

POTENTIAL IMPACT OF BIOENERGY WASTES AS FERTILIZER SUPPLEMENT ON SOIL PROPERTIES

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Wastes materials produced from bio-energy plants such as ash, slurry etc., may be problematic if they are not safely disposed. The wastes utilization as soil amendments is a viable approach as these are rich source of mineral nutrients. Therefore, the present experiment was conducted to evaluate the nutrient release and changes in physicochemical properties of soil after incubation with a different combination of bio-energy residues. Two variants of sugarcane bagasse ash, (A) combustion ash from sugarcane industry and (G) ash from gasification plant were mixed in 1:1 ratio with wheat straw biochar (B) and dairy manure digestate (D) @ 10, 15 and 20 t ha⁻¹. Soil samples were taken after 15, 30, 45, and 60 days of incubation and analyzed for soil physico-chemical and biological parameters. Various amendments significantly influenced the soil pH, electrical conductivity, soil respiration, P, and K contents at each sampling interval. The P and K contents in soil amended with digestate+ combustion ash were significantly increased after 60 days of incubation compared to their initial soil contents. Ammonium contents were increased up to 15 days of incubation and then decreased while nitrate contents increased in the same treatment. While macro-aggregate (77 g kg⁻¹) and organic carbon contents (1.40%) were maximum in biochar + combustion ash treatment. The application of bioenergy wastes improved respiration, soil organic carbon, soil P and K contents. The study warrants that these wastes combinations should be tested under field conditions for further recommendations.

Keywords: Digestate, biochar, bagasse ash, soil properties.

INTRODUCTION

The global population is increasing and as per estimates; it will increase up to 9 billion by 2050. Maximum increase is expected in developing countries particularly in South Asia and Africa. A big challenge for these developing economies is to fulfill ever-increasing food and energy demand. In addition to this, urbanization has increased significantly particularly in Pakistan, as the urban population has risen from 36% in 1995 to 54% in 2018 (DEAS, 2018). This major shift from rural to the urban population, improved lifestyle and recent economic development, has further increased energy demands.

The Government of Pakistan is promoting renewable energy resources including bioenergy. Abundant biomass resources of the country include fuel wood, agricultural residues, animal dung and municipal solid waste. Biomass energy referred to as bioenergy has emerged as a promising renewable energy source and has an enormous potential to fill the demand-supply gap of 6620 MW (Javed *et al.*, 2016), and to decrease the oil import bill.

Bioenergy plants through thermochemical and biochemical reactions convert these biomass resources to bioenergy. During operation, a significant amount of waste (such as ash, slurry, etc as a byproduct) is produced which may be

problematic, if they are not disposed off safely. Usually, the waste is either spread on the uncultivated arable land in close vicinity of the industry or landfill, ponds, and rivers. Inevitably, it may pollute the environment and encroach upon vast productive land. Economic issues, unavailability of land for waste (ash) disposal, and cost on landfilling, environmental concerns have forced to search for alternate solutions (Cabral *et al.*, 2008).

As the raw material (biomass) in bioenergy plants is organic, the byproducts/waste has sufficient quantities of mineral nutrients (P, K, Ca and Mg in ash) and some of the organic matter (slurry), thus can be utilized as a soil amendment (Gonfa *et al.*, 2008; Thind *et al.*, 2012). The positive effect of ash (Bagasse ash from sugar industries), slurry and biochar, also include improvement in soil physical conditions such as aggregate stability, porosity, water holding capacity and aeration (Dhindsa *et al.*, 2016; Webber *et al.*, 2017) and is an appropriate strategy of recycling the nutrients (Sales and Lima, 2010; Charles *et al.*, 2016, Sharma and Rajwar, 2016). Various wastes such as ash, biochar, slurry, have quite distinct characteristics (Elbasher *et al.*, 2017; Greenberg *et al.*, 2019) and may have a variable effect on soil health and crop production. For example, bagasse ash, has sufficient quantity of P, K, and Ca, but no organic matter, have a high surface area, while digestate (from anaerobic digestion) is organic and

may have sufficient quantities of N, P, K, and some micronutrients, hence first has potential for its use as a soil amendment and later as organic fertilizer (Funke *et al.*, 2013; Riseberg, 2015). Anaerobic digestate can be applied alone or sometimes after enrichment with compost/biochar to make its nutritional value high (Elbasher *et al.*, 2017; Udall *et al.*, 2017; Greenberg *et al.*, 2019).

Biochar is also a carbon rich solid byproduct of energy generation which is used as soil amendment and being explored for soil remediation (Kim *et al.*, 2017). The biochar produced at a lower temperature (300°C) has lower pH, greater nutrient retention ability due to having more carbon functional groups with oxygen and hydrogen that present more exchange sites after oxidation (Wu *et al.*, 2012). Biochar has a liming effect in acidic soil due to an increase in pH while in alkaline calcareous soil there is a decreasing trend in pH (Liu and Zhang 2012; Dume *et al.*, 2016). Co-application of biochar with mineral fertilizers speeds up the reactivity of biochar (Hagemann *et al.*, 2017).

Sugar industry is a major agricultural industry in Pakistan and produces about 0.26 M tons of bagasse ash annually (Kazmi *et al.*, 2017) which needs to be disposed of safely. Considering the potential of this bagasse ash as a soil amendment, we hypothesized that a combination of bagasse ash with other bioenergy wastes such as bioslurry, and biochar may improve its usefulness as soil amendment cum organic fertilizer. The combined application may not only improve soil physical properties but also will increase nutrient availability and soil fertility. So, the present incubation study was planned to evaluate the effect of different combinations of bioenergy residues on nutrient release within soil and soil physical properties.

MATERIALS AND METHODS

The current study was conducted at the Institute of Soil & Environmental Science, University of Agriculture Faisalabad, Pakistan.

