non photochemical quenching (NPQ) in lettuce (*Lactuca sativa* L.) plants grown in soil microcosms under different treatments in a growth chamber for 28 days. Chlorophyll fluorescence parameters were estimated weekly after 7, 14, 21 and 28 days from transplanting. Within each sampling period, different letters indicate significant differences among soil treatments (Tukey's HSD test at $P < 0.05$). Statistics from two-way ANOVA (compost dose × FEP) is shown in Table 3.

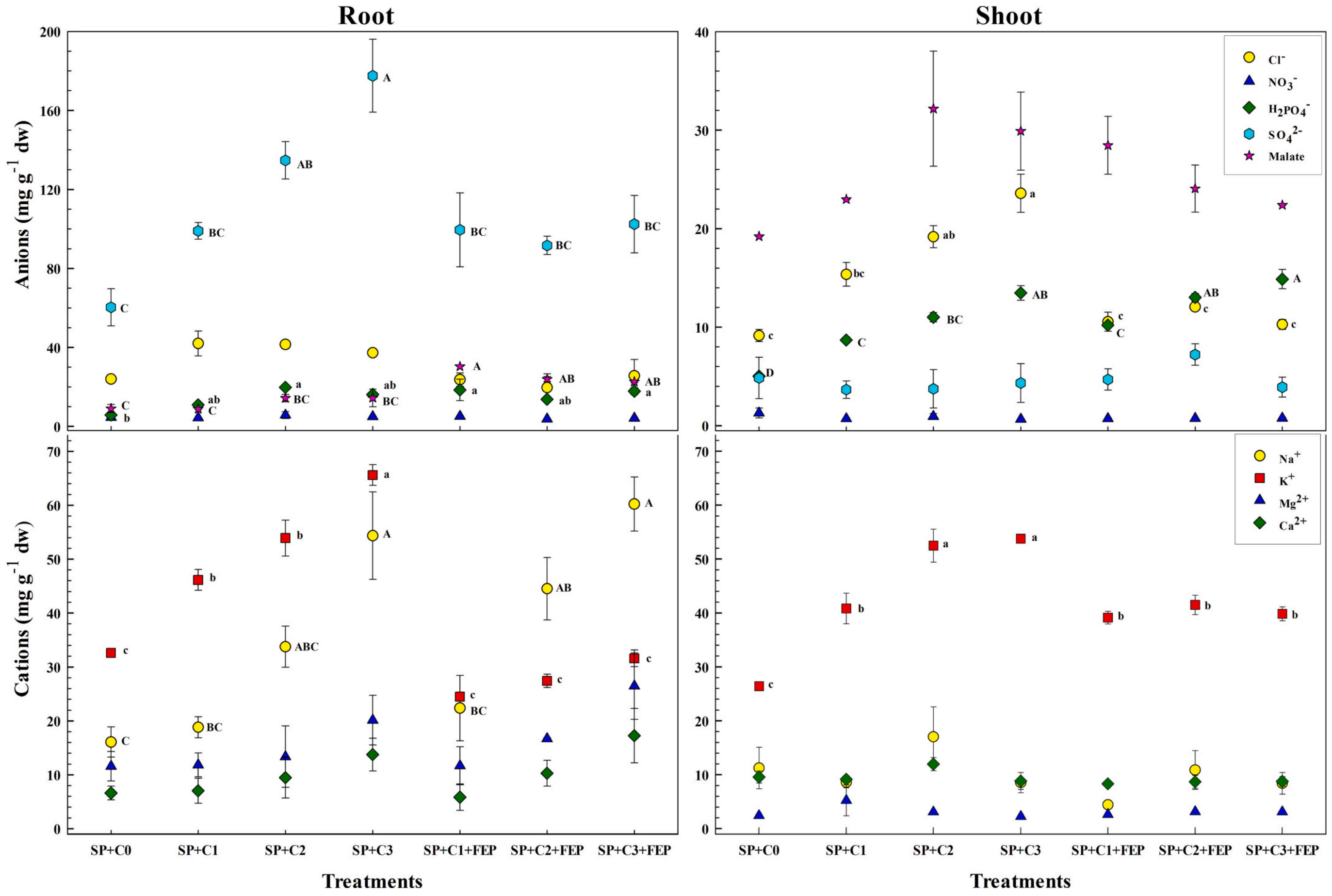

Fig. 4. Total content of inorganic cations (sodium, potassium, magnesium, calcium) and anions (chloride, nitrate, phosphate, sulphate) (mean ± SD, $n = 3$) in root and shoot tissues of lettuce (*Lactuca sativa* L.) plants grown in soil microcosms under different treatments in a growth chamber for 28 days. Within each plant portion and ionic species, different letters indicate significant differences among soil treatments (Tukey's HSD test at $P < 0.05$). Statistics from two-way ANOVA (compost dose × FEP) is shown in Table 6.

