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Fly Bioash Ameliorates Acid Luvisol and Increases Sunflower (*Helianthus annuus* L.) Yield in Field Conditions without Compromising the Risk of Radioactive Contamination

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Abstract: Fly bioash (FBA) as a by-product of biomass-fuelled facilities exhibits alkaline properties and is enriched with phytonutrients, thereby offering the potential to effectively ameliorate acidic and nutrient-deficient soils. However, concerns about health risks due to a potential FBA radioactive contamination are still not well studied, notably under field conditions. This study examined pH changes and concentrations of natural (²³⁸U, ²³²Th, ²²⁶Ra, ⁴⁰K) and anthropogenic (¹³⁷Cs) radionuclides after application of very alkaline (pH > 12) FBA in: (i) highly acid (pH_{KCl} = 4.1) Luvisol and (ii) sunflower (*Helianthus annuus* L.) seeds, grown in organic farming and rain-fed conditions. FBA (originated from a modern cogeneration, fuelled on certified deciduous forest wood chips) was applied at increasing doses; 0, 4.5, 8.6, 13, and 17.2 t/ha. After 54 months of application, FBA significantly increased soil pH_{KCl} by up to 1.8 unit and the seed yield by 15%, compared with no amended Control, without compromising soil electrical conductivity (salinity). The activity concentrations (A_c) of all observed radionuclides, measured using high-resolution gamma-ray spectrometry, were not altered under FBA application, neither in the surface (0–30 cm) Luvisol horizon nor in the sunflower seed. Moreover, the A_c of ²³⁸U, ²³²Th, and ¹³⁷Cs in the seed were below detection limit, whereas the A_c of ⁴⁰K and ²²⁶Ra were lower by up to 2.6 and 61 times, respectively, than their corresponding A_c in the soil treatments. The radiological footprint of FBA exhibited lower A_c for most of the observed radionuclides compared with both (i) Croatian non-arable topsoils (with reductions of ²³⁸U 3.6 times, ²³²Th 1.8 times, ²²⁶Ra 1.7 times, and ¹³⁷Cs 1.5 times) and (ii) widely used mineral N/P/K fertilisers in conventional agroecosystems (with reductions of ²³⁸U 12.5 times; ²²⁶Ra 1.3 times, and ⁴⁰K 2.4 times). Our findings provide evidence that the application of FBA as a soil conditioner does not pose radiological health or environmental risks, contributing to more sustainable agri-food production and circular bioeconomy. However, it is essential to conduct further studies to comprehensively investigate the effects of FBA application on soil and crop quality across diverse environmental conditions and extended spatiotemporal scales.

Keywords: bioash; acid agricultural soil; radioactivity; stable radionuclides; soil-to-plant transfer factor

1. Introduction

Acid soils, characterised by a pH reaction below 6.5, pose a significant obstacle to global agricultural productivity and efficient land management. Around one-third of global

soils and even up to 50% of potentially arable lands exhibit soil acidity [1]. Sustainable management of acid soils is vital for achieving optimal crop yields and environmental sustainability. Generally, acid soils in the surface horizon are typically characterised by deficiencies in exchangeable basic cations (Ca^{2+} , Mg^{2+} , K^+) and the prevalence of phytotoxic ions (Al^{3+} or H^+) at colloidal exchange sites (CECs), hindering nutrient availability [1] and causing many other constraints (heavy texture, poor water permeability) related to acid soils [2]. Consequently, the development of effective and more sustainable soil amelioration strategies (e.g., recycling of potentially valuable waste streams) is an imperative to enhance soil fertility and maximise crop performance in acid soils. In such a context, fly bioash (FBA) presents one of the possible transformative solutions.

FBA is a complex by-product generated from biomass-fuelled plant facilities. It exhibits alkaline properties and is enriched with phytonutrients, making it a promising candidate for effective amelioration of acidic and/or nutrient-deficient soils [3,4]. Some of the recent studies have demonstrated the positive effects of bioash application on pH and nutrient recovery [3,4], as well as some other soil constraints [5,6]. The strong alkaline pH reaction (>12) of bioash percolates, attributed to its dominant basic minerals (NaOH , KOH , CaCO_3 , $\text{Ca}(\text{HCO}_3)_2$, CaCO_3), can effectively displace exchangeable H^+ , Al^{3+} , and Mn^{2+} from the soil's CEC under acidic pedo-conditions [7–9]. Consequently, bioashes exhibit very efficient neutralising properties in various acid soil types, enhancing the availability of macro and micronutrients. Additionally, certain forms of bioash, such as finely powdered FBA, have shown superior pH recovery and higher acid-neutralising capacity compared to some commonly used liming materials such as limestone and/or dolomite fractions [4,7,10]. This is attributed to a highly developed and reactive FBA matrix, as well as the prevalence of more reactive alkaline hydroxide fractions [6]. Thus, the application of FBA as a soil ameliorant offers a promising solution to address challenges related to acid soils. Furthermore, the utilisation of FBA as a soil amendment aligns with the principles of circular bioeconomy and waste valorisation. By transforming FBA, the agricultural sector can contribute to reducing waste generation and promoting a more sustainable use of increasing waste streams [6], thereby fostering more resilient and ecologically balanced (agro)ecosystems.