Experimental materials: The soil used in the study was collected from the farm area of the Institute of Soil and Environmental Sciences, University of Agriculture, (31.391533°N and 73.014199°E), Faisalabad. The soil was alkaline and calcareous with sandy clay loam texture and the physicochemical parameters of the soil are given in Table 1. The anaerobic digestate (D) used in the study was collected from a biogas plant located in Ashraf Zia Textile industries, Khurrianwala Road Faisalabad, Pakistan. The biogas plant was operating under mesophilic conduction using cattle manure as feedstock. The one type of sugarcane bagasse ash (A) was collected from the Madina sugar mills Ltd, Chiniot, where bagasse was burnt under high combustion temperature to produce steam for boilers/turbines resulting in the production of ash at the bottom of the boiler. The second type of ash (G) was collected from the gasification plant of PARS campus, University of Agriculture, Faisalabad in 2017. The wheat straw biochar (B) prepared at 300°C pyrolytic temperature, was taken from the lab of the Institute of Soil & Environmental Science, University of Agriculture Faisalabad, Pakistan. The nutrients contents of the digestate and sugarcane bagasse ash and biochar are presented in Table 2 which showed that bagasse ash was enriched with high P contents and K contents were high in both bagasse ash and digestate.

Incubation study: The interactive influence of bioenergy by-products on the physicochemical and biological properties of soil by incubating soil with four wastes (mixed at 1:1 ratio) at three rates i.e., 10, 15 and 20 t ha⁻¹. Twelve treatments: DA₁, DA₂, DA₃, DG₁, DG₂, DG₃, BA₁, BA₂, BA₃, BG₁, BG₂, and BG₃ were tested taking in consideration zero day soil as control. Description of the treatments is given in Table 3. All amendments were mixed well in 1 kg soil then this soil was distributed into four plastic cups each with 250 g soil and incubated at 25 °C. Paraffin films were used to cover cups and maintained moisture at field capacity (An *et al.*, 2015). Soil sampling was collected after 15, 30, 45, and 60 days of incubation.

Table 1. Basic properties of soil used in the experiment.

Parameters (symbol)	Unit	Value	Protocol
pH		8.10±0.03	McLean, 1982
Electrical Conductivity (EC)	dS m ⁻¹	1.93±0.02	McLean, 1982
Bulk density (lb)	g cm ⁻³	1.45±0.04	Blake & Hartge, 1986
Organic Carbon (OC)	%	0.37±0.05	ImamulHaq & Alam, 2005
Phosphorus (P)	mg kg ⁻¹	8.14±0.12	Jackson, 1962
Potassium (K)	mg kg ⁻¹	180±12.0	Rowell, 1994
NO ₃ -N	mg kg ⁻¹	5.11±0.09	Keeney & Nelson, 1982
NH ₄ -N	mg kg ⁻¹	4.17±0.02	
Particle size distribution			Bouyoucos, 1962
Sand	%	35.8±0.06	
Silt	%	38.5±0.02	
Clay	%	25.7±0.05	

Table 2. Nutrient status of bioenergy by-products.

Parameters	pH	Nitrogen (N)	Phosphorus (P)	Potassium(K)
Unit		%	% %	
Protocol	(Rowell, 1994)	(Jackson, 1960)	(Sultanpour& workman, 1979)	
Digestate	8.10±0.01	2.30±0.04	0.58±0.03	0.88±0.03
Biochar	7.71±0.16	1.38±0.62	0.26±0.06	3.00±1.47
Combustion Ash	9.1±0.03	-	4.97±0.14	2.98±0.09
Gasification Ash	9.7±0.16	-	0.38±0.03	0.17±0.02

Table 3. Description of treatments used in the study.

Treatment Abbreviation	Description
DA ₁	Anaerobic digestate + Sugarcane bagasse ash from combustion plant @ 10 t ha ⁻¹
DA ₂	Anaerobic digestate + Sugarcane bagasse ash from combustion plant @ 15 t ha ⁻¹
DA ₃	Anaerobic digestate + Sugarcane bagasse ash from combustion plant @ 20 t ha ⁻¹
DG ₁	Anaerobic digestate + Sugarcane bagasse ash from gasification plant @ 10 t ha ⁻¹
DG ₂	Anaerobic digestate + Sugarcane bagasse ash from gasification plant @ 15 t ha ⁻¹
DG ₃	Anaerobic digestate + Sugarcane bagasse ash from gasification plant @ 20 t ha ⁻¹
BA ₁	Wheat straw biochar + Sugarcane bagasse ash from combustion plant @ 10 t ha ⁻¹
BA ₂	Wheat straw biochar + Sugarcane bagasse ash from combustion plant @ 15 t ha ⁻¹
BA ₃	Wheat straw biochar + Sugarcane bagasse ash from combustion plant @ 20 t ha ⁻¹
BG ₁	Wheat straw biochar + Sugarcane bagasse ash from gasification plant @ 10 t ha ⁻¹
BG ₂	Wheat straw biochar + Sugarcane bagasse ash from gasification plant @ 15 t ha ⁻¹
BG ₃	Wheat straw biochar + Sugarcane bagasse ash from gasification plant @ 20 t ha ⁻¹

Analytical methods

Chemical parameters of Soil: Soil samples taken for the assessment of physicochemical and biological properties were air-dried, ground, and sieved through a 2 mm stainless steel sieve before analysis. Soil pH of soil saturated paste was determined using pH meter (Jenway 3510, Chelmsford, UK). While the electrical conductivity (EC) of extract from soil saturated paste was measured employing Conductivity/TDS Meter (Jenway 4510, Chelmsford, UK). Soil organic carbon was determined following Walkley & Black chromic acid wet oxidation method (AOAC, 2005). Briefly, oxidizable organic carbon in the soil is oxidized by 1N potassium dichromate (K₂Cr₂O₇) solution in concentrated sulfuric acid. Afterwards, the organic carbon was estimated by measuring the remaining unreduced dichromate through back-titrating with ferrous sulphate using diphenylamine as an indicator. The contents of extractable potassium were analyzed after soil extraction with 1N ammonium acetate at pH 7.0 using flame photometer (PFP-7 Jenway, Chelmsford, UK) (Rowell, 1994). The contents of available phosphorus were analyzing following Olsen's method on spectrophotometer (Corning 220, Corston Bath, UK) (Jackson, 1962). The contents of NH₄⁺ and NO₃⁻ contents in the soil were determined after extraction with 2M KCl. Then distillation was performed with MgO and Devarda's alloy for analyzing NH₄⁺ and NO₃⁻ respectively (Keeney and Nelson, 1982).