significantly in the root system, and then was rapidly translocated towards the aerial portion (Fig. 4; Table 6). A similar sole compost-driven effect was observed for SO_4^{2-} uptake but, unlike phosphate, sulphate was mainly accumulated in root tissues. Differently, the amount of detected malate did not significantly vary among treatments in lettuce shoot; whereas it increased significantly in the roots of plants grown in microcosms added with compost (+38 %, on average) and especially with exhausted FEP-enriched compost (+183 %, on average) (Fig. 4; Table 6).

In brief, the addition of increasing dosages of compost increased the adsorption of sulphate (which remained stored in the root system), and chloride and phosphate (which were translocated towards the shoot system) (Supplementary Table 1). On the contrary, absorption (and root storage) of sulphate and chloride declined in treatments with the exhausted FEP-enriched compost, thus determining a significant lower amount of negatively charged equivalents entering the root system (Fig. 4; Table S1).

3.8. *Plant responses: cations content*

Concentrations of cations found in the root and in the shoot systems were not as different as previously seen for anions. However, selective quantitative responses to the treatments were also observed. Which were linked to the chemical species considered (Fig. 4). Addition of compost in the microcosm soil stimulated the Na^+ uptake and hence its accumulation in lettuce plant tissues, particularly root tissues (up to 60 mg g^{-1} dw corresponding to a ca 3.6-fold increase at the highest dose, on average), with a compost dose-dependent trend irrespective of the FEP-enrichment (Fig. 4; Table 6). This was rather unexpected since most Na^+ release was from FEP-enriched compost, suggesting that compost was the main driver of sodium adsorption. Conversely, the Na^+ concentration in the shoot remained to low control value, slightly ranging between 4.4 and 17.0 mg g^{-1} dw, with no significant differences among treatments. This means that preferential root accumulation of excess Na^+ prevented the cation from aboveground translocation, thus avoiding the onset of phytotoxic responses in aerial tissues. On the contrary, selective K^+ adsorption and translocation were observed in lettuce plants depending on either the compost dosage or the FEP-enrichment. Although compost addition induced a marked dose-dependent release of K^+ in the microcosm soil (as seen before), increased K^+ uptake and its accumulation in root tissues took place only in compost treated plants, where stored potassium increased from 32.6 to 65.6 mg g^{-1} dw, positively related to the compost application rate. It is also worth noting that adsorbed K^+ was equally shared between shoot and root tissues (Fig. 4). Noteworthy, lower but not significantly different values respect to the untreated control (32.6 mg g^{-1} dw) were found in plant roots exposed to increasing dosage of FEP-enriched compost (values varied between 24.6 and 31.6 mg g^{-1} dw) (Fig. 4). In this case, K^+ concentration in lettuce leaf tissues equally increased to 41.2 mg g^{-1} dw, on average, a value higher than the reference one (26.4 mg g^{-1} dw). Differently from monovalent cations, Ca^{2+} showed a somewhat increasing trend (from 6.6 to 15.5 mg g^{-1} at the highest dose, on average), albeit not significant, in plant root tissues, which appeared positively and primarily related to the compost dose, but not to the FEP-enrichment (Fig. 3; Table 4). Whereas Mg^{2+} concentration in lettuce plant tissues remained practically unaffected by the compost addition, either with or without FEP-enrichment, and again only slight, non-significant increases due to the compost dose were observed in the root system (11.6–26.4 mg g^{-1} dw range variation). Similarly, their concentration in the shoot system remained unchanged respect to control value, slightly ranging between 8.3 and 11.8 mg g^{-1} dw (Ca^{2+}), and between 2.2 and 5.2 mg g^{-1} dw (Mg^{2+}), with no significant differences among the treatments (Fig. 4; Table 6).

