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Abstract: Soils in Qatar are relatively poor in fertility. Hence, imported top soils and soil enhancing materials are used to improve agricultural yields. Therefore, this work investigated the potential of using gas-to-liquid (GTL) biosludge as a soil conditioner. It sought to increase crop yields in an arid soil with positive environmental footprint in terms of fertilizer application savings, waste utilization and minimization of landfilling. A fodder crop, alfalfa (Medicago sativa), was grown under semi-controlled pot conditions for 12 months. The plant-growth media involved soil, soil + fertilizer, soil + 3% compost, and soil plus five (0.75 - 12%) biosludge contents. Pertinent properties of the soils, the resulting leachates, and plant growth parameters were analyzed at set periods. Biosludge content generally increased the total porosity and volumetric abundance of different pore types, which in turn affected plant performance, especially the plant height. Alfalfa yield in terms of plant height, aboveground fresh biomass weight and the number of tillers decreased with increasing biosludge content. Mixtures with 0.75 - 3% biosludge content showed comparable or better plant yield in contrast to the soil, fertilizer and compost controls. The concentration of chemical species in the leachate and plant biomass of biosludge treatments were either lower or similar to the fertilizer and compost controls. Regression modeling identified leachate phosphorus concentrations, soil iron concentration and clay content as the most influential variables for the aforementioned plant performance parameters. The results suggest that GTL biosludge could potentially enhance arid soil properties and improve alfalfa yields.

Effect of gas-to-liquid biosludge on soil properties and alfalfa yields in an arid soil

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Highlights

- Biosludge from wastewater of a gas-to-liquids plant improved arid soil properties.
- Compared different biosludge content treatments with use of fertilizers and compost.
- Alfalfa growth performance was relatively better with 0.75 - 3% biosludge content.
- Concentrations of chemical species in leachates and plant biomass were satisfactory.
- Leachate P concentration, soil Fe and clay contents mostly influenced plant growth.

Graphical Abstract

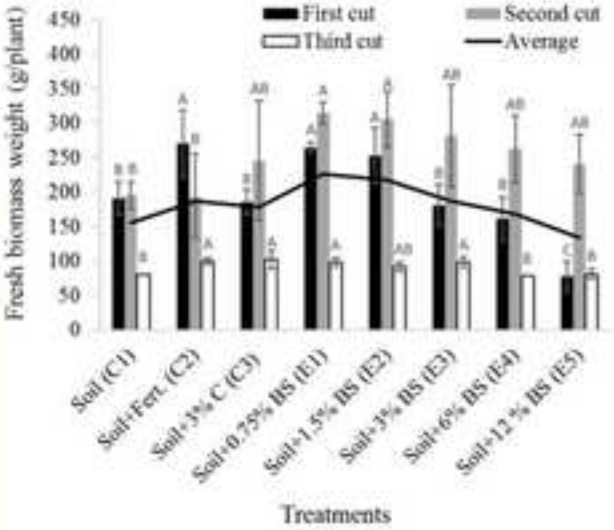
Biosludge from wastewater of Gas-to-Liquids (GTL) plant

Landfilling of biosludge

Minimize landfilling &
consider utilization

Land application of biosludge

Biosludge recycled for alfalfa production, by mixing (0.75 – 12%) with arid soils, for fertilizer savings



Mixtures with 0.75 - 3% biosludge content showed comparable or better plant yield in contrast to fertilizer and compost controls

Word count = 8,285 words

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Abstract

Soils in Qatar are relatively poor in fertility. Hence, imported top soils and soil enhancing materials are used to improve agricultural yields. Therefore, this work investigated the potential of using gas-to-liquid (GTL) biosludge as a soil conditioner. It sought to increase crop yields in an arid soil with positive environmental footprint in terms of fertilizer application savings, waste utilization and minimization of landfilling. A fodder crop, alfalfa (*Medicago sativa*), was grown under semi-controlled pot conditions for 12 months. The plant-growth media involved soil, soil + fertilizer, soil + 3% compost, and soil plus five (0.75 - 12%) biosludge contents. Pertinent properties of the soils, the resulting leachates, and plant growth parameters were analyzed at set periods. Biosludge content generally increased the total porosity and volumetric abundance of

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Keywords: alfalfa; arid soil; gas-to-liquid biosludge; plant growth parameters; porosity; soil conditioner.

