Influence of biomass ash fertilization on soil pH, CaCO₂, K₂O and P₂O₅ **under** *Miscanthus x giganteus*

Utjecaj primjene pepela na reakciju tla, sadržaj CaCO3, K2O i P2O5 pod kulturom *Miscanthus x giganteus*

Dija BHANDARI¹, Stella ARLOVIĆ², Marija GALIĆ³, Nikola BILANDŽIJA⁴, Josip LETO⁵, Darija $BILANDŽIJA³ ($

¹ University of Zagreb Faculty of Agriculture, Graduate Study programme in "Sustainability in Agriculture, Food Production and Food Technology in the Danube Region" (DAFM), Svetošimunska cesta 25, Zagreb, Croatia

 2 University of Zagreb Faculty of Agriculture, Graduate Study programme in "Agroecology", Svetošimunska cesta 25, Zagreb, Croatia

³ University of Zagreb Faculty of Agriculture (AGR), Division for Agroecology, Svetošimunska cesta 25, Zagreb, Croatia

⁴ University of Zagreb Faculty of Agriculture, Division for Agricultural Engineering and Technology, Svetošimunska cesta 25, 10000 Zagreb, Croatia

⁵ University of Zagreb Faculty of Agriculture, Division for Plant Sciences, Svetošimunska cesta 25, Zagreb, Croatia

i Corresponding author: dbilandzija@agr.hr

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ABSTRACT

The cultivation of energy crops and the use of biomass ash are part of a sustainable energy economy and have a major impact on the environment. Biomass ash can be used as a soil conditioner due to its chemical composition with essential micro- and macronutrients and has a proven positive effect on the physicochemical properties of soils. With the increasing interest and use of bioenergy species, the production of biomass ash is also increasing and is expected to increase further in the future. Therefore, the correct use of this product is important. Ensuring its use to enrich soils will strengthen the ecological aspect and close the gaps in the circular economy. In this context, investigating the influence of biomass ash on individual soil properties under different agro-ecological conditions is important to determine its possible uses and limitations. The aim of the study was therefore to determine the influence of different ash dosages (2 and 5 t/ha) on soil pH, carbonate content (CaCO₃), the content of plant-available phosphorus (P₂O₅) and plant-available potassium (K₂O) under the perennial energy crop Miscanthus x giganteus. The study was conducted in the years 2016-2019 in the continental part of Croatia near Zagreb city. The study showed that the application of ash had no significant effect on soil pH, but did have an effect on CaCO₃, P₂O₅ and K₂O. At both ash doses studied, there was a significant decrease in P₂O₅ and a significant increase in CaCO₃, while the K₂O content only increased significantly at the higher ash dose compared to the control treatment. Biomass ash proved to be a good soil conditioner in terms of CaCO₃ and K₂O content under the agroecological conditions studied. Further studies on various agroecological conditions such as soil types, vegetation and climate will provide further insights into the effective use of biomass ash. Likewise, research on the influence of biomass ash on other important soil properties in combination with studies on plant uptake is necessary to understand the complex interaction of biomass ash.

Keywords: forest biomass ash, chemical soil properties, perennial energy grass, sustainability, circular economy