However, the knowledge related to the relatively new FBA matrix from modern cogeneration plant facilities and its amelioration potential in acidic Luvisol, notably to soil pH, electrical conductivity (EC), and crop growth and yield performances, has not been systematically investigated in field conditions. In addition, the potential health risks associated with FBA's radioactive contamination have raised concerns among researchers and practitioners. Namely, the precursor of FBA, its forest/agricultural biomass, is recognised for its comprehensive composition, encompassing naturally occurring and anthropogenic elements, including ultra-rare isotopes and radionuclides (e.g., ^{238}U , ^{232}Th , ^{226}Ra , ^{210}Pb , ^{40}K , ^{137}Cs , ^{208}Tl) [11,12], which can be absorbed by the root system or deposited on aboveground tissues [6]. Thus, it is crucial to thoroughly investigate and understand the radioactivity levels in FBA and its implications for soil and crop quality.

In this study, we aimed to examine pH changes and concentrations of radionuclides (^{238}U , ^{232}Th , ^{226}Ra , ^{40}K , ^{37}Cs) 54 months after the application of very alkaline FBA in: (i) highly acidic ($\text{pH}_{\text{KCl}} = 4.1$) Luvisol and (ii) sunflower (*Helianthus annuus* L.) seeds. The findings of the study will provide important insights into the viability of using FBA as a soil amendment and contribute to the assessment of the environmental and health risks associated with FBA application in agroecosystems affected by acid soils.

2. Material & Methods

2.1. Study Site

The experimental site was placed in the Đakovština area, Eastern Croatia ($45^{\circ}24'13''$ N; $18^{\circ}29'06''$ E) (Figure 1a), characterised by an intensive organic agricultural production on different types of native acidic soils. The study area belongs to the Pannonian region, covered by Quaternary sediments, where river-marsh sediments predominate, with subor-

minated alluvial–proluvial deposits, aeolian sands, loesses, and deluvial–proluvial deposits as well. The study location belongs to the Đakovo–Vinkovci–Vukovar loess plateau, with sandy–clayey silt mechanical composition [13]. According to Köppen’s classification, the climate in the study area (Cfb) is temperate humid with warm summers and cold winters [14]. The annual average air temperature (11.1 °C) ranges between -0.6 (January) to 21.7 °C (July), the average precipitation is 694 mm, with the average reference evapotranspiration (ET_0) of 750 mm. Before conducting the main field experiment, a preliminary pre-screening soil survey was carried out in a wider area. A total of 32 topsoil samples (0–30 cm) were collected from different locations, as illustrated in Figure 1a. Among these locations, the site with the lowest pH, as depicted in Figure 1b, was selected for a comprehensive soil survey by opening the pedological soil profile and collecting both disturbed and undisturbed soil samples for detailed physicochemical analyses, following the guidelines outlined by [15].