Physical Parameters of Soil: The method described in Tang *et al.* (2011) was employed for analyzing wet aggregate stability at each sampling. Moreover, macro-aggregates (250-2000 µm) and micro-aggregates (53-250 µm) were

characterized by the sieving method. Soil samples (50 g) were immersed for 5 min; the aggregates were then separated by moving sieve up and down (around 3 cm) by hand for 2 minutes. The aggregates remained on each sieve were collected, oven-dried at 105°C for 24 h, and weighed. Mean weight diameter of soil aggregates was calculated as an index of soil aggregate stability as follow

$$MWD = \sum_{i=1}^n A_{si} d_i$$

MWD=mean weight diameter (g kg⁻¹); *i*= 1, 2...*n* is the index of sieves; *A_{si}* is the corresponding weight of the aggregate fraction; *D_i*= mean diameter of the *i*th class

Microbial activity: Microbial activity was assessed by calculating total CO₂ evolved through microbial respiration. According to the modified method of Jain *et al.* (2003) the evolved CO₂ was trapped in the viol contained 0.1N NaOH solution. The alkali was replaced at each sampling time. The excess of alkali was titrated back with standard HCl to estimate CO₂-C liberated from the soil using the following equation:

$$\text{Evolved CO}_2 = (B-V) \times N \times E$$

Where, V was the volume of acid required for titration of alkali, B was acid volume used for blank. N was the normality of acid 0.1N and E was the equivalent weight of CO₂. The carbon emission is expressed as mg wk⁻¹ kg⁻¹ of soil (Hossain *et al.*, 2017).

Statistical Analysis: Statistical analysis was performed using completely randomized design (CRD) with two factors i.e., type of amendment and incubation time (12 amendments × 4

days \times 3 replications). Significant differences in the main effects were further analyzed through pairwise comparison under the Tukey HSD test. Moreover, Pearson correlation matrix was calculated to compare the effects of different parameters.

RESULTS

Soil pH and electrical conductivity: The soil used was alkaline in nature and the addition of various treatments initially (up to 15 days) increased soil pH, having a maximum value of 9.49 in treatment with combined application of biochar and gasification ash (Fig. 1a). It decreased with time and buffered to its original soil pH value at the end of incubation (60 days). The decrease in pH at a later stage was higher in treatments receiving digestate compared to biochar applications. Similarly, electrical conductivity (EC) of soil differed among treatments significantly ($P < 0.001$) and it increased with the incubation period. Maximum EC (3.15 dS m^{-1}) was observed in DA3 followed by DA2 and DG3 (3.10 dS m^{-1}) at 60 days after incubation (Fig. 1b). Digestate and combustion ash (DA3) showed a 16% increase in EC after 60 days of incubation when compared to control/zero day soil (1.93 dS m^{-1}).

Available nutrient concentration in soil: Ammonium (NH_4^+) concentration was increased initially in various treatments compared to control/zero-day soil (4.17 mg kg^{-1}) and reached to maximum (20.3 mg kg^{-1}) in treatment receiving digestate+ combustion ash @ 20 t ha^{-1} at 15 days of incubation (Fig. 2a). The release of NH_4^+ concentration decreased significantly in treatment DA3 after 60 days of incubation when compared with 15 DAI (days after incubation). A similar trend of decrease in NH_4^+ concentration was found in all treatments. Contrary to NH_4^+ , the NO_3^- contents were increased with time in all treatments, and a maximum concentration of 17% was found in DA3 and DG3 at 60DAI over 15DAI. Among combustion ash amendments, more increase was found with digestate compared to biochar with time. Similarly, gasification ash along with digestate DG3 caused a 10% increase in nitrate concentration as compared to BG3 at 60DAI. Higher nitrate concentration (20.3 mg kg^{-1}) was found in DA3 and DG3 on 60 days and the minimum was found with biochar amendments either with combustion ash or gasification ash (Fig. 2b).

Available P concentrations (Olsen P) increased in all treatments and a higher level observed in treatments receiving higher doses and longer incubation period irrespective of the amendments. The interactive effect of days and treatment was not significant (Fig. 3a). Potassium concentration also increased in treatments receiving wastes compared to control. A maximum potassium release of 36% over zero-day soil was observed in DA3 after 60 days of incubation. The minimum release was found in biochar and gasification ash applications. Combined application of digestate and combustion ash (DA3)

caused 6.5% more release in potassium contents over biochar and combustion ash (BA3) at 60 days after incubation (DAI). The minimum release was found in gasification ash treatments along with biochar BG1 at 60 DAI (Fig. 3b).

Soil respiration and organic carbon contents: Treatments differ significantly (Fig. 4a) for soil organic carbon contents. The maximum organic carbon contents (1.40 %) were found in treatment with biochar + gasification ash at 15 days after incubation. There was a rapid decrease in organic carbon contents in the case of digestate as compared to biochar amendments with increasing days of incubation (Fig. 4).

The soil respiration was measured in terms of CO_2 evolved during the period. Maximum CO_2 ($372 \text{ mg wk}^{-1} \text{ kg}^{-1} \text{ soil}$) was evolved in digestate treatment when applied along with combustion ash @ 20 t ha^{-1} at 60 DAI (Fig. 4b). The lowest gas content ($64 \text{ mg wk}^{-1} \text{ kg}^{-1} \text{ soil}$) was recorded at 15 DAI with BG1 in biochar + gasification ash applied @ 10 t ha^{-1} . The maximum respiration was found in digestate treatment applied with combustion ash (DA3) compared with combination of digestate and gasification ash (DG3) at 60 DAI. The minimum release was found in treatment with combination biochar and gasification ash (BG1) at 60 days incubation period.

The combined effect of bioenergy residues on the soil physical properties: The application of these waste materials improved soil structure as indicated by the aggregate formation (Fig. 5a). The addition of digestate significantly improved macro-aggregate formation and highest contents (77, 70, and 70 g kg^{-1}) were observed in the treatments where combustion ash was applied with digestate @ 10, 15, and 20 t ha^{-1} after 45 days of incubation. The 17% increase in aggregate formation in DA3 was found over DG3 after 45 days of incubation. There was an increase in macro-aggregate formation in the start in BA3 after 15 days of incubation, reached at peak (54 g kg^{-1}) after 30 days of incubation, and reached to maximum (60 g kg^{-1}) after 60 days of incubation. The temporal changes in micro-aggregate formation were also observed (Fig. 5b), indicating maximum micro-aggregate formation in BG3 (734 g kg^{-1}) after 15 days of incubation. Minimum micro-aggregate (426 g kg^{-1}) was found in DA1 after 60 days of incubation. In the case of biochar amendments, the maximum micro-aggregate formation was observed after 15 days of incubation either with combustion ash or gasification ash.