SAŽETAK

Uzgoj energetskih kultura i primjena pepela iz biomase dio su održivog energetskog gospodarenja i imaju značajan utjecaj na okoliš. Pepeo iz biomase može se koristiti kao gnojivo ili poboljšivač tla zbog svog kemijskog sastava odnosno sadržaja važnih mikro i makro hranjiva, kao i pozitivnog učinka na fizikalno-kemijska svojstva tla. Sa sve većim interesom i korištenjem bioenergetskih kultura, proizvodnja pepela iz biomase također raste i očekuje se da će se u budućnosti dodatno povećati. Stoga je važna pravilna primjena ovog proizvoda. Osiguravanje njegove primjene kao poboljšivača tla ojačati će ekološki aspekt i zatvoriti praznine u kružnom gospodarstvu. U tom kontekstu, istraživanje utjecaja pepela iz biomase na pojedinačna svojstva tla u različitim agroekološkim uvjetima važno je za određivanje njegove moguće primjene i ograničenja. Stoga je cilj istraživanja bio utvrditi utjecaj različitih doza pepela (2 i 5 t/ha) na pH vrijednost tla, sadržaj karbonata (CaCO₃) u tlu, sadržaj biljkama dostupnog fosfora (P₂O₅) i kalija (K₂O) u tlu pod višegodišnjom energetskom kulturom *Miscanthus x giganteus*. Istraživanje je provedeno tijekom razdoblja 2016.-2019. u kontinentalnom dijelu Hrvatske u blizini grada Zagreba. Istraživanjem je utvrđeno da primjena pepela nije značajno utjecala na pH tla, ali je imala značajan utjecaj na sadržaj CaCO₃, P₂O₅ i K₂O u tlu. Kod obje istraživane doze pepela došlo je do značajnog smanjenja sadržaja P₂O₅ i značajnog povećanja sadržaja CaCO₃, dok se sadržaj K₂O u tlu značajno povećao samo pri većoj dozi pepela u usporedbi s kontrolnim tretmanom. Pepeo iz biomase pokazao se kao dobar kondicioner tla u pogledu sadržaja CaCO₃ i K₂O u proučavanim agroekološkim uvjetima. Daljnja istraživanja o primjeni pepla u različitim agroekološkim uvjetima poput različitih tipova tala, vegetacijskog pokrova i klimatskih uvjeta pružit će detaljniji uvid u učinkovito korištenje pepela iz biomase. Također, istraživanje o utjecaju pepela iz biomase na druga važna svojstva tla u kombinaciji s istraživanjima o biljnom usvajanju hranjiva potrebno je za razumijevanje složenog ponašanja pepela iz biomase.

Ključne riječi: pepeo šumske biomase, kemijska svojstva tla, višegodišnje energetske kulture, održivost, kružno gospodarstvo

INTRODUCTION

The cultivation of energy crops and the use of biomass ash are part of a sustainable energy economy and have a major positive impact on the environment. Energy from renewable sources like biomass has the potential to replace fossil fuels as they are considered carbon neutral. Biomass as a renewable source is being intensively researched and it is predicted that its use will increase significantly in the future. Biomass burning produces biomass ash. It is estimated that the combustion of biomass for energy currently produces a total of around 170 million tons of ash per year worldwide, and in the future, this figure could rise to as much as 1000 million tons per year (Zhai et al., 2021).

One of the main concerns in the field of bioenergy is therefore the most innovative, economical and sustainable use and/or environmentally sound disposal of this large amount of biomass ash. Vassilev et al. (2013b) states in the review that the main applications of biomass ash include soil improvement and fertilization, followed

by building materials and sorbents, and occasionally the synthesis and production of minerals, ceramics and other materials. Among the options for ash utilization, the use of biomass ash as a secondary raw material for soil improvement measures on agricultural and forestry land (direct or indirect application) falls under sustainable ash utilization among other utilization options (Obernberger and Supancic, 2009). For sustainable use of biomass, both domestically and on an industrial scale, it is important to recognize that ash can largely be returned to the soil, renewing and replacing the nutrients removed (Ilyushechkin et al., 2012).

The solid residue that is produced when biomass is burned is biomass ash. This combustion residue is a complex inorganic-organic mixture with multicomponent, heterogeneous and variable composition and contains closely interconnected solid, liquid and gaseous phases of different origins (Vassilev et al., 2013a). The composition, content and origin of the biomass components as well