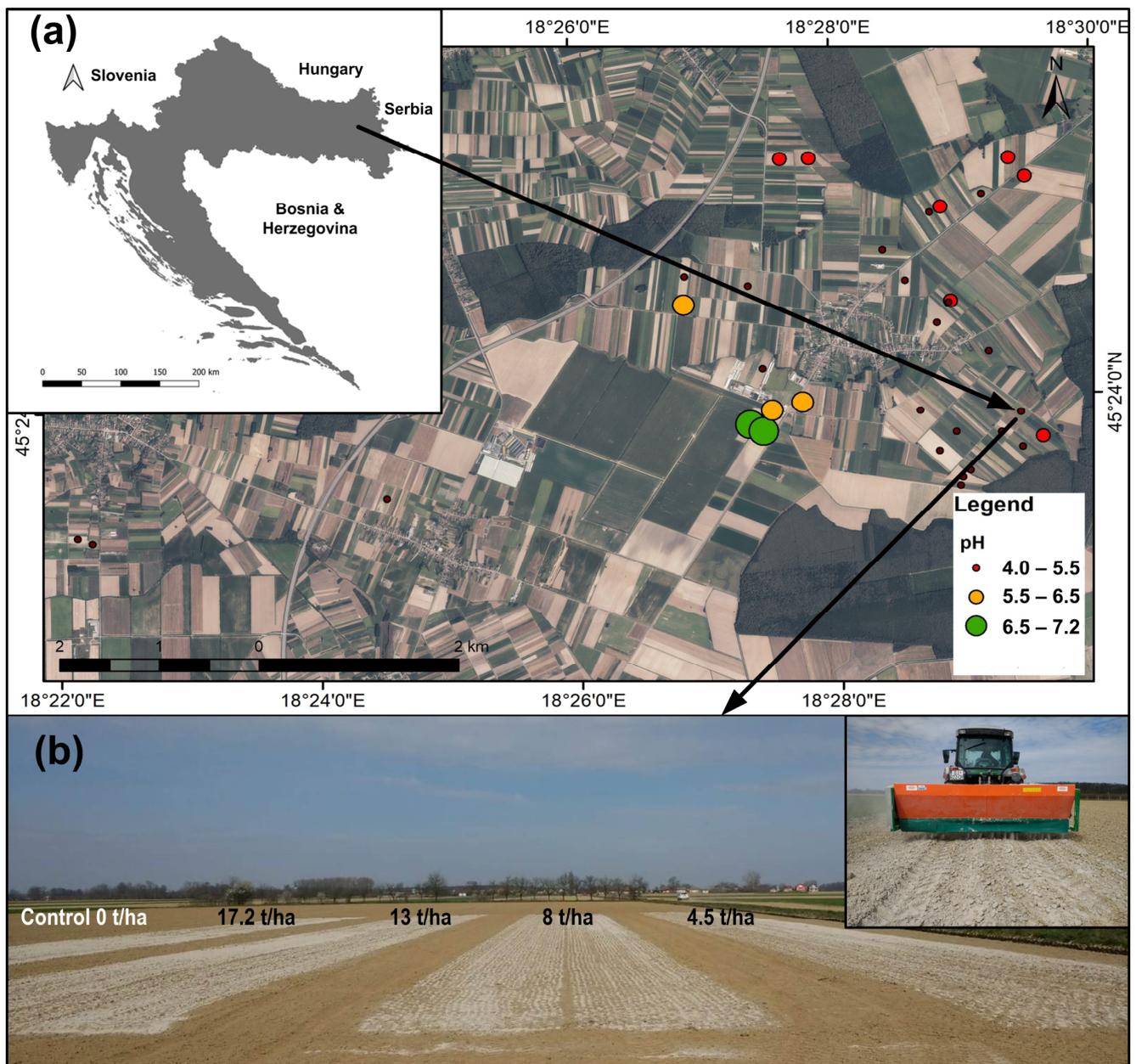


Figure 1. Study area overview with soil pH pre-screening locations (a) and fly bioash (FBA) application at the experimental site (b).

2.2. Experimental Setup & Sampling

The FBA was provided from the biomass cogeneration plant facility Uni Viridas, B. Greda, Croatia (45°11'09" N; 18°51'17" E), operating with certified wood chips of deciduous trees, specifically oak, hornbeam, and ash, accounting for approximately 85%, 10%, and 5%, respectively. The FBA was applied in five treatments at increasing doses; 0, 4.5, 8.6, 13, and 17.2 t/ha in April 2021 by an applicator for dusty materials, following topsoil incorporation by seeder (Figure 1b). Applied treatments were organised according to a randomised block design in triplicate totalling 15 experimental plots sized 6 × 50 m² (Figure 1b). For the next 54 months, during two vegetation seasons, uniform agricultural practices were performed on the experimental site using standard agro-technical operations valid for organic farming in rain-fed conditions [16]. In the second vegetation season, at the stage of technological and physiological maturity (on 25th of August 2022), sampling of the test crop and soil was performed from the experimental plots. A test crop sunflower (*Helianthus annuus* L., Hybrid NK Neoma, Syngenta, planted with a spacing of 22 cm × 70 cm per plant) was sampled manually, extracting all plants from five randomly selected sub-plots from each plot. Then, all seeds were separated manually from the heads, cleaned, and weighed fresh and dried (at 70 °C for 48 h), then stored for chemical and radiological analyses. Immediately after the harvest, soil samples were collected from the adjacent root zone for chemical (0–30 cm depth) and radiological analyses (0–10 cm, 10–20 cm, and 20–30 cm depths), air-dried, sieved through a 2 mm mesh, homogenised, and stored.

Table 1. Physicochemical composition of applied fly bioash (FBA) (Means ± Standard Error).

Parameter	Unit	Value		
pH _{H₂O}		12.1 ± 1		
Electrical conductivity (EC)	mS/cm	0.91 ± 0.3		
Loss by ignition at 550 °C	%	2.2 ± 0.2		
Loss by ignition at 1100 °C	%	27 ± 2		
Solubility in H ₂ O	%	15 ± 0.9		
Solubility in 0.1 M HCl	%	57 ± 4		
Dry inorganic matter	%	100 ± 4		
Organic C	%	1.22 ± 0.01		
P ₂ O ₅	%	2.4 ± 0.4		
K ₂ O	%	6.5 ± 0.2		
CaO	%	39 ± 1		
MgO	%	3.15 ± 0.4		
MnO	%	0.20 ± 0.01		
Fe ₂ O ₃	%	1.62 ± 0.01		
Al ₂ O ₃	%	0.46 ± 0.01		
SiO ₂	%	10 ± 0.8		
Na ₂ O	%	0.70 ± 0.01		
Radionuclides				
⁴⁰ K	Bq/kg	1690 ± 40	423 *	4000 **
²³⁸ U	Bq/kg	12.5 ± 0.7	45 *	150 **
²²⁶ Ra	Bq/kg	39 ± 1	57 *	52 **
²³² Th	Bq/kg	23 ± 2	41 *	5.9 **
¹³⁷ Cs	Bq/kg	17.1 ± 0.6	25 *	0.18 **

* Average radionuclide activity concentration in Croatian non-arable topsoil (0–10 cm) [17,18]. ** Average radionuclide activity concentration in widely used mineral fertilisers in Croatian conventional agroecosystems [12].