To sum up, the addition of increasing dosages of compost stimulated the adsorption (and root storage) of increasing amounts of base cations, especially monovalent species (Table S2). Interestingly, addition of exhausted FEP-enriched compost maintained this trend, but strongly altered the flow of monovalent cations entering the root system, as evidenced by the marked drop of the K/Na molar ratio and unchanged K/Mg, K/Ca and Mg/Ca molar ratios (Fig. 4; Table S2).

4. Discussion

The present study reports on lettuce plants grown in enclosed microcosm systems and provides distinctive responses to compost addition, either enriched or not with exhausted fire-extinguishing powders. It is worth saying that the observed beneficial effects of composted materials on promoting nutrient accumulation, plant growth and the soil fertility status corroborate what previously observed either at field or nursery crops scale (Diacono and Montemurro, 2010; Raviv, 2011; Maruthi Sridhar et al., 2014; Chen et al., 2018; Singh et al., 2022). Then, a major emphasis will be given to main findings observed when enclosed soil plant systems were treated with exhausted FEP-enriched compost.

4.1. Soil responses to amendment with FEP-enriched compost

Compost addition (either with or without exhausted FEP enrichment) significantly varied major soil chemical properties, which showed an immediate and marked compost dose-dependent increase, whose persistence was significantly affected by the plant occurrence. In fact, in planted microcosms TOC and TN values (which are known to suddenly rise following compost amendments; Tambone et al., 2007) declined with elapsing time to final values not statistically different among treatments. To explain this finding, we hypothesize that the plant root growth and activity (in terms of spatial modification of soil fabric, nutrient uptake and rhizosphere interactions) created favourable physical and biological conditions for an enhanced mineralization of C substrates. This can also help explain the marked release over time of compost-derived soluble nutrients, which were not equally balanced by the plant removal as observed in bare microcosms. Moreover, addition of an alkaline organic amendment brought about an increase of pH which resulted even greater when using a growing mixture enriched with quartz sand, an inorganic substrate with a typical low buffering capacity. Nonetheless high salinity and pH of composted materials can represent influential conditions that negatively affect both plant growth and productivity performance (Raviv, 2013). However, this was not the case likely due to (i) the use of a controlled dosage (5 %) of exhausted FEPs for compost enrichment preserved the substrate EC from exceeding the yield-limiting level for many horticultural plants of 2 dS m^{-1} (Gómez-Brandón et al., 2016; Siles-Castellano et al., 2020), especially the more susceptible lettuce (Costa et al., 2021); (ii) the relatively high content of quartz sand in the growing medium (~50 % by volume) which caused a dilution effect thus preventing from excess rise of growing substrate salinity.

Further inside, both compost and exhausted fire-extinguishing powders concurrently contributed to release soluble nutrients as water-soluble anionic and cationic inorganic forms. We found that compost represented the main source of soluble K and N-forms, thus corroborating what reported by several authors (Raviv, 2013; Kutu and Masowa, 2018; Dey et al., 2019). Whereas the phosphate-rock derived inner mineral core of the exhausted fire extinguishing powder massively contributed to the addition not only of phosphates (and chemical treatment-derived ammonium sulphates), but also of its naturally co-occurring alkali metals such as Ca, Mg and Na. In fact, differing from igneous rocks, phosphate rock deposits of sedimentary origin, which are especially exploited for commercial world phosphate production being often higher in grade and thus representing ~80 % of the globally mined P, contain varieties of carbonate-fluorapatite with significant quantities of Ca, Mg and Na (van Kauwenbergh, 2010). To note that, under some circumstances, a huge release of unbalanced amounts of base cations into the soil can negatively interfere with the plant nutrient uptake, as discussed below.