1. Introduction

Agriculture in Qatar is limited to growing date palm and fodder crops in open fields, and selected variety of vegetables in greenhouses due to challenging soil and climatic conditions (Huda et al., 2018). Soils in Qatar are generally poor in fertility, low in water and nutrient holding capacity and high in leaching and water evaporation (Frenken, 2009). Consequently, the agricultural industry in Qatar is highly dependent on using imported top soils and soil enhancing materials including kaolinite clays and compost. The addition of soil enhancers or conditioners can modify some soil properties, especially water and nutrient holding capacities (Ahmedna et al., 2016). This helps mitigate the risk of water and nutrients depletion during the plant life cycle. Soil enhancers such as biosludge can improve the organic content of the soil and supply plant nutrients, which could lead to fertilizer application savings. Generally, biosludge is the solid organic waste matter produced by a wastewater treatment plant (WWTP) during wastewater treatment. It is mostly organic matter and it is rich in plant nutrients (macro and micro-nutrients). Biosludge is commonly used in agriculture in various parts of the world including the USA, Canada, Australia and Europe to increase soil nutrients and microbial activities (Cano Londoño et al., 2017; Miller-Robbie et al., 2015).

The addition of biosludge was reported to improve the availability of micronutrients in calcareous soils normally deficient in Fe and Zn (Laha and Parker, 2003). Biosludge contains several essential micronutrients for plants (e.g., B, Cu, Fe, Zn, etc), which are not provided by most conventional chemical fertilizers (Lu et al., 2012). Nevertheless, a major concern of land-applied biosludge is the transport of excessive nutrients such as nitrogen and phosphorus, which can cause eutrophication of surface waters (Paramashivam et al., 2017). Hence, biosludge should be applied based on crop phosphorus requirement. It has also been argued that repeated or heavy biosludge application rates may pose an environmental threat over time, especially as significant pH reductions can affect metal solubility (Lu et al., 2012). However, several studies lasting 10 - 15 years made contrary observations (Laha and Parker, 2003; Lu et al., 2012). Biosludge utilization as soil amendments has advantages of reducing fertilizer costs and adding organic matter to the soil, which improves soil structure and reduces surface runoff and erosion. The added organic matter, in turn, enhances crop yields. The challenges related to biosludge utilization include initial odors (which disappears eventually), the presence of certain metals, and potentially harmful pathogens – although not applicable for GTL biosludge as sewage water is not treated in the biotreater. More details of the benefits and concerns of land application of biosludge can be found in recent review papers (Kumar et al., 2017; Paramashivam et al., 2017).

The biosludge used in this study was sourced from an onsite WWTP in a GTL plant located in Ras Laffan Industrial City, North of Doha. GTL biosludge is a by-product of the GTL plant onsite-effluent treatment system and is mainly produced from process water from the GTL reactors as a reaction by-product and contains mostly organic acids and some alcohols. The WWTP produces around 15 - 18 tons/day of dry biosludge, resulting in an annual production of approximately 6,000 tons. Sewage water from the plant offices is not mixed with the GTL effluent but is sent off-site for treatment. Recycling the GTL biosludge as soil conditioner can potentially provide nutrients for plants and improve soil properties thereby increasing crop yields. Moreover, this can also reduce landfill-tipping fees alongside positive environmental footprint in terms of water and fertilizer application savings, waste utilization and landfill dependency minimization. Currently, the GTL biosludge is sent to landfill in Qatar but in some other countries, it is used as a source of nutrients. Further, due to the arid climate in Qatar, it is important to determine the right dosage for land application of the biosludge.