DISCUSSION

The improvement in soil physicochemical and biological properties by the combined use of different bioenergy wastes was investigated in the current incubation study. Combined use of different bioenergy wastes increased soil pH up to first 15 days of incubation; however, the soil pH was lowered down to its initial value at 60 days (Fig. 1a).

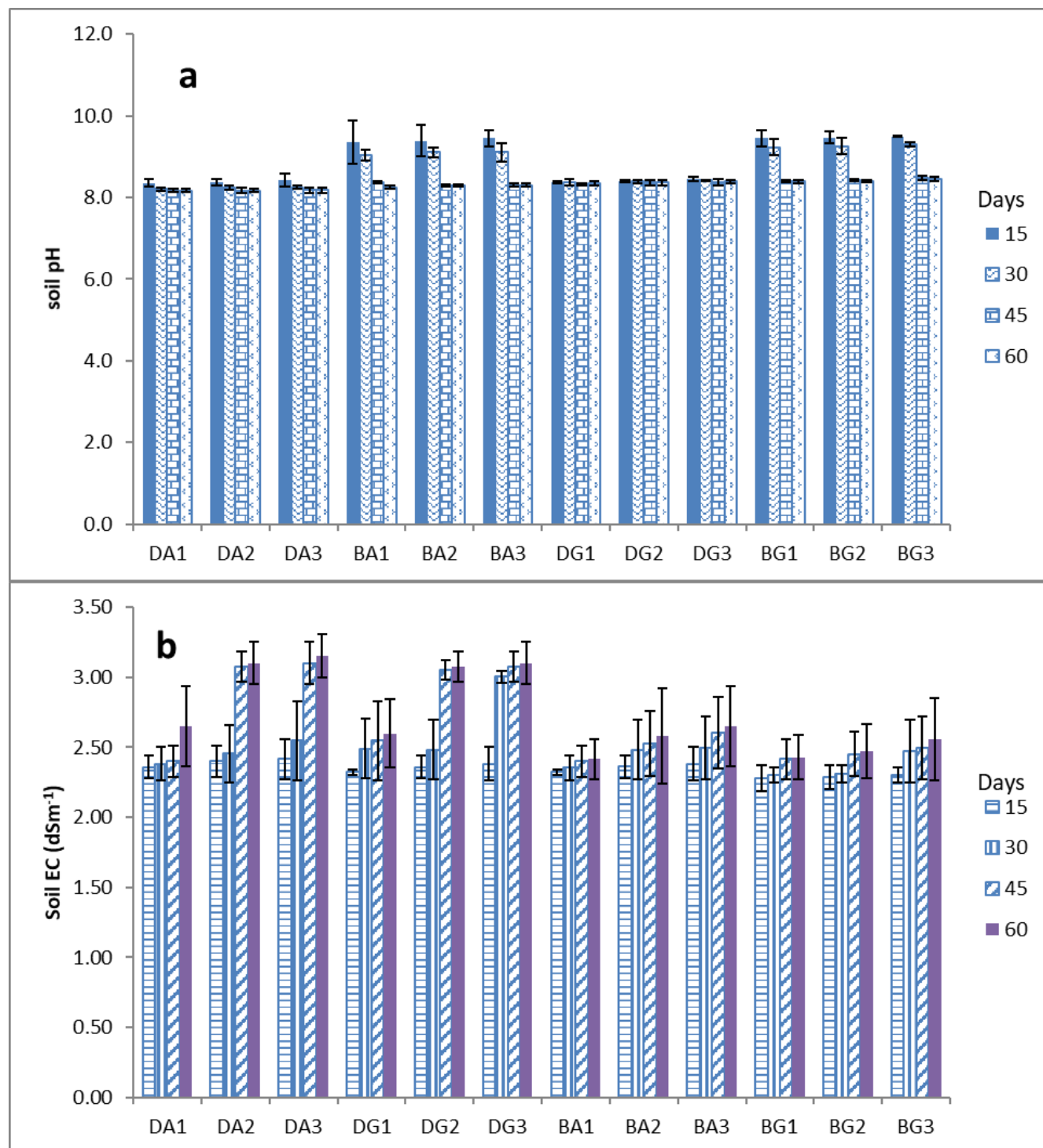


Figure 1. Soil pH (a) and electrical conductivity (b) as affected by the application of bioenergy residues after 60 days of incubation. Whereas DA1, DA2 and DA3 (digestate and boiler ash @ 10, 15 and 20t/ha); DG1, DG2, DG3 (digestate and gasification ash @ 1.0, 1.5 and 2.0t/ha); BA1, BA2, BA3 (biochar and boiler ash@10, 15 and 20t/ha); BG1, BG2, BG3 (biochar and gasification ash @10, 15 and 20t/ha). A least significant difference for pH is ± 0.43 and EC is ± 0.47 .