2.3. Physicochemical & Radiological Analyses

Physicochemical analyses of all samples were conducted at the University of Zagreb, Faculty of Agriculture, in two accredited laboratories (MELILAB and ALIB), and in the laboratory of the Department of Pedology. A mechanical composition of soil samples was determined by the pipette method [19]. Physical soil properties (density, porosity, water field capacity, wilting point, water retention capacity, air capacity) were determined in undisturbed samples, collected using inox rings (100 cm³) and following the procedures as described in detail by [4,20]. A soil pH was determined potentiometrically according to [21], and electrical conductivity (EC) according to [22] using a dual-channel Mettler Toledo MPC 227 instrument. Humus content was detected by the modified Walkly–Black method according to [23]. The physicochemical characterisation of FBA was conducted following the protocols and techniques outlined by [3] (Table 1). In brief, elemental composition analysis was performed using ICP-OES after microwave-assisted digestion (HNO₃:HClO₄:HF, 2.5:2). Total C content was determined by dry combustion utilising a CN Analyzer (Thermo Fisher Scientific). The pH and EC values of the ash water eluate were determined using the same methodology as described above for soil analysis. Additionally, losses were determined gravimetrically at temperatures of 550 and 1100 °C.

Radiological analyses were carried out in the Radiation Protection Unit of the Institute for Medical Research and Occupational Health, following procedures recommended by the International Atomic Energy Agency [24]. In short, dried seed samples were homogenised using an inox grinder and then ashed at 400 °C. The resulting samples, as well as homogenised soil and BFA samples, were placed in 1 L cylindrical containers, sealed, and left to rest for 30 days. This led to the establishment of secular equilibria within the ²³⁸U and ²³²Th decay chains before measurements of A_c [25]. By following these procedures, we achieved compatibility with internationally recognised guidelines and protocols, thus ensuring the accuracy and reliability of the radiological measurements conducted in this study [25]. In all of the samples, the A_c of radionuclides of interest (²³⁸U, ²³²Th, ²²⁶Ra, ²¹⁰Pb, ⁴⁰K, and ¹³⁷Cs) was determined by means of high-resolution gamma spectrometry [17,18]. For measurements of radionuclides in soil and FBA samples, a High Purity Germanium (HPGe) ORTEC GEM50 photon detector system was utilised. This system had a relative efficiency of 50% and a resolution of 1.9 keV, both measured at 1.33 MeV. For the detection of the same radionuclides in sunflower seed samples, an HPGe ORTEC HP GMX photon detector system was employed, having a relative efficiency of 74% and a resolution of 2.26 keV, also measured at 1.33 MeV. A_c was determined from γ-ray spectra using the following energy peaks: ⁴⁰K (1460.82 keV), ¹³⁷Cs (661.66 keV), and ²¹⁰Pb (46.54 keV). The activities of weak γ-emitters were assessed assuming a secular equilibrium with their short-lived γ-emitting progenies. In a secular equilibrium, the activity of a long-lived parent nucleus and its short-lived progeny become equal after a sufficient amount of time has passed. This approach was employed to quantify the activities of ²³⁸U (through ²³⁴Th; peak at 63.29 keV and doublet at 92.38–92.80 keV), ²²⁶Ra (through ²¹⁴Pb; peaks at 295.22 and 351.93 keV), and ²²⁸Ra/²³²Th (through ²²⁸Ac; peaks at 338.32, 911.20, and 968.97 keV). The transmutations involved in this analysis included: ²³⁸U → ²³⁴Th (with a half-life of 24 days) → ..., ²²⁶Ra → ²²²Rn (with a half-life of 3.8 days) → ²¹⁸Po (with a half-life of 3.1 min) → ²¹⁴Pb (with a half-life of 27 min) → ..., and ²³²Th → ²²⁸Ra → ²²⁸Ac (with a half-life of 6.1 h) → ...

2.4. Data Processing and Statistical Analysis

Transfer factor (TF) is a commonly employed index in evaluating the accumulation of a specific element in plants and its corresponding concentration in the soil [12] or other growing media [26]. TF has a significant importance in environmental risk assessment and phytoremediation, as it provides insights into a plant's capacity to extract and accumulate contaminants from the soil [27]. However, in the context of food production, a lower TF is

desirable to prevent the uptake of hazardous substances by consumers. In this study TF values (from soil-to-seed) for specific radionuclides of interest were calculated as [12]:

$$A_c(R)_{\text{seed}} / A_c(R)_{\text{soil}} \quad (1)$$

where A_c is the activity concentration of a particular radionuclide in the sunflower seed $A_{c, \text{seed}}$ and tested soil $A_{c, \text{soil}}$ samples. TF values were calculated on a dry weight basis and are expressed in Bq/kg.

The influence of applied FBA doses on observed parameters in soil and test plant tissue was determined using Analysis of Variance (ANOVA). Mean values for each variant were then tested for differences using the Tukey–Kramer HSD test. Statistical data analysis for all observed parameters was conducted using SAS software, version 9.4.