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as the ash yield are important when it comes to the use of biomass ash. Ash content varies among different types of biomass and can be as low as 0.5% on a dry basis for some types of pulpwood or as high as 20% for some cereals or forest and agricultural wastes (Garba et al., 2013). The composition of biomass ash is influenced by a variety of factors. The combustion process (fuel preparation, combustion technology and conditions, collection and cleaning facilities), as well as the biomass resources (type of biomass, plant species or plant part, cultivation processes and conditions, age of plants, application of agrochemicals, type of harvesting) and also a number of processes such as transportation, storage, contamination, processing, etc., affect the properties and composition of biomass (Vassilev et al., 2010, 2013a). The chemical composition of biomass ash varies greatly depending on the composition of the biomass. The most common components of ash are CaO, K_2O and SiO₂ (Zhai et al., 2021), two of which are essential macroelements for plants. In addition, the elements in biomass ash can be generally divided into micro, macro and trace elements based on their elemental concentration. Based on the chemical data on the composition of 86 types of biomass, their groups and subgroups, the macro and micro elements in decreasing order are usually $O > Ca > K > Si$ > Mg > Al > Fe > P > Na > S > Mn > Ti and some Cl, C, H, N and trace elements (Vassilev et al., 2010). In addition to the alkali (Na, K) and alkaline (Ca, Mg) earth metals typical of biomass, ash can also contain metals (Cd, Co, Cr, Cu, Fe, Mn, Ni, Pb, Zn) that limit the direct use of ash in the soil i.e. environment (Kalembkiewicz et al., 2018). The ash resulting from industrial incineration can be divided into two different types: bottom ash and fly ash, which also have different compositions. This distinction must be taken into account, as fly ash has a higher concentration of heavy metals (Obernberger and Supancic, 2009).

Forest residue biomass is a relatively abundant renewable resource, mostly originating from forestry activities and forest management practices (e.g. forest fire prevention), and is ideally suited for thermochemical processing into energy vectors (Silva et al., 2019). The resulting biomass ash must be used effectively, and

recycling the ash on agricultural land can help reduce the use of artificial fertilizers and close the natural mineral cycle. The estimates of biomass ash derived from forestry are dominated by wood fuel ash (Zhai et al., 2021). The aim of this paper is to evaluate the effects of using forestry-derived biomass ash on soil chemical properties and the possibility of using biomass ash as a soil conditioner during cultivation of giant miscanthus (*Miscanthus x giganteus*). The soil chemical properties to be investigated are i) soil pH, ii) calcium carbonate content, iii) plant-available potassium content and iii) plant-available phosphorus content.

Soil pH is referred to as the "main soil variable" that influences biological, chemical and physical properties and processes in the soil that affect plant growth and biomass yield (Neina, 2019). Neutral soil pH is considered the most favorable pH for plant growth and the problem of soil acidification has been identified as the main threat to soil quality (Caon and Vargas, 2017). It is estimated that up to 30% of the Earth's ice-free land area is occupied by acidic soils, which corresponds to about 4,000 Mha (Von Uexküll and Mutert, 1995). In agricultural practice, soil acidification is usually corrected by the application of calcareous calcium carbonate ($CaCO₂$) to increase the pH of the soil. Due to the alkaline nature of biomass ash, it can be used as a soil-liming material (Qin et al., 2017). CaCO₃ is the main source of calcium (Ca) in soil. It is not only an essential plant nutrient, but also a major component of soil and forms the main exchangeable base of clay minerals. Although Ca is present as a natural mineral in the soil, Ca is lost from minerals over time through weathering and is completely lost as a structural component in mature soils (Barker and Pilbeam, 2015). In more mature soils, the Ca present is bound to cation exchange sites, where it usually makes up a high proportion of the total exchangeable cations, so the amounts present depend on the cation exchange capacity (CEC) of the soil. Biomass ash application can provide Ca and also prevent Ca leaching by increasing the pH and site of ion absorption. Potassium (K) is an essential nutrient required by plants and is routinely added to the soil as fertilizer. K is one of the major constituents of biomass