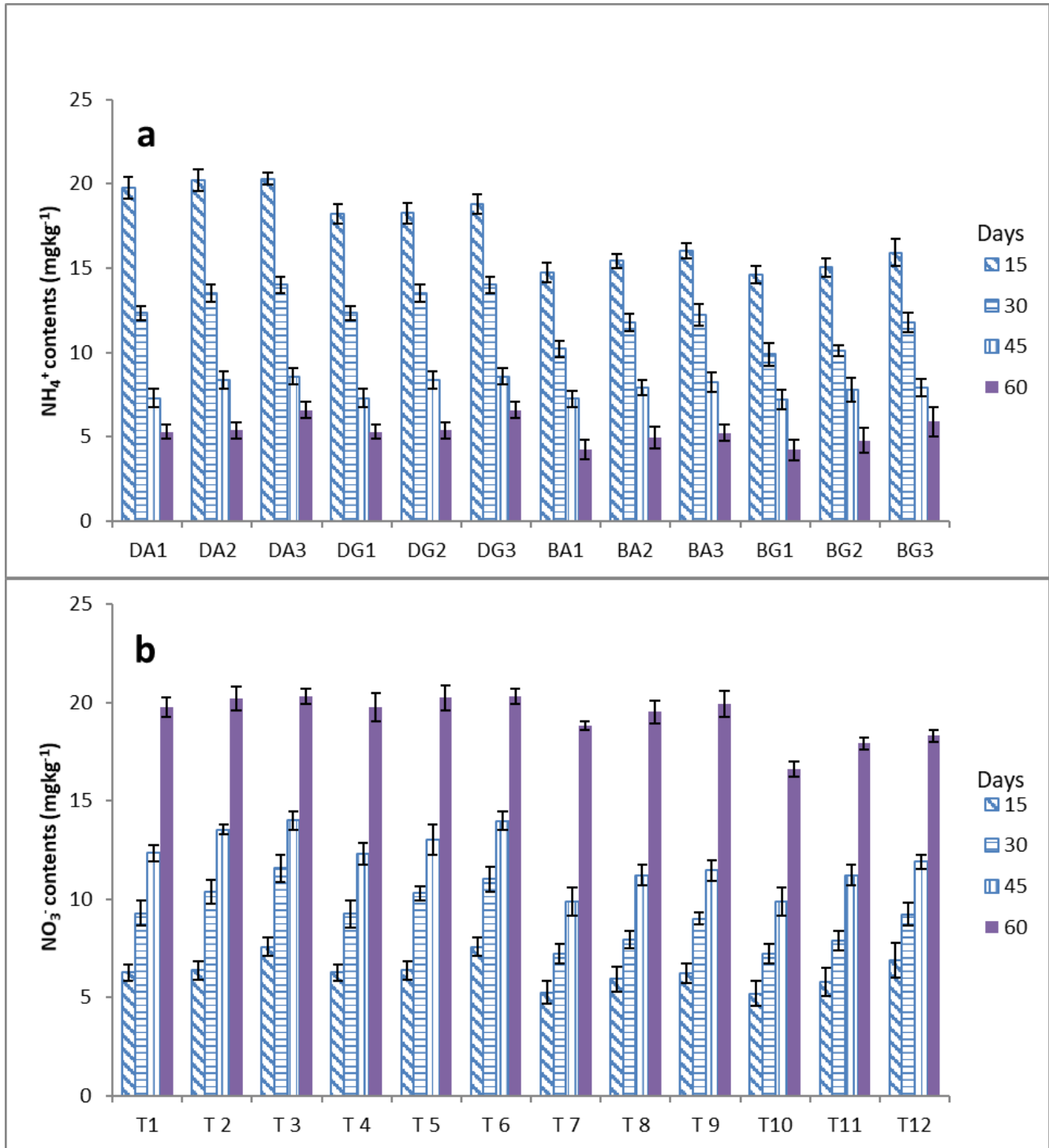


Figure 2. NH₄-N (a) and NO₃-N (b) content of the soil as affected by the application of bioenergy residues during 60 days of incubation. Whereas DA1, DA2 and DA3 (digestate and combustion ash @ 10, 15 and 20 t ha⁻¹); DG1, DG2, DG3 (digestate and gasification ash @ 10, 15 and 20 t ha⁻¹); BA1, BA2, BA3 (biochar and combustion ash @10, 15 and 20 t ha⁻¹); BG1, BG2, BG3 (biochar and gasification ash @10, 15 and 20 t ha⁻¹). A least significant difference for NH₄⁺ is ±1.68 and ± NO₃⁻ is 1.69.