3. Results and Discussion

3.1. Assessing the Influence of Fly Bioash (FBA) Application on Pedoecological and Crop Performances

In terms of tectonics, the study area is situated within the Pannonian Basin; this area sequentially housed Central Paratethys marine deposits during the Middle Miocene, followed by Lake Pannon brackish deposits in the Late Miocene, and ultimately witnessed the deposition of freshwater sediments from Lake Slavonia in the Pliocene [28]. By opening the pedological profile, it was confirmed that the study location was characterised by very acid (pH_{KCl} 4.0) Luvisol Siltic, Epidystric soil [29], developed on the Pleistocene loess plateau with a predominance of silt (74–80%) particle size distribution over the soil profile (Figure 2) (Table 2). In detected pedogenetic horizons the pH followed an increasing trend, starting from a very acidic pH reaction (4.01) in the surface Ap horizon (0–35 cm) and first subsurface (35–70 cm) horizon (4.41), and ending with an acidic pH reaction (4.68) in the second subsurface horizon (70–130 cm; Table 2). The organic carbon content across the soil profile was low, decreasing from 0.89% in Ap to only 0.41% in the subsurface horizon (Table 2). Parameters such as soil porosity (P), water retention capacity (WPC), and air capacity (AC) exhibited values within the ranges of 42–43% vol., 35–36% vol., and 6–8% vol., respectively (Table 2). Water field capacity (WFC) and wilting point (WP) were measured at 26–28% m and 9.3–12% m, respectively, whereas the soil density ranged from 1.51 to 1.54 g/cm^3 (Table 2). All detected physicochemical properties were consistent with Luvisol developed on loess parent material (substrate), as terrestrial clastic sediment that accumulates through the wind-blown accumulation of predominantly silt-sized particles. The loess worldwide covers approximately 10% of the Earth’s surface, whereas in Croatia, Pleistocene sediments are present on approximately 36% of the national territory, with significant coverage in the southern continental Pannonian region and a limited presence in small areas in the Dinaride Mountains and Mediterranean region [28].

Table 2. Physicochemical properties over the Luvisol Siltic, Epidystric soil profile at study location.

Horizon Depth cm	Particle Size Distribution %			$\text{pH}_{\text{H}_2\text{O}}$	pH_{KCl}	Organic C %	WFC	WP	Density	P	WRC	AC
	Sand	Silt	Clay									
Ap 0–35	1.8	80	19	5.6	4.0	0.89	26	9.3	1.51	43	35	8
E 35–70	1.3	74	25	6.1	4.4	0.41	27	11	1.54	42	36	6
Bt 70–130	1.3	74	25	6.4	4.7	-	28	12	1.53	43	36	7

P—porosity; WFC—water field capacity at 0.033 MPa; WP—wilting point at 1.5 MPa; WRC—water retention capacity; AC—air capacity.

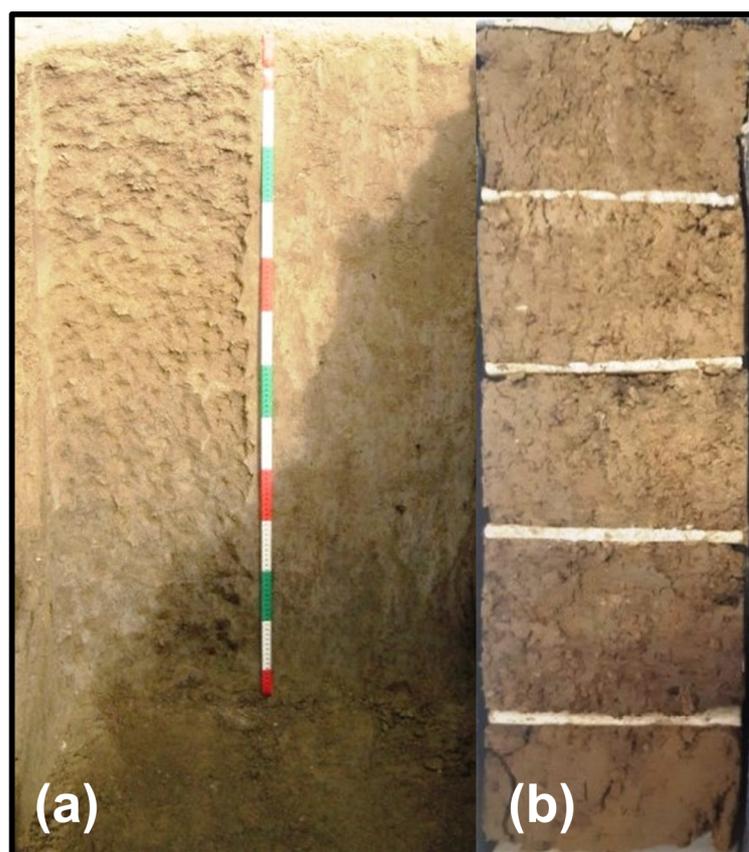


Figure 2. Pedological profile of the Luvisol Siltic, Epidystric soil profile at study location (a) with micro monoliths from a particular profile depth (b).

One of the key features of Luvisols is the presence of a prominent clay enrichment or eluviation (leaching) horizon, known as the Bt horizon. This horizon is characterised by the movement of clay particles, Fe, Al, Ca, and/or Mg compounds downwards due to the leaching process. The clay accumulates in the lower layers of the soil profile, resulting in a clay-enriched subsoil. Luvisols typically have a distinct soil structure with well-developed granular aggregates in the surface horizon. These aggregates contribute to good soil porosity and permeability, allowing adequate water infiltration and root penetration. The surface Ap horizon of Luvisols often has a higher organic matter content compared to the subsoil layer(s), as confirmed here (Table 2). The colour of Luvisols can vary depending on soil drainage conditions, organic matter content, mineralogical composition, and range from brown, reddish-brown, yellowish-brown, or greyish brown (e.g., Figure 2).