4.2. Plant responses to amendment with FEP-enriched compost

Plant biomass readings provided clear evidence of (i) lack of any phytotoxic effect, (ii) selective growth promotion of different plant organs, (iii) changes in root morphology and (iv) nutrient uptake. As for the first point, it is worth reminding that, according to national (legislative decree n. 75/2010) and European regulations (Regulation EU n. 2019/1009), the assessment of lack of phytotoxicity represents a mandatory procedure before proceeding with agricultural use of residual biomass and composted biosolids (*i.e.*, biogas digestates, agro-industrial wastes, sludge, *etc.*). Several ecotoxicological assays were developed and improved during last decades (Kapanen and Itävaara, 2001) and nowadays constitute widely accepted and validated methods for testing the potential toxicity of composted materials before agricultural or environmental applications. These methods are primarily based on seed germination, root elongation and seedling growth of terrestrial plants, including lettuce (ISO, 2012; Lyu et al., 2018). It must be observed that the only evidence of ecotoxicology responses to fire-extinguishing power exposition has been provided by Loboichenko et al. (2019), who reported a negative impact on aquatic ecosystems of dry powders of classes A, B, C and D, due to the release of phosphate-ammonium species from the inner mineral particle core. Interestingly, no phytotoxic response was observed when using a mature compost from the organic fraction of municipal solid waste enriched with exhausted FEPs. In fact, a marked dose-related increase of both the aboveground and belowground lettuce biomass was observed, thus clearly evidencing the lack of any phytotoxic effect, while proving a strong plant growth promoting action at either shoot or root system level. Since a different biomass allocation between the root and the shoot apparatus was found, as clearly evidenced by changes in the root (and shoot) mass ratio, we hypothesize that two main processes were acting simultaneously: (i) internal nutrient resources were redirected aboveground towards the plant leaf system in order to adequately support a higher photosynthetic activity and larger biomass production; (ii) the increased availability of essential soluble nutrients released from FEP-compost altered the morphological parameters of the root system and prevented it from further expanding.

This former point is also confirmed by the chlorophyll fluorescence measurements. In fact, these measures are currently considered particularly useful non-destructive strategies both in continuous monitoring the beneficial effects of various treatments on growth and physiological activity of plants, and in the early assessment of plant responses to most types of stresses. The fate of absorbed light energy is to ultimately power photochemistry, which generates chemical energy in the form of ATP and NADPH. Light energy in excess is dissipated as heat. Some of the light that is neither used in photochemistry nor dissipated as heat is emitted as fluorescence (Malnoë, 2023). Chlorophyll fluorescence is thus a good proxy to assay these processes (Baker, 2008). The increase in parameters related to the photochemical quenching (Fv/Fm ratio, YII and ETR) observed in lettuce plants interfered with the compost enriched with exhausted FEPs clearly indicate an increase in the photosynthetic efficiency of the plant. This finding is consistent with the observed decline of the non-photochemical component of the fluorescence quenching (NPQ), thus suggesting that PSII core inactivation due to the possible damage of the photosynthetic reaction-center, mediated by reactive oxygen species (ROS), is strongly reduced if compared to all other treatments. Needless to say, an Fv/Fm value higher than 0.70, as observed in the study, constitutes an indication of a healthy plant (Murchie and Lawson, 2013). These findings agree with Qiu et al. (2020), who reported a compost dose-dependent positive response on chlorophyll fluorescence indices and potential photosynthetic capacity, which in turn can influence carbon assimilation and photoassimilate accumulation. These plant biochemical processes became clearly visible in our microcosms study where a larger plant growth and total dry biomass production were found and determined a dilution effect of the large amount of absorbed nutrients, resulting in opposite trends between relative and absolute ash content.

It must also be observed that significant changes were noticed in the structural parameters of the root system of lettuce plants exposed to the exhausted FEP-enriched compost (*i.e.* in the specific root length and tissue density). Precisely, lettuce plants grown on soil amended with not-enriched compost produced longer and finer roots, with a higher specific surface and a reduced tissue density, in order to enhance water and nutrient uptake from a larger soil volume. Conversely, the root system of plants treated with FEP-enriched compost was characterized by roots shorter in length, larger in diameter and higher in tissue density. These morphological features are better suited with a fast inner transport when water and nutrients are largely available in soil (van der Bom et al., 2020). Taken together these findings corroborate what is largely known: the plant root system shows a surprisingly high degree of plasticity in its development, and this makes it able to efficiently and rapidly respond to changing external conditions, and help the plant exploit belowground resources (namely nutrient and water) from that "…heterogeneous, variable and porous system…" which the soil environment is (Ryser, 2006).