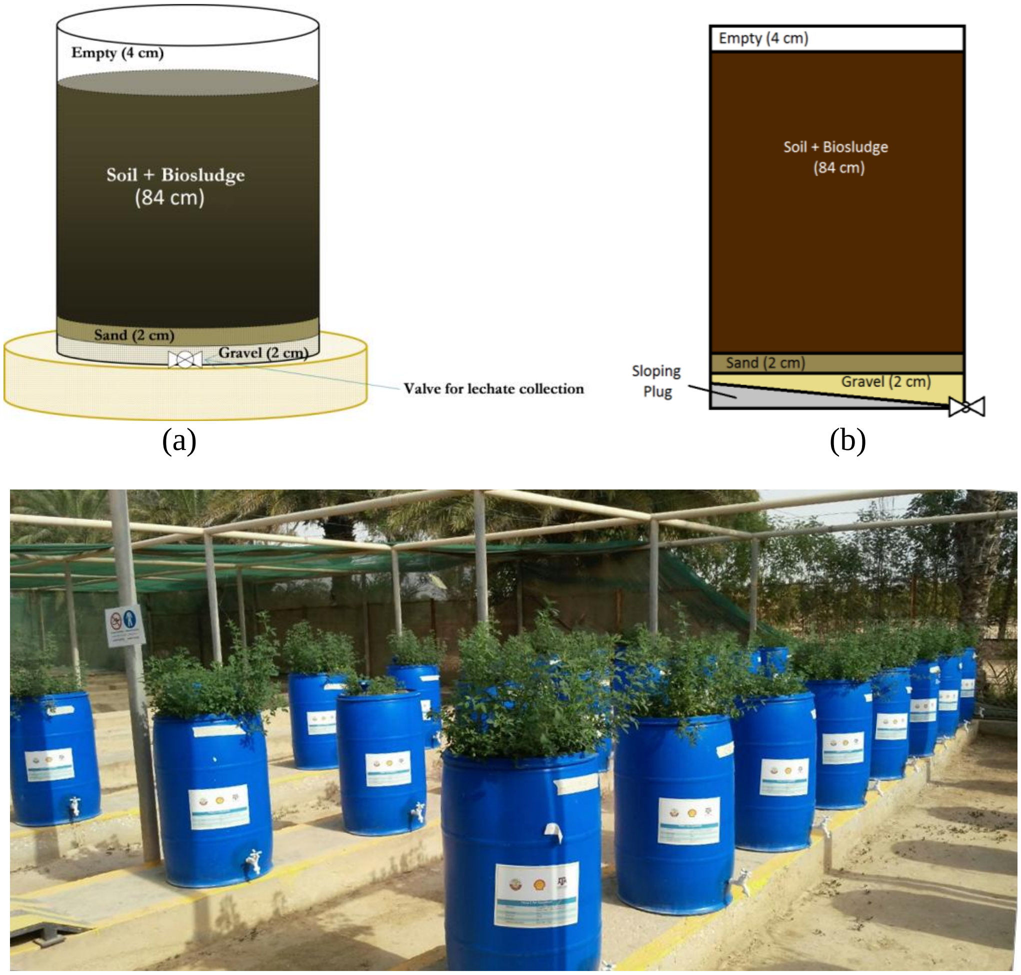
In light of the above, this research sought to understand the application rate of GTL biosludge on soils in Qatar and the impact of utilizing GTL biosludge as a soil conditioner on plant growth, soil properties and groundwater. It considered a fodder crop, namely, alfalfa (*Medicago sativa*) as a sort of 'worst-case scenario' as undesirable components can enter the food chain via this route. Alfalfa is a perennial leguminous plant known for its adaptability and high year-round dry matter yield. It is currently being cultivated in Qatar for fodder. The plant can grow in different soil, temperature and rainfall conditions through water content adjustment as water-logging is detrimental to its growth (Radović et al., 2009). Phosphorus has been identified as the nutrient needed in the largest quantity and most commonly in short supply for alfalfa production (Meyer et al., 2007). Other nutrients commonly in short supply include K, S, Mo and B, although fertilizer is less frequently or seldom required for these nutrients. The application of nitrogen fertilizer is seldom beneficial or results in an economic yield response as adequate nitrogen is usually provided by the symbiotic nitrogen-fixing bacteria (*Rhizobium meliloti* Dang.) that live in alfalfa root nodules (Meyer et al., 2007). The findings of two separate studies have shown that alfalfa requires about 27 kg/ha of N, 7 kg/ha of P_2O_5, and from 15 - 33 kg/ha of K_2O (Agafonova, 2008; Katalin, 2011).