In Croatia, approximately 1.26 Mha, which account for more than 22% of the total land surface, are occupied by acidic soils with a $\text{pH}_{\text{H}_2\text{O}} \leq 6.0$ [30]. A significant portion of acidic soils falls under the category of Luvisols, covering over 703,000 ha (12.6% of Croatian soils [31]), where pedogenetic processes over time have led to leaching of essential nutrients such as Ca^{2+} , Mg^{2+} , K^+ [32], as confirmed in the studied profile (Table 2). Arable Luvisols generally have a good water-holding and nutrient retention capacity in the surface Ap layer (Table 2); however, they are not optimised for high-yield production and require chemical conditioning. Namely, because of their deficiencies in essential nutrients and limited capacity for stable soil structure, acid soils often exhibit elevated levels of potentially toxic metals (Al, Mn, Fe), leading to phytotoxicity [33,34] and many other soil constraints (more below). A recent study encompassing approximately 5.6 M km² of agricultural areas across 33 European countries found that the average pH in the surface Ap layers was 5.8 and even lower at 5.5 in the deeper Gr layers [35]. It is estimated that acidic soils cover more than 4 billion ha worldwide, accounting for over 70% of potentially arable

land [36]. Soil acidity can significantly reduce crop yield and quality, while also negatively impacting other pedovariables such as water permeability, aggregate stability, and soil structure [33,37]. To ameliorate soil acidity, various materials enriched with Ca/Mg, such as lime, lime and dolomite fractions, and saturation mud from sugar refineries, are commonly used for pH correction [6,33]. For instance, the conditioning of acid Luvisols with lime not only increases soil pH and nutrient availability but also ameliorates soil aggregate structure stability, promotes flocculation through its electrolyte properties, increases exchangeable Ca^{2+} , provides physicochemical protection to soil organic C, stimulates soil biological activity, and ultimately improves crop productivity [37]. In addition, the use of the synthetic conditioner polyacrylamide has been found effective in ameliorating other soil constraints commonly found in acid Luvisols, including crusting and erosion, lower water retention, and/or drainage capacity [37]. Therefore, the utilisation of FBA in soil amelioration, within the framework of the circular bioeconomy, offers a more sustainable and environmentally friendly alternative in such contexts, which should be further investigated. Namely, our results provide strong evidence of the significant potential of FBA for pH amelioration in acid Luvisols under open field conditions, with a notable increase of 1.8 pH units observed at a dosage of 17.2 t/ha (Table 3). Additionally, the addition of FBA did not compromise soil electrical conductivity (EC) (Table 3). Moreover, application of FBA resulted in substantial improvements in sunflower fresh and dry seed yields, with increases of up to 15.4% and 13.4%, respectively, compared to the non-amended Control soil (Table 3). With the significant increase in the generation of FBA in Croatia over the past decades, this valuable by-product possesses substantial potential for successful reuse in agricultural (and forestry) chemical amelioration of acidic soils [6]. Utilising FBA allows for a valuable contribution towards promoting a greener bioeconomy.

Table 3. The effect of fly bioash (FBA) treatments 54 months after application on pH and electrical conductivity (EC) in the topsoil of acid Luvisol, and on fresh and dry seed yield of sunflower (*Helianthus annuus* L., Hybrid NK Neoma) (Mean \pm Standard Error, significant at $p < 0.05$ *; $p < 0.01$ **; $p < 0.001$ ***).

FBA Treatments t/ha	pH _{H2O}	pH _{KCl}	EC mS/cm	Fresh Seed Yield g/Plant	Dry Seed Yield g/Plant
0	5.4 \pm 0.02	4.1 \pm 0.03	0.08 \pm 0.02	510 \pm 10	454 \pm 8
4.5	5.6 \pm 0.07	4.3 \pm 0.06	0.07 \pm 0.01	580 \pm 20	520 \pm 20 *
8.0	5.8 \pm 0.09	4.5 \pm 0.1	0.06 \pm 0.01	556 \pm 15	490 \pm 10
13	6.5 \pm 0.2 ***	5.5 \pm 0.2 **	0.11 \pm 0.03	580 \pm 40	480 \pm 10
17.2	6.8 \pm 0.2 ***	5.9 \pm 0.4 ***	0.12 \pm 0.02	520 \pm 15	460 \pm 10

3.2. Assessing the Influence of Fly Bioash (FBA) Application on Radionuclides Activity Concentration and Soil-to-Seed Transfer Factor

Our radiological results demonstrated clearly that the application of FBA had no significant impact on the concentration of radionuclides in the surface Ap horizon of acid Luvisol, irrespective of either depth (0–30 cm) or the relatively wide range of applied FBA dosages (0–17.2 t/ha; Table 4). The average activity concentrations of almost all of the studied radionuclides in the non-amended acid Luvisol (Control treatment; Table 4) were higher (by 127 Bq/kg for ^{40}K , 15 Bq/kg for ^{238}U , 23 Bq/kg for ^{226}Ra , and 11 Bq/kg for ^{232}Th) than their corresponding averages in Croatian non-arable topsoil (Table 1). The activity concentration of ^{137}Cs in the Control surface Ap horizon was markedly lower, showing an approximately 7-fold decrease (3.5 Bq/kg; Table 4) in comparison with the concentration in Croatian non-arable topsoil (25 Bq/kg; Table 1).

Table 4. The effect of fly bioash (FBA) treatments 54 months after application on radionuclide activity concentrations (Bq/kg) in the surface Ap horizon of acid Luvisol (Mean \pm Standard Error).