ash. The study conducted by Samadhi et al. (2019) on the feasibility of K recovery from biomass ash and its use as a raw material for the production of fertilizer solutions highlights the importance of biomass ash as a K additive. The direct or indirect application of biomass ash can therefore replace artificial mineral fertilizers. Like nitrogen and potassium, phosphorus (P) is also an important macronutrient for plants, which is required for important life functions. It is also one of the most limited nutrients in the soil. Extensive cultivation of vegetables and other crops, as well as natural hazards, deplete the natural phosphate content in the soil (De Silva et al., 2015). If the soil is unable to supply sufficient P, this leads to deficiency symptoms and crop yield losses. Farmers have used phosphate fertilizers to supplement the plants' needs. The phosphorus taken up by plants from the soil comes from the soil solution, where it is present as the inorganic orthophosphate ion H_2 PO₄, HPO₄² and PO_4^3 , with 'orthophosphate' being the most available (Vidyapeetham, 2013). This inorganic form, which occurs in the soil solution, is therefore the available P for the plants. Together with other macronutrients, biomass ash contains P and can serve as an important supplement in the soil. Although much research has been conducted on the production and properties of biomass ash, there are still gaps in knowledge and research opportunities, particularly with regard to the beneficial reuse of ash (Zhai et al., 2021). Considering the context and importance of biomass ash for agricultural use and to address the gaps in the circular economy, the following research questions were formulated: What are the effects of biomass ash (forest biomass) on soil chemical properties? How well can biomass ash be used as a potential soil conditioner?

MATERIALS AND METHODS

Site Description

The study was conducted at the experimental station "Šašinovec" of the Faculty of Agriculture University of Zagreb (FAUZ), located in the continental part of northwestern Croatia, near the Zagreb city (N 45°51'01.32"; E 16°10'35.85", at the elevation of 123 m above sea level). The experimental field with giant miscanthus (*Miscanthus x giganteus* Greef et Deu) was established in 2016.

Climate

The research area has a temperate continental climate. According to the Köppen classification, the research area has a "Cfwbx" climate (a temperate precipitation climate). The area has an average annual air temperature of 11.8 °C, an average annual precipitation amount of 867 mm, evapotranspiration is 618 mm, the growing degree days for the growing season are 1616 mm and a rain factor is 73.3, indicating a semi-humid climate in the period 1991 – 2018 (Bilandžija, 2019). Temporal variation of average annual air temperature and precipitation amount are presented at Figure 1.

Soil

Soil sampling to determine the chemical soil properties at the research site was carried out in 2016 before the field was established. Sampling was carried out in three repetitions using an Eijkelkamp soil probe in the surface soil layer of 0-30 cm soil depth. The soil at the research site has alkaline reaction (pH_{KCl} = 7.26), contained 0.11% total nitrogen and 1.88% humus, the carbonate content was low (7.6%), it was well supplied with plantavailable potassium (18.7 mg $K₂O/100$ mg soil) and very well supplied with plant-available phosphorus (43 mg $P_2O_5/100$ g soil). In order to determine the changes in chemical soil properties due to the application of ash, further soil sampling was carried out in three repetitions in 2020 using the Eijkelkamp soil probe in the surface soil layer of 0-30 cm soil depth.

Experimental Design

In a 3-year-old Miscanthus plantation, an additional experiment was set up in 2019 with 2 different doses of ash as a soil conditioner in 3 repetitions (Table 1). The ash was applied in spring, before the beginning of vegetation period, i.e. the sprouting of miscanthus shoots.

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Figure 1. Average annual air temperature, precipitation amount and water balance at the site (source: Bilandžija, 2019)

Table 1. The experiment treatments

The ash applied is obtained from forest biomass and has an alkaline reaction, a low humus content, a medium carbonate content, well supplied with plant-available phosphorus and very well supplied with plant-available potassium (Table 2).

Laboratory analysis

The analysis of the soil samples sampled at the experimental field in 2020 was carried out in the analytical laboratory of the Department of General Agronomy, Faculty of Agriculture, University of Zagreb. Analyzed soil properties are: the soil reaction (pH), plant-available

Table 2. Properties of applied biomass ash

phosphorus (P_2O_5) and plant-available potassium (K₂O), and calcium carbonate (CaCO₂) content. The soil reaction was determined by potentiometric measurement in a suspension of soil and extractant (1 M KCl) in a ratio of 1:2.5 in accordance with the HRN ISO 10390:2005 standard. The plant-available phosphorus and potassium were extracted with an ammonium-lactate-acetic acid solution (Egner et al. 1960). Potassium was then determined using the chemical flame photometry method and phosphorus using the colorimetric method. The CaCO₃ content was determined according to the volumetric method.