Majority of previous studies involving the land application of biosludge were carried out on arable soils. In contrast, the present study considers an arid soil for which there is a paucity of literature. Moreover, the impact of GTL biosludge on plant, soil and the resulting leachate is not yet researched. This study utilized mixtures of a typical Qatari farming soil and different (0.75 – 12%) GTL biosludge contents for growing alfalfa in semi-controlled pot experiments. Analyses of pertinent properties of the soils at different plant growth stages were done using several materials characterization equipment. Plant growth parameters and leachates collected from the pots were also analyzed at different growth stages. The aim of the study was to investigate the effects of GTL biosludge addition to soil on alfalfa growth and soil properties, as well as potential risks to groundwater. The study also sought to investigate the possibility of bioaccumulation of undesirable components in the plant in unacceptable levels.

2. Materials and methods

2.1. Materials for pot experiments

The experiment was conducted in cylindrical pots, 92 cm long and 52 cm in diameter, with a valve connected at the bottom to permit leachate collection. Gravel (> 2 mm) and fine sand were used in the bottom layer to avoid clogging, and to facilitate water movement as well as leachate collection from the bottom of the pot as illustrated in Figure 1. A slope of 6-7 degrees was created at the bottom of the pot by filling it with glass-reinforced plastic at a slight tilt. This enabled direction of the leachate to the water collection valve (Figure 1b).



(a) (b)

(c)

Figure 1. Details of the pot experiments showing (a) front view of the pot and materials inside, (b) side-view cross-section of the pot, and (c) photo of the pots.

The pots initially had overhead netting (see Figure 1c) to prevent strong sunlight and heat injury and to reduce evaporation and increase seedling survival rate. The nets were removed after 10 weeks once the plants established. The soil sample used is the typical soil available in farms in Qatar. It was obtained from the research experimental farm of the Agricultural Department of Qatar Ministry of Municipality and Environment at Rawdat Al-Faras, Al Khor. A commercially available 20-20-20 NPK fertilizer was used together with Urea in one of the control treatments as shown in Table 1. The fertilizer was applied in three doses at 2, 12 and 24 weeks after planting (Ayotamuno et al., 2009). Commercially available compost corresponding to the type usually used in the farm was employed for the third control treatment. GTL biosludge with 90-95% dry solids obtained from a GTL plant in Qatar was used in the experiments as shown in Table 1. The physical and chemical properties of the materials used are shown subsequently.

Table 1. Details of pot experiments

Pot	Treatment	Detailed composition (% wt.)			
		Soil	Compost	Inorganic fertilizer	Biosludge
C1	Control 1 (Soil)	100	0	0	0
C2	Control 2 (Soil + fertilizer)	100	0	NPK 100 kg/ha and Urea 75 kg/ha. (2.12 g and 1.59 g per pot)	0
C3	Control 3 (Soil + 3% compost)	97	3	0	0
E1	Soil + 0.75% Biosludge	99.25	0	0	0.75
E2	Soil + 1.5% Biosludge	98.5	0	0	1.5
E3	Soil + 3% Biolsudge	97	0	0	3
E4	Soil + 6% Biosludge	94	0	0	6
E5	Soil + 12% biosludge	88	0	0	12

2.2. Seeding and irrigation

The pots were first irrigated to set the soil columns before sowing of alfalfa seeds at 1 cm depth at 10 locations for each pot. Irrigation was applied to each pot manually every three days during the winter and daily during the summer. The amount of water applied was based on the irrigation water requirements of alfalfa for different months, which has an annual average of 2.71 mm/day, the lowest being 1.3 mm/day in January and the highest 5.6 mm/day in July. This was conducted to be in line with the normal irrigation practice of the Qatar Ministry of Municipality and Environment. The properties of the irrigation water used are shown in the supplementary section (Table A1).

2.3 Mixture details and sampling

The pots were filled with samples of soil, and mixtures of soil, and inorganic fertilizer, 3% compost or 0.75 – 12% biosludge according to the details presented in Table 1. Each treatment had three replicate pots arranged in a completely randomized design containing alfalfa seedlings. The inorganic fertilizer (C2) and compost (C3) controls were compared with the biosludge treatments (E1 – E5) to assess soil fertility improvement caused by biosludge amendment in contrast to typical fertilizer and compost application levels on farmlands in Qatar. Similarly, the soil-only control allows for assessment of soil fertility improvement caused by biosludge amendment compared to some natural soils in Qatar with improved fertility.