Radionuclide	Soil Depth cm	FBA Treatments (t/ha)				
		0	4.5	8.0	13	17.2
^{40}K	0–10	560 \pm 20	580 \pm 20	590 \pm 20	580 \pm 20	560 \pm 20
	10–20	550 \pm 20	590 \pm 20	580 \pm 20	580 \pm 20	550 \pm 20
	20–30	540 \pm 20	590 \pm 20	600 \pm 20	570 \pm 20	570 \pm 20
^{238}U	0–10	56 \pm 6	59 \pm 6	57 \pm 6	57 \pm 6	57 \pm 6
	10–20	57 \pm 8	59 \pm 6	58 \pm 6	55 \pm 6	56 \pm 6
	20–30	57 \pm 8	61 \pm 7	58 \pm 6	58 \pm 6	58 \pm 6
^{226}Ra	0–10	45.4 \pm 1.5	46 \pm 1	46.3 \pm 1.5	48 \pm 1	45 \pm 1
	10–20	44.9 \pm 1.5	48 \pm 1	45.9 \pm 1.5	47 \pm 1	44 \pm 1
	20–30	43.8 \pm 1.5	48 \pm 1	48.0 \pm 1.5	46 \pm 1	46 \pm 1
^{232}Th	0–10	52 \pm 2	52 \pm 2	53 \pm 2	53 \pm 2	50 \pm 2
	10–20	51 \pm 2	53 \pm 2	52 \pm 2	52 \pm 2	48.8 \pm 1.5
	20–30	50 \pm 2	54 \pm 2	54 \pm 2	52 \pm 2	51 \pm 2
^{137}Cs	0–10	3.5 \pm 0.2	3.5 \pm 0.2	3.4 \pm 0.2	4.5 \pm 0.2	3.9 \pm 0.2
	10–20	3.4 \pm 0.2	3.5 \pm 0.2	3.3 \pm 0.2	4.4 \pm 0.2	3.9 \pm 0.2
	20–30	3.4 \pm 0.2	3.5 \pm 0.2	3.2 \pm 0.2	4.5 \pm 0.2	4.1 \pm 0.2

In general, our results agreed with some recent studies conducted in Croatian continental agroecosystems, in both no amended [38] and fertilised soils [12]. Some possible explanations for these results could be the soil management practices employed on the study area. In this context, deep ploughing (35–40 cm) and shallow inter-row cultivation (15–20 cm) likely played a role in facilitating the uniform mixing and distribution of the FBA matrix across the surface horizon of the Luvisol. In addition, the significant soil mass of the Ap horizon compared to the quantity of FBA applied, coupled with the radionuclide activity concentration in the FBA matrix (Table 1), led to a dilution effect, explaining why even at relatively higher amelioration rates (>10 t/ha) FBA did not alter the radiological profile of the acid Luvisol (Table 4). The average activity concentrations of all of the studied radionuclides in the examined FBA matrix, except for K (Table 1), were lower (3.5 times for ^{238}U , 1.5 times for ^{226}Ra , 1.8 times for ^{232}Th , and 1.5 times for ^{137}Cs) compared with their corresponding averages in Croatian non-arable topsoil (0–10 cm) [17,18]. The activity concentration of ^{40}K in the FBA was nearly four times higher (1690 Bq/kg) than in Croatian non-arable topsoil (423 Bq/kg; Table 1). These findings supported recent studies that highlight how the radioactivity concentration of ^{40}K tends to be lowest in the Dinaric karstified region (located ~400 km away from the study location) characterised by sedimentary limestone and dolomite bedrocks, while ^{238}U , ^{232}Th , ^{226}Ra , ^{210}Pb , and ^{137}Cs in the same region exhibit the highest radioactivity concentrations [17,18]. According to the same study, the concentration of ^{137}Cs is typically lower in Croatian Pannonian agroecosystems (i.e., study location); however, observations indicated that the concentration tends to increase with altitude, annual precipitation, and vegetation density.

Furthermore, our findings presented in Table 5 clearly show that the amelioration of test soil with FBA had no substantial impact on the concentration of radionuclide activity in the analysed sunflower seeds, as well as their specific transfers (TFs) to edible seed tissues. The measured levels of ^{40}K and ^{226}Ra in the seed samples were detectable, but consistently lower, even by up to 2.6 times and 61 times, respectively, compared to the corresponding concentrations in the soil (Table 5). On the other hand, the A_c of ^{238}U , ^{232}Th , and ^{137}Cs were below the detection limit (DL) in all of the seed samples (Table 4). All the

TFs observed for ^{226}Ra consistently fell within the lower range of TF values reported in some previous studies, including [39–42]. Remarkably, some TF values even fell below the TF interval presented by [43] for various plant species. Additionally, the TFs for ^{40}K were found to be in line with the established ranges reported in [39–41] for similar plants and transfer mechanisms. Thus, our results indicated a very limited transfer of radionuclides from the soil to the seeds, even for the macronutrient isotope ^{40}K . However, albeit not essential, other radionuclides in the soil constitute a pool available for (i) downward migration within the soil profile [25], (ii) the root uptake [12], and/or their (iii) root-to-shoot translocation [39,43]. Determining the migration depth of radionuclides in soil is vital for decrement of external dose rates from soils contaminated with radionuclides. This also facilitates soil decontamination strategy methods such as phytoremediation [27].