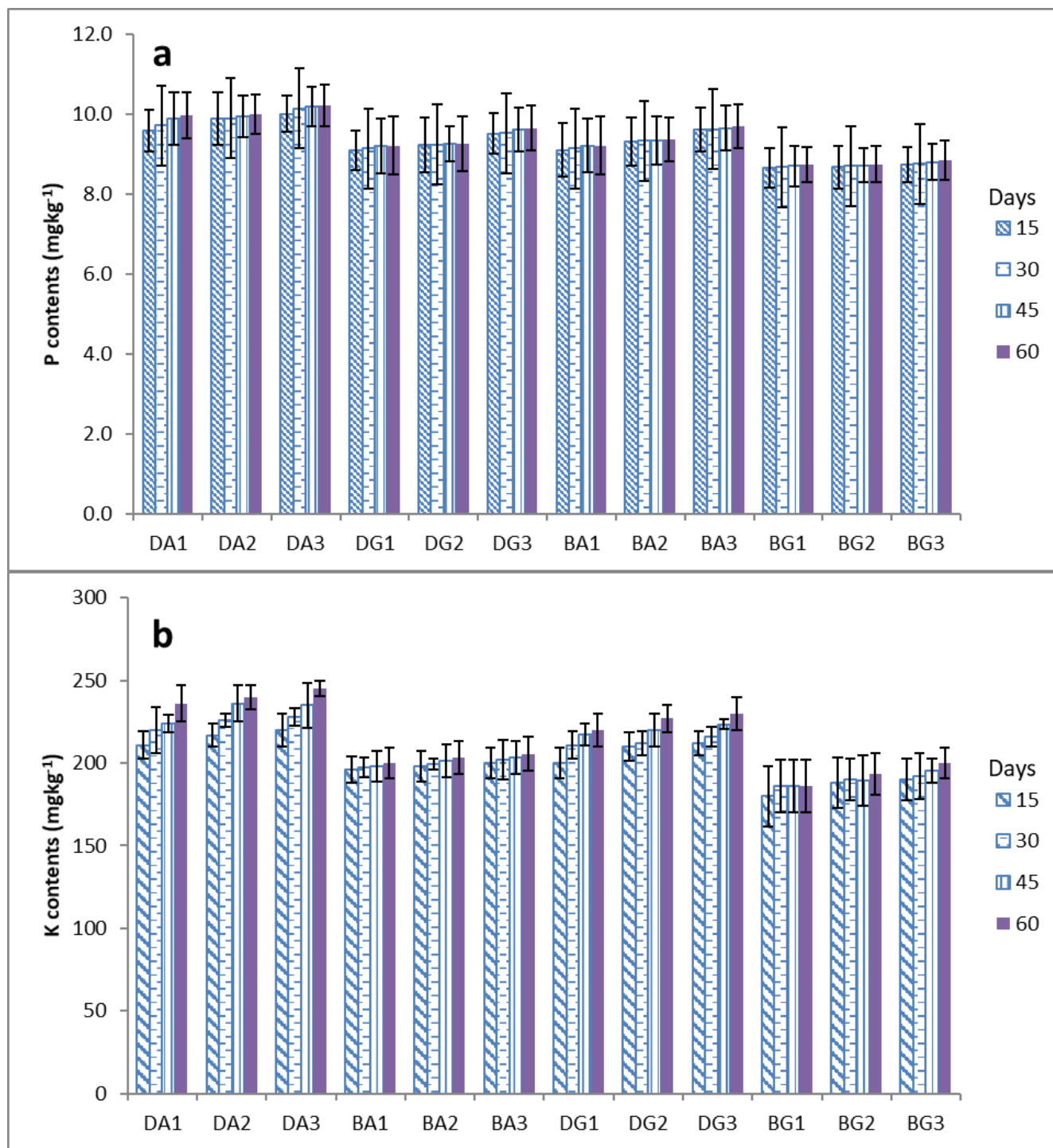


Figure 3. Phosphorus (a) and potassium (b) content of the soil as affected by the application of bioenergy residues during 60 days of incubation. Whereas DA1, DA2 and DA3 (digestate and combustion ash @ 10, 15 and 20 t ha⁻¹); DG1, DG2, DG3 (digestate and gasification ash @ 10, 15 and 20 t ha⁻¹); BA1, BA2, BA3 (biochar and combustion ash @10, 15 and 20 t ha⁻¹); BG1, BG2, BG3 (biochar and gasification ash @10, 15 and 20 t ha⁻¹). A least significant difference for P is ± 1.78 and for K is ± 27.19 .

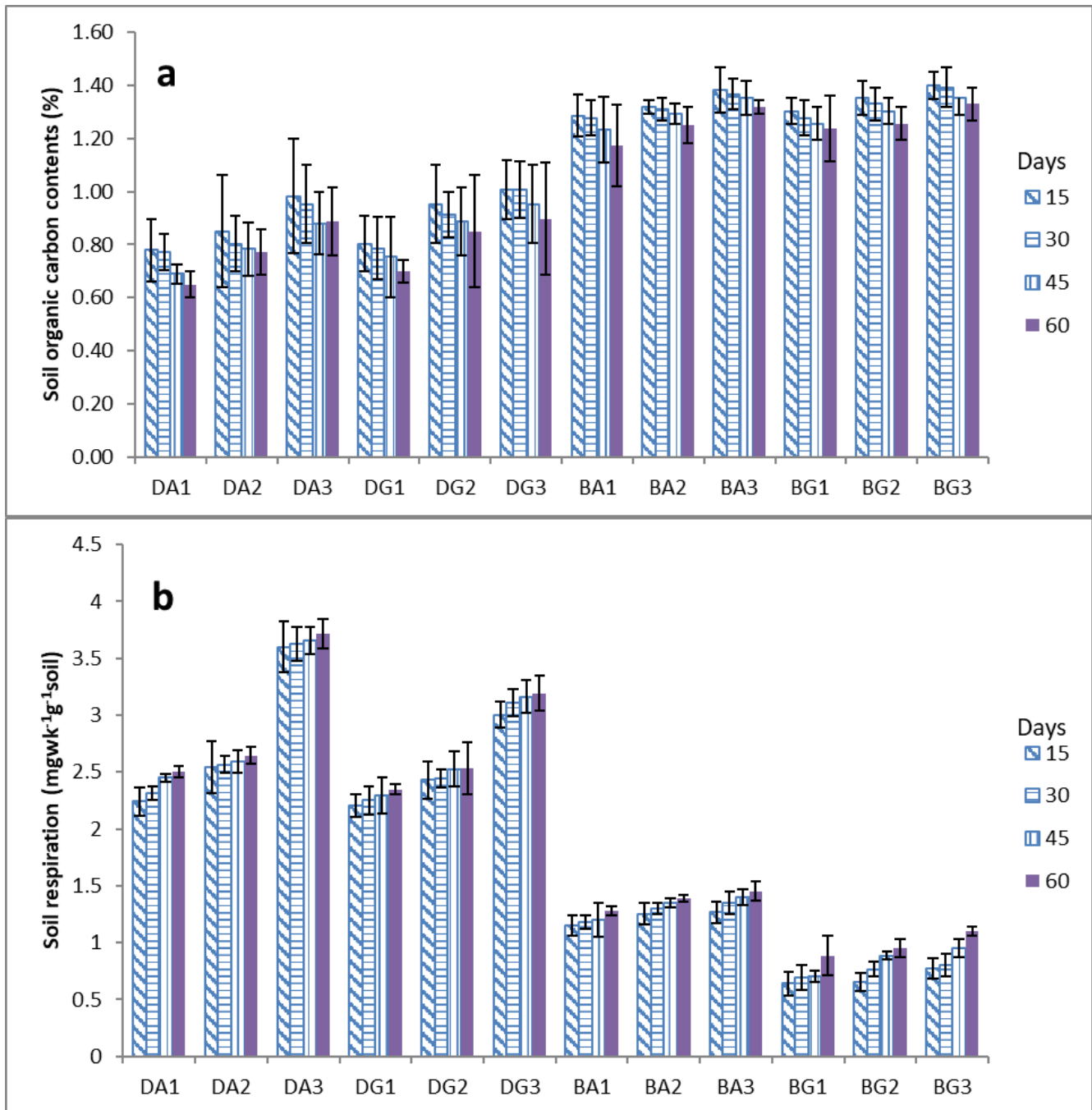


Figure 4. Organic carbon (a) and Soil respiration (b) of soil as affected by the application of sugarcane bioenergy residues during 60 days of incubation. Whereas DA1, DA2 and DA3 (digestate and combustion ash @ 10, 15, and 20 t ha⁻¹); DG1, DG2, DG3 (digestate and gasification ash @ 10, 15 and 20 t ha⁻¹); BA1, BA2, BA3 (biochar and combustion ash @10, 15 and 20 t ha⁻¹); BG1, BG2, BG3 (biochar and gasification ash @10, 15 and 20 t ha⁻¹). A least significant difference for OC is ± 0.36 and soil respiration is ± 9.19 .