Soil samples were collected from the pots for initial analysis before seed sowing and at the final-growth stage (12 months) using a tube sampler (auger). At the final-growth stage, soil samples were collected from the top (top 20 cm depth) and bottom (remaining depth) portions of the pots to evaluate the spatial variability of selected parameters. Plant samples were collected after each cut (harvest). All pots were checked simultaneously for leachate formation every 2 - 4 weeks. When formed, the entire leachate volume drainable via the collection valve of the pots was collected in clean glass bottles during each sampling. Hence, the leachate formation and collection period lasted much longer than the plant-growth study period.

2.4 Testing methods

The biosludge, soil, plant and leachate samples were subjected to a series of analyses. It should be noted that mixtures of soil and other planting materials (fertilizer, compost and biosludge) are referred to as soil in this section for simplicity. The following provides a brief description of the characterization/testing methods employed.

Particle size distribution: A complete particle size distribution of representative soil samples was produced by merging together data from standard sieve sizes (> 2 mm) and laser diffraction (< 2 mm) particle size analyzer (Model LS 13 320, *Beckman Coulter*, Fullerton, CA). The instrument measures across a range of 0.04 – 2000 microns in a single analysis (Xu, 2001).

Soil elemental composition: X-ray fluorescence (XRF) was first used for semi-quantitative analysis of the elemental composition of the soil samples to provide knowledge of the major elements present. The absolute concentrations of metallic elements were then determined using inductively coupled plasma-optical emission spectrometry (ICP-OES). In the XRF analysis, a homogenised soil sample was ground to powder form using an automatic ball mill and the sample loaded onto a 40 mm diameter aluminium cup. Thereafter, a powder pellet was prepared using a 20T power press. A ZSX-Primus II X-ray fluorescence (XRF) spectrometer (*Rigaku Corporation*, Tokyo, Japan) was then used for elemental analysis of the powdered samples. Soil samples for ICP-OES analysis were first digested in nitric acid and the metal concentrations determined thereafter using an iCAP 6000 Series ICP-OES (*Thermo Scientific*, USA). The total nitrogen of the soil samples was analysed in line with APHA Method 4500 NO_3-E / 4500 NO_2-B (Rice et al., 2017).

Soil mineralogical composition: The mineralogical composition (crystalline minerals/phases) of the soil samples were monitored using X-ray diffraction (XRD) analysis. The analysis was conducted using a Rigaku Ultima IV multipurpose X-ray diffractometer (Rigaku Corporation, Tokyo, Japan). XRD pattern was collected at 2theta (2θ) angle from 3 to 80 degrees with a step size of 0.01 degree and scanning speed of 0.5°/min. The XRD pattern was then analysed using the integrated Rigaku PDXL2 powder diffraction software.

Porosity and pore size distribution: The porosity and pore size distribution were characterized using a 2 MHz nuclear magnetic resonance (NMR) rock core analyzer with a 54 mm probe (*Magritek*, New Zealand). The T_2 relaxation data was determined on a water-saturated soil sample placed in a 20-ml cylindrical plastic container. The Carr-Purcell-Meiboom-Gill (CPMG) sequence was used with 100 μs echo time, an inter-experimental delay time of 6,500 ms and 200 scans. A Lawson and Hanson non-negative least square fit method was then employed to analyse the CPMG decay using Prospa software (*Magritek*, New Zealand). The software also outputs the T_2 log-mean, which is a proxy for the mean pore size. Details of the NMR technique are provided in Kogbara et al. (2015).

Aboveground biomass: Samples for biomass determination were collected from 10 plants using a stainless steel grass shear to snip plants at about 5 cm above ground level during each cut (Hedlund et al., 2003). The fresh biomass weight was then taken. Three cuts were carried out on the plants, 3, 6 and 7 months after planting in line with the normal agronomic practice in Qatar.

Plant height and number of tillers/branches: The plant height was determined by measuring the distance from the soil level to the terminal bud of the longest stem on that plant (Barney et al., 1974). The number of main tillers was determined by counting them from three randomly selected plants.