Imbalanced release of huge amounts of water-soluble anionic and cationic inorganic forms derived from compost and, at major extent, from the compost enriched with the exhausted fire-extinguishing powder markedly affected the process of mineral nutrient uptake in lettuce plants, in a way that promoted selective accumulation or antagonistic influx of cationic and anionic species. This was particularly true for alkali metals such as Na and K, whose content in root tissues changed dramatically and an altered K/Na ratio was found - a result in line with what observed by Bie et al. (2004) in lettuce plants grown under increasing Na provision. However, detrimental effects on plant growth and yield were not appreciated. To explain this several concurring factors may be invoked. Magnesium and calcium influx was reduced to mitigate charge perturbations and maintain structural and functional integrity of the plant membrane. Moreover, absorbed K^+ was preferentially translocated towards the shoot system to help the plant tolerate the Na^+ excess. As for the K/Na ratio, Kronzucker et al. (2013) went on to show that unless K^+ is completely deprived, high Na^+ cytosolic concentrations are rarely primary causes of toxicity, and great caution should be used before considering the K/Na ratio as the main proxy of toxicity. We also noticed a raised amount of malate in root tissues of plants treated with exhausted FEP-enriched compost. It is known that imbalanced influx of ions triggers a biochemical pH-stat mechanism including carboxylation and decarboxylation of organic acids like malate, with the following release of H^+, or OH^- (HCO_3^-). In fact, malate is considered a versatile compound in plant metabolism, which can easily be transported across subcellular membranes and plays a pivotal role as a regulator of pH homeostasis (Maurino and Engqvist, 2015). Cytosolic accumulation of malate is therefore a key cell strategy for buffering pH variations and counterbalancing the variable accumulation of ions in the cytosol, thus maintaining ion transport and osmotic regulation (Sakano, 2001; Gaxiola et al., 2007). The close relationship between malate accumulation and excess cationic species (precisely potassium) becomes evident in shoot tissues, thus corroborating the role of organic acids as indicators of plant tolerance to stress (El-Nakhel et al., 2020a). It has been also shown that modulating the available amount of macrocations, especially Mg in relation to Ca and K, lettuce plants increase cytosolic concentrations of bioactive compounds and reduce anti-nutritional components such as nitrate (El-Nakhel et al., 2020b). In accordance with Silber (2019) ionic interrelationships were observed among potassium and phosphate, sulphate and chloride uptake. Being mobile within plant tissues, phosphate and chloride were translocated towards the shoot system; whereas the scarcely mobile sulphate remained stored in root tissues.

5. Conclusions

Exhausted fire-extinguishing powders (FEPs) represents a still poorly valorised industrial solid waste which is constituted by mine-derived phosphorous industrially added with ammonium sulphate during the manufacturing process. At expiration of their service time (36 months), exhausted FEPs must be treated and disposed as an industrial waste due to the silicon-based external coating additives, which make problematic the recovery of the inner salt mixture for use in agriculture as a source of plant essential nutrient elements. This study shows that biological processes acting during the composting process help lyse the water repellent external coating, thus making immediately accessible the inner mineral core of the powder represented by inorganic N-, P-, and S-containing species together with a minor content of rock-derived Ca, Mg and Na. Findings from a microcosm study with lettuce plants grown in soilless cultivation under complete replacement of mineral nutrients with mature compost enriched (5 %, by weight) with exhausted FEPs showed absence of phytotoxic responses, balanced release of nutrients, photosynthetic efficiency increase, plant growth promotion, and adaptive responses in root morphology to facilitate water and nutrients. According to circular economy principles, composting represents a solid waste management measure that can significantly contribute to recycle energy and nutrients from a wide range of raw materials. Within the context, key findings from the present study show the feasibility of both agricultural and industrial wastes to be valorised into added-value products for use as a inorganic fertilizers and soil conditioner use in soilless cultivation. These results are of great interest in search and development of eco-friendly waste-to-resource technologies for non-renewable resource management and sustainable agricultural production (Razza et al., 2018).

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CRediT authorship contribution statement

Antonio Gelsomino: Conceptualization, Methodology, Investigation, Resources, Data curation, Formal analysis, Writing – review & editing. **Beatrix Petrovičovà:** Methodology, Investigation, Formal analysis. **Maria Rosaria Panuccio:** Conceptualization, Methodology, Resources, Formal analysis, Writing – original draft, Supervision, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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Appendix A. Supplementary data

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