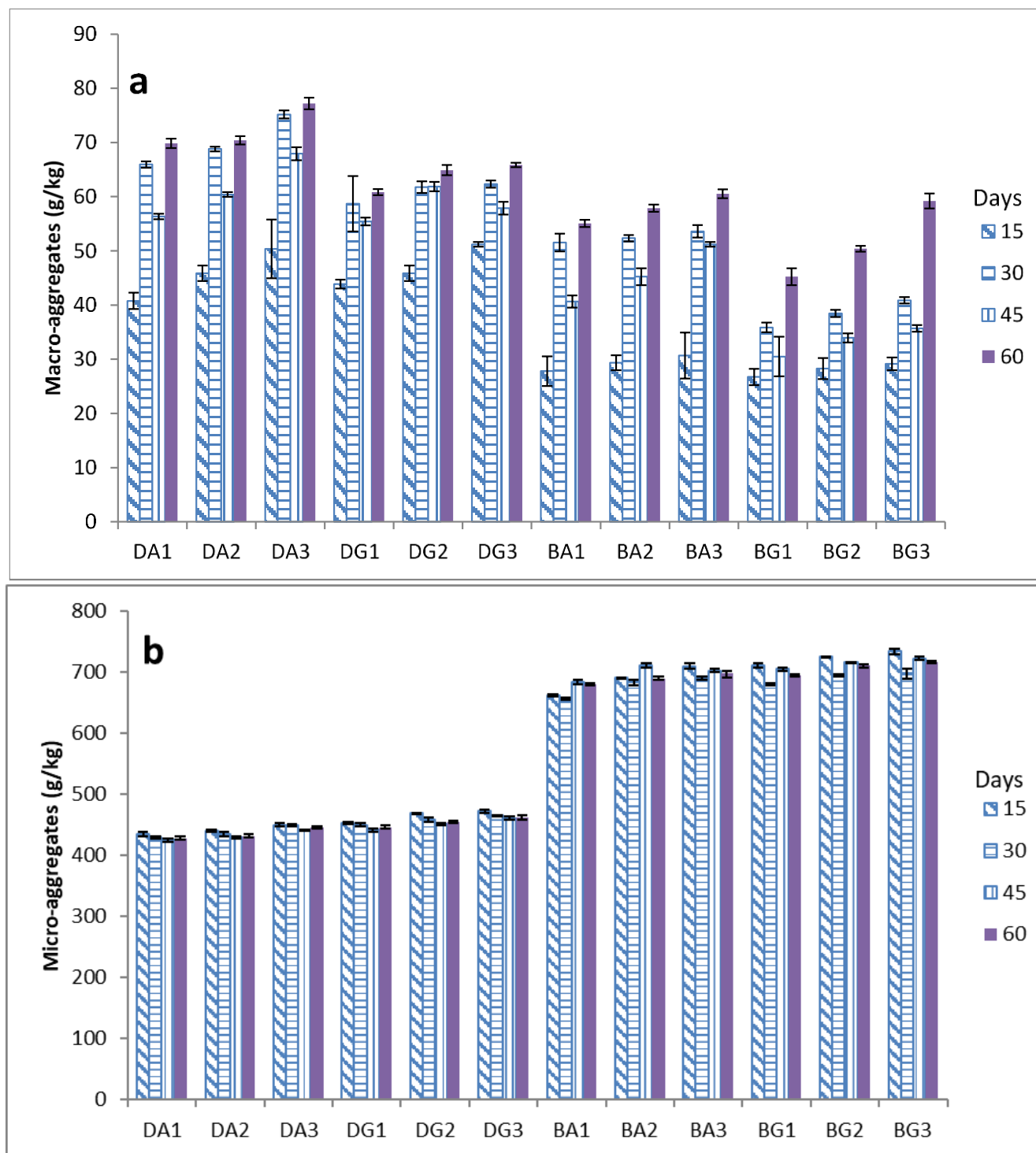


Figure 5. Formation of macro-aggregates (a) and micro-aggregates (b) as affected by the application of sugarcane bioenergy residues during 60 days of incubation. Whereas DA1, DA2 and DA3 (digestate and combustion ash @ 10, 15 and 20 t ha⁻¹); DG1, DG2, DG3 (digestate and gasification ash @ 10, 15 and 20 t ha⁻¹); BA1, BA2, BA3 (biochar and combustion ash @10, 15, and 20 t ha⁻¹); BG1, BG2, BG3 (biochar and gasification ash @10, 15 and 20 t ha⁻¹). A least significant difference for macro-aggregate is ± 5.47 and micro-aggregate is ± 10.2 .

The ash generally has more oxides and hydroxide contents, which might enhance the soil pH initially (Barthod *et al.*, 2018; Cruz-Paredes *et al.*, 2017). Moreover, wheat straw biochar produced at 300°C pyrolytic temperature has lower pH, but when it application along with ash @ 20 t ha⁻¹ incremented pH to 9.5 after 15 days of incubation. The decrease in pH at later stages is due to the buffering capacity of the soil (Cabral *et al.*, 2008; Naeem *et al.*, 2017). Soil incubation with bioenergy wastes significantly uplifted electrical conductivity (EC) of the soil (Fig. 1b). More increase in EC was observed in the soils receiving blend of digestate and ash compared with biochar and ash combined application, which was due to high salts contents and nutrient release from the digestate (Yu *et al.*, 2010; Voelkner *et al.*, 2015).

The soil incubated with mix of digestate and ash has higher contents of NH₄⁺ at initial stage of incubation compared with the soil receiving mixture of biochar and ash (Fig. 2a). Moreover, it was observed that the contents of NH₄⁺ decreased and nitrate contents were increased with the increase in incubation time because the N incorporated into the soil with digestate go through various transformations such as mineralization, immobilization, nitrification (Möller, 2015). Normally, the mineral N in the digestate is not immobilized in soil because of the low carbon C/N ratio and the content of highly stable organic substances (Gutser *et al.*, 2005). Furthermore, the NH₄⁺ undergo an immediate process of nitrification upon incorporation into the soil which triggered decrease in ammonium contents and increase in nitrate contents at incremented incubation period in the

current study (Johansen *et al.*, 2013; Song *et al.*, 2018). Strong negative ($r=0.81$) correlation between NH₄⁺ and NO₃⁻ concentration (Table 4) also indicated the nitrification (Olowoboko *et al.*, 2018).