Plant elemental content: The elemental content of the plants was determined to evaluate potential accumulation of elements from the biosludge in plant tissues. Biomass from plant cuts were dried and ground, and subjected to wet digestion with nitric acid. Thereafter, elemental content analysis was done using the aforementioned ICP-OES instrument.

Leachate analysis: Leachates collected from the pots were filtered using 0.45-micron syringe cartridge filters to eliminate solid particles. The pH and conductivity of leachate samples were measured using a Mettler Toledo SevenMulti dual (conductivity/pH) meter. The leachate samples were subjected to ion chromatography (IC) following ASTM D 4327 (ASTM, 2003) using an 850 Professional IC (*Metrohm*, Switzerland) for analysis of key anions (e.g. NO_3^-, PO_4^{3-} and SO_4^{2-}). Analysis of metals in leachate samples was carried out using an ICP-OES instrument after dilution with a 2% nitric acid solution following ASTM UOP714 (ASTM, 2007). The total nitrogen (TN) content of the leachate samples was analysed using a TOC-L series total organic carbon analyzer (*Shimadzu*, Japan) in line with APHA Method 5310 (Rice et al., 2017).

2.5 Statistics

The mean values of different parameters over time in the different treatments were compared using Analysis of variance (ANOVA). Significant means at 5% probability level were separated using the Duncan’s multiple range test. Multiple linear regression was carried out to determine properties that significantly influence plant growth parameters using the best model method (Harrell, 2001). The minimum and maximum variables were chosen as 2 and 5, respectively, and the adjusted coefficient of determination (R^2) chosen as criteria to determine the best model. The average concentrations (between the initial and final growth stages) of specific elements abundant in the soil that are likely to affect plant performance were used as explanatory variables. The average soil total porosity and mean pore size, leachate composition and properties, biomass elemental composition and sand, silt and clay contents were also used as explanatory variables since a myriad of factors influence plant growth. Fresh biomass weight, plant height and the number of tillers were the dependent variables. The analyses were carried out using XLSTAT v2017.3 software (*Addinsoft*, New York, USA).

3. Results and discussion

This section compares the performance of the different treatments between the initial and final growth stages. Where applicable, there are letters assigned above different columns of the graph(s) to indicate significant differences between mean values based on the Duncan’s multiple range test. Treatments not sharing a letter are significantly different from each other.

3.1. Particle size distribution

The particle size distribution of the different treatments before planting as well as the biosludge and compost used is shown in Figure 2. The soil in all treatments, as well as the biosludge and compost, had less than 10% of particles within the finer (silt and clay) division as the majority of the particles fell within the sand fraction. The soil has about 91.5% sand, 7% silt and 1.5% clay, thus it has a fine sand texture according to the United States Department of Agriculture (USDA) classification system. The particle size distribution of the treatments (E1 – E3) and controls (C1 – C3) were similar as there was no significant difference (p = 0.91) due to differences in biosludge or compost content.

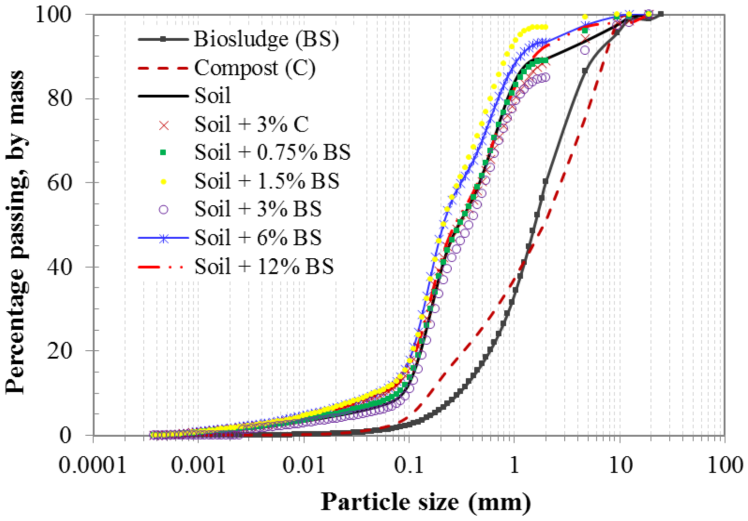