Statistical Analysis

Data analysis was performed using STATISTICA 12 software (StatSoft Inc, 2014). The variability between the treatments studied was analyzed using analysis of variance (ANOVA) and, if necessary, tested using the Tukey test. The significance threshold for all analyses was 5%. The prescribed quality control procedures are carried out in the analytical laboratory of the Department of General Agronomy, Faculty of Agriculture, University of Zagreb.

RESULTS AND DISCUSSION

The results of the statistical analysis show that no significant change in pH was observed. However, the CaCO₃ content, measured as a percentage (%), increased significantly with both two ash doses of 2 t/ha and 5 t/ ha studied. There was a significant increase in the plantavailable $K₂O$ (mg/100 kg) of the soil at the higher dose of 5 t/ha applied. However, application with an ash dose of 2 t/ha did not result in a significant change in the measured plant-available $K₂O$ content. With regard to the plant-available P_2O_5 content (mg/100 kg soil), the results showed a decreasing effect when both ash doses were applied. The results obtained in the research are presented in Figure 2.

The alkaline nature of biomass ash plays a role in improving soil reaction by increasing the pH. Maintaining the proper soil pH is paramount to soil processes, including microbial functions, solubility of nutrients, and minimizing leaching and immobilization problems in agriculture. The effectiveness of applying biomass ash to increase soil pH has been successfully documented (Füzesi et al., 2015; Quirantes et al., 2016; Shi et al., 2017). Our analysis showed that the pH increased but not significantly with the applied doses. Gibczyńska et al. (2014) also found no significant increase in soil pH.

The alkalizing effect associated with the application of biochar to the soil environment is related to the content of alkaline compounds and the application rate (Saletnik et al., 2018). Significant effects on soil pH when applying biomass ash can be achieved by applying higher doses of 10-20 t/ha (Park et al., 2005; Saletnik et al., 2018). Füzesi et al. (2015) discovered that when ash was applied at 10 t/ha, the pH increased by about one unit. However, such high doses are not recommended as they could lead to excessive amounts of nutrients or heavy metals entering the soil. Another reason for the non-significant

Figure 2. Effects of different ash doses applied on soil pH, CaCO₃ (%), K₂O (mg/100g), and P₂O₅ (mg/100g) during cultivation of *Miscanthus x giganteus*

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increase could be the duration of the period after the ash application (one year) and the original pH of the soil, which was slightly alkaline. According to the existing literature, the most significant changes can be achieved in highly acidified soils, with a visible decrease in the proportion of H^+ and Al^{3+} (Zong et al., 2016).

The results show that the plant-available phosphorus (P_2O_5) decrease after ash treatment, which is in contrast to the results of the literature (Wierzbowska et al., 2020; Füzesi et al., 2015; Li et al., 2016; Gibczyńska et al., 2014). However, Omil et al. (2011) also observed a low response of phosphorus to the different ash treatments. The reasons for this could be the comparatively low availability of P in the ash, the chemical and microbial immobilization of the element and also the rapid uptake by the plants. The ability of ash to supply macronutrients, including P, has been widely documented in the literature and Wierzbowska et al. (2020) also found that the fertilizing effect of P from ash is comparable to the effect of readily soluble phosphorus fertilizers. The dissolution of ash in soil is a complex process, as is the rate at which nutrients become available to plants, as each cation present in biomass ash is usually present in multiple forms such as oxides, hydroxides, carbonates and bicarbonates (Mercl et al., 2016). Similarly, the time factor after application also plays an important role in the presence of plant-available nutrients in the soil solution (Ochecova et al., 2016). The exact impact of ash application on the soil also depends on the type of cultivated crops on that soil (Wierzbowska et al., 2020). It was concluded that the behavior of biomass ash and nutrient dynamics in the soil is a complex phenomenon and can behave differently depending on several conditions.