Higher contents of P were observed in soil supplemented with combination of digestate and combustion ash compared with soil receiving biochar and ash integration (Fig. 3a). Organic amendments may influence P availability in the soil either directly, through an application of inorganic and organic P compounds, or indirectly, by prompting the activity of soil microorganisms as a consequence of the supply of organic C (Bachmann *et al.*, 2014). Moreover, anaerobic digestates can be a source of protons (H⁺) and lead to soil acidification, thus causing release of nutrients (Makadi *et al.*, 2012). Herein, the soil was characterized by high amounts of Ca and pH therefore, it is very likely that organic acid anions from organic matter of digestate might compete for sorption sites with Pi (ligand exchange), thereby reducing the P adsorption capacity by Ca compounds thus increasing its availability (Barlog *et al.*, 2020). Moreover, the ratio between readily soluble P fractions and total P content in digestates was more than 70%, which was higher than in case of feedstock and other amendments like ash or biochar, therefore, the use of digestate increased contents of plant available P in the soil (Bachmann *et al.*, 2016). Both digestates and ash are considered a valuable source of K in soil (Zhao *et al.*, 2014; Zhang *et al.*, 2018). Herein, the increased plant-available K in soil after digestate and ash application comparing with biochar and ash treatments probably resulted from a stimulation of ash decomposition and/or ion exchange of

Table 4. Correlation matrix among different parameters

	EC	K	NH ₄	NO ₃	OC	P	SR	macro	micro
K	0.38* (0.0000)								
NH ₄ ⁺	-0.38 (0.0000)	-0.16 (0.0555)							
NO ₃ ⁻	0.57** (0.0000)	0.36* (0.0000)	-0.81 (0.0000)						
OC	-0.34 (0.0000)	-0.29 (0.0005)	-0.01 (0.8696)	-0.25 (0.0025)					
P	0.02 (0.8205)	0.36* (0.0000)	-0.03 (0.6926)	0.09 (0.3058)	-0.04 (0.6584)				
SR	0.54** (0.0000)	0.49** (0.0000)	0.14 (0.0842)	0.26* (0.0014)	-0.74 (0.0000)	0.10 (0.2237)			
Macro	0.05 (0.5388)	0.20 (0.0152)	-0.64 (0.0000)	0.42** (0.0000)	-0.86 (0.0000)	0.23* (0.0054)	-0.45 (0.0000)		
Micro	0.44** (0.0000)	0.25* (0.0022)	0.21* (0.0114)	0.17* (0.0450)	0.54** (0.0000)	-0.04 (0.6372)	0.89** (0.0000)	-0.67 (0.0000)	
pH	-0.36 (0.0000)	-0.55 (0.0000)	0.48** (0.0000)	-0.5 (0.0000)	0.28* (0.0007)	-0.33 (0.0001)	-0.29 (0.0005)	-0.29 (0.0004)	-0.08 (0.3107)

The correlation of parameters is shown at the significance level of $P < 0.01$, values given in Parenthesis showed computed P values. ** showed highly significant correlation if $r \geq 0.40$, * showed moderate correlation if $r = 0.2-0.3$, weak correlation if r value was between 0.3 – 0.4. Positive r-values showed that an increase or decrease in one parameter could shift the equilibrium and effect on the size of others

NH₄⁺/K⁺ in spaces between sheets of clay minerals (Chappell *et al.*, 2000; Möller *et al.*, 2015).

The soil organic carbon (SOC) is a good indicator of soil fertility and its concentration depends on management practices including the addition of mineral and organic manures (Abrar *et al.*, 2020). The improvement in SOC was more with the blend of biochar and ash compared with digestate and ash combined application (Fig. 4). This is because of inert portion of biochar and increased in soil microbial biomass by biochar application (Biederman and Harpole, 2014). Likewise, retention of native dissolved organic matter on the charged surface of biochar also causes increase in SOC (Deenik *et al.*, 2010). Moreover, SOC is related to organic matter, hence higher the organic C would result in higher microbial activity (Naeem *et al.*, 2017; Ashraf *et al.*, 2020). The higher soil respiration in the amended soils also supported this hypothesis that SOC improved the microbial activity (Fig. 4) which is directly related to mineralization of OC (Li *et al.*, 2018; Ashraf *et al.*, 2020). The sharp increase in soil respiration with digestate amended treatments compared to biochar (Fig. 4) might be attributed to increased organic carbon mineralization in these treatments. The lower respiration or microbial activity in biochar amended soils may be due to the recalcitrant nature of biochar and less organic carbon available for microbial respiration/activities (Karhu *et al.*, 2011). As the microbial activity was linked to organic carbon fraction, therefore, the ash did not play any direct role in microbial activity due to the paucity of organic carbon contents however; it promoted microbial activity when applied along with digestate by providing essential nutrients.

Bioenergy residues significantly improved soil structure by stabilizing aggregate formation. Macro-aggregates (250-2000 µm) had more water stability compared to micro-aggregates (<250 µm). Quality and amount of residue affected the formation and stabilization of aggregates (Bossuyt *et al.*, 2001; Six *et al.*, 2004; Abrar *et al.*, 2020). The maximum macro-aggregate formation was found in digestate and ash amended soil compared with the soil receiving blend of biochar and ash (Fig. 5). Micro-aggregates are water stable and enhance the physical structure of soil (Ou *et al.*, 2016). Maximum micro-aggregate formed with biochar+ ash amendment, where ash may have acted as a binding agent by filling the biochar's surface area at the molecular level and provided essential minerals for the formation and stability of aggregates (Liang *et al.*, 2006). There was a significant positive correlation among SOC and micro-aggregates (0.54, Table 4) which indicated SOC has a significant role in soil structure formation and its stabilization as well. More recalcitrant the nature of soil organic carbon contents, more stable will be the formation of micro-aggregates as found in the case of biochar. There were consecutive formation and breakdown of macro-aggregate in digestate+ combustion ash treatments during 60 days incubation period due to their short

life span and microbial decomposition of organic matter (Abrar *et al.*, 2020).

Conclusions: The combined use of bioenergy by-products not only improved soil quality but also improved soil fertility through increased nutrient release over time, better soil structure formation, and microbial growth. However, biochar mixed with sugarcane bagasse ash was a good stabilizing agent that retained nutrients for a longer time. Digestate mixed with sugarcane bagasse ash from industries proved to be the best combination of fertilizer for release of soluble nutrient over time, improve chemical and microbial properties through organic matter decomposition by microbes. Therefore, bagasse ash mixed with digestate when applied @ 20 t ha⁻¹ might be a good approach to supply plant-available nutrients and improve fertility of the soil. However, field-scale studies are direly needed for a better understanding of the effect of these waste products on soil health and crop production.

Conflict of interest: The authors declare no conflict of interest whether financial or relational during the preparation and submission of this work.

Acknowledgements: This paper is a part of Ph.D. thesis research of the first author. The authors are thankful to the Institute of Soil and Environmental Sciences, University of Agriculture Faisalabad, Pakistan for providing the required facilities for conducting the experimental work.

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[Received 21 Dec 2020; Accepted 22 Jun. 2021;

Published (online) 25 Jun 2021]