Table 5. The effect of fly bioash (FBA) treatments 54 months after application on radionuclide activity concentrations (Bq/kg) in the seed of sunflower (*Helianthus annuus* L., Hybrid NK Neoma) and soil-to-seed transfer factor (TF) (Mean \pm Standard Error).

Radionuclide	FBA Treatments (t/ha)									
	Radionuclide Activity Concentrations (Bq/kg)					Soil-to-Seed Transfer Factor (TF)				
	0	4.5	8.0	13	17.2	0	4.5	8.0	13	17.2
^{40}K	229 \pm 8	231 \pm 9	227 \pm 8	233 \pm 9	237 \pm 9	0.42	0.39	0.38	0.40	0.42
^{238}U	<4	<3	<4	<4	<3	<0.07	<0.05	<0.07	<0.07	<0.05
^{226}Ra	1.1 \pm 0.1	1.6 \pm 0.1	1.1 \pm 0.1	2.0 \pm 0.2	0.73 \pm 0.08	0.025	0.034	0.024	0.043	0.016
^{232}Th	<1	<1	<1	<1	<1	<0.02	<0.02	<0.02	<0.02	<0.02
^{137}Cs	<0.3	<0.3	<0.3	<0.3	<0.3	<0.09	<0.11	<0.09	<0.07	<0.08

For the radionuclides where measured activity concentrations were below detection limit (DL), the transfer factor is expressed as < (“less than”) the DL divided by the activity concentration in soil. Value < X in table means that the measured value was below DL, the smallest value needed to quantify the result.

Because of its chemical similarity to Ca and Mg, Ra tends to be taken up by plants in place of these elements, particularly in Ca- and/or Mg-deficient soils, as observed here in tested acid Luvisol. This characteristic explains the detection of ^{226}Ra in the sunflower seed samples while the parent radionuclide in the decay chain, ^{238}U , was not detected (Table 5). Additionally, it is important to note that ^{226}Ra , being an alpha emitter, can pose significant harm to plants and, subsequently, to humans if it enters the food chain through crop consumption. Nevertheless, the activity concentrations observed in the different varieties of sunflower seeds showcased in this study were sufficiently low to ensure the safety of the general population (Table 5). By employing the ingestion dose coefficients provided by [44], even an individual from one of the most vulnerable populations, such as a young teenager, would need to consume a staggering 333 kg of the sunflower seed with the highest TF for ^{226}Ra in order to receive a radiation dose of 1 mSv, which is a yearly dose limit for the general public stemming from non-natural sources.

Although further research and comparative studies are necessary, the findings of this study suggest that the application of FBA, under specific soil management practices and soil characteristics, may not significantly impact radionuclide activity concentrations in the soil. This information can be valuable for decision makers and practitioners involved in utilising FBA as a soil amendment, providing insights into the potential outcomes and considerations regarding radiological contamination in agroecosystems and/or forestry. Recent study has also corroborated these findings, demonstrating the absence of radiological contamination across various ash matrices, including fresh fly ash, aged fly ash, and bottom ash generated from coal [9]. Namely, in the same study it was concluded that all of these examined ashes, considering their radiological and technical footprint, have the potential for utilisation as secondary green raw materials in various sectors, including civil and chemical engineering, for the generation of value-added products (e.g., nano-materials, nano-polymers, sorbents, zeolites). The affirmation of positive results across diverse ash

matrices highlighted the extensive potential of these complex materials as valuable secondary resources for sustainable applications in multiple fields, fostering the development of innovative value-added products and promoting the principles of circular economy and environmental sustainability.

4. Conclusions

This study investigated the effects of highly alkaline FBA (applied at doses of 0–17.2 t/ha) on (i) pH, (ii) salinity (EC), and (iii) radionuclide activity concentrations (measured by high-resolution gamma-ray spectrometry) in very acidic Luvisol, Siltic, Epidystric soil, and (iv) sunflower seed. The radiological fingerprint of hardwood-derived FBA revealed significantly lower A_c of most of the studied radionuclides compared with: (i) Croatian non-arable topsoils (with reductions of ^{238}U by 3.6-fold, ^{232}Th by 1.8-fold, ^{226}Ra by 1.7-fold, and ^{137}Cs by 1.5-fold) and (ii) synthetic N/P/K fertilisers commonly used in Croatian conventional farming (with reductions of ^{238}U by 12.5-fold, ^{226}Ra by 1.3-fold, and ^{40}K by 2.4-fold).

Application of FBA at a dosage of 17.2 t/ha significantly increased soil pH (by up to 1.8 unit) and seed yield by 15% compared to the Control treatment (0 t/ha FBA) without impacting soil EC, i.e., salinity. The A_c of all observed radionuclides remained largely unchanged under FBA application in both (i) the surface Ap Luvisol layer and (ii) sunflower seeds. Moreover, in the seed, the A_c values of ^{238}U , ^{232}Th , and ^{137}Cs were below the detection limit, while those of ^{40}K and ^{226}Ra were lower by up to 2.6-fold and 61-fold, respectively, compared to their corresponding levels in the soil treatments.

Our findings provide strong evidence that FBA can serve as an effective soil conditioner, in organic and/or conventional farming, with no significant radiological health or environmental risks, thereby contributing to more sustainable agri-food production and circular green bioeconomy. However, additional comprehensive studies are necessary to investigate the long-term effects of FBA application on other pedovariables and crop performance in diverse agro-environmental conditions.

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