The plant-available potassium (K, O) content in the soil did not change significantly when the lower ash dose of 2 t/ha was applied, but increased significantly when 5 t/ ha was applied, which could indicate the need for a higher dose. In an incubation experiment, a 1% ash addition was usually not sufficient to significantly increase the plantavailable nutrient content, but a 5% ash addition resulted

in a significant increase in the plant-available nutrient pool (Ochecova et al., 2016). Wierzbowska et al. (2020) found that soil concentrations of available forms of K_2O increased in parallel with increasing ash doses. Similarly, Gibczyńska et al. (2014) and Füzesi et al. (2015) found that the amounts of plant-available K in the soil increased with the application of biomass ash. However, Saletnik et al. (2018) analyzed the uptake of ions in miscanthus when fertilized with different combinations of biochar and biomass ash and found that among the analyzed ions, the highest concentration in the biomass of giant miscanthus was found for potassium – both after the first and after the second year of cultivation. Uptake by the plants could therefore be one of the main reasons for the change in contribution only at the higher dose applied.

CaCO₃ levels increased significantly with the application of ash doses, even at a lower ash dose of 2 t/ha. This effect could be due to the high CaCO₃ content in the applied ash as well as the high alkali content of the biomass ash with a pH $_{\text{KCl}}$ of 12.84. Calcium is an important component of biomass ash and its release to the soil increases its availability in the soil (Shi et al., 2016). Mercl et al. (2016) found that the exchangeable Ca content in soil increased with the application of biomass ash. Omil et al. (2011) found that treatment with ash increased the content of carbonate lime in the soil. The increase in available Ca in the soil has an effect on the formation of calcium carbonate in the soil. With increasing pH and excess Ca, CaCO₂ (lime) is formed. Even if the change in soil pH was not significant, a higher calcium carbonate content in the soil contributes to the increase in soil pH.

Biomass ash has the potential to enrich the soil with important macronutrients and improve the pH value of the soil. In addition, the application of ash also improves other soil properties such as increasing available micronutrients (Gibczyńska et al., 2014), dehydrogenase activity, electrical conductivity (Quirantes et al., 2016) and CEC (Shi et al., 2016). However, the behaviour of biomass ash in soil is complex and depends on a variety of factors.

CONCLUSION

Although biomass ashes are currently classified as waste, they can provide valuable resources as alternative raw materials in the circular economy, provided that their utilization is regulated by an appropriate legal framework. The study found that the application of biomass ash led to a significant increase in CaCO₂ and K₂O content. An increase in pH was found although it was no significant. The effects on the content of plant-available phosphorus in the soil decreased after ash application. It was concluded that the behavior of biomass ash is a complex phenomenon and requires further investigation. Research can continue with monitoring other soil properties along with crop yield assessment for plant uptake. It can also be extended to monitoring the unfavorable aspect of applying higher doses of ash as these could lead to excessive amounts of nutrients or heavy metals entering the soil to better understand the nutrient dynamics and behavior of biomass ash. Similarly, research can be coupled under different agroecological conditions, including different soil and vegetation types. Such research will help to develop practices for utilization and provide specific recommendations for practical use. Appropriate use of biomass ash would fit into the spheres of the circular economy and contribute to better environmental health. The research is only preliminary in terms of the duration of ash application to the soil. However, the role of biomass ash use in enhancing economic and environmental value is recognized. In the future, research can be extended to the growth parameters and yield of bioenergy crops to understand nutrient uptake and accurately predict the behavior of biomass ash. Extending the research to different agro-ecological conditions, soil and vegetation types with a cost-benefit analysis may be beneficial to provide recommendations for practical use. Investigating biomass ash in combination with nature-friendly products could also further improve its effectiveness as a substitute for artificial fertilizers and as a soil conditioner, which needs to be investigated in more detail in the future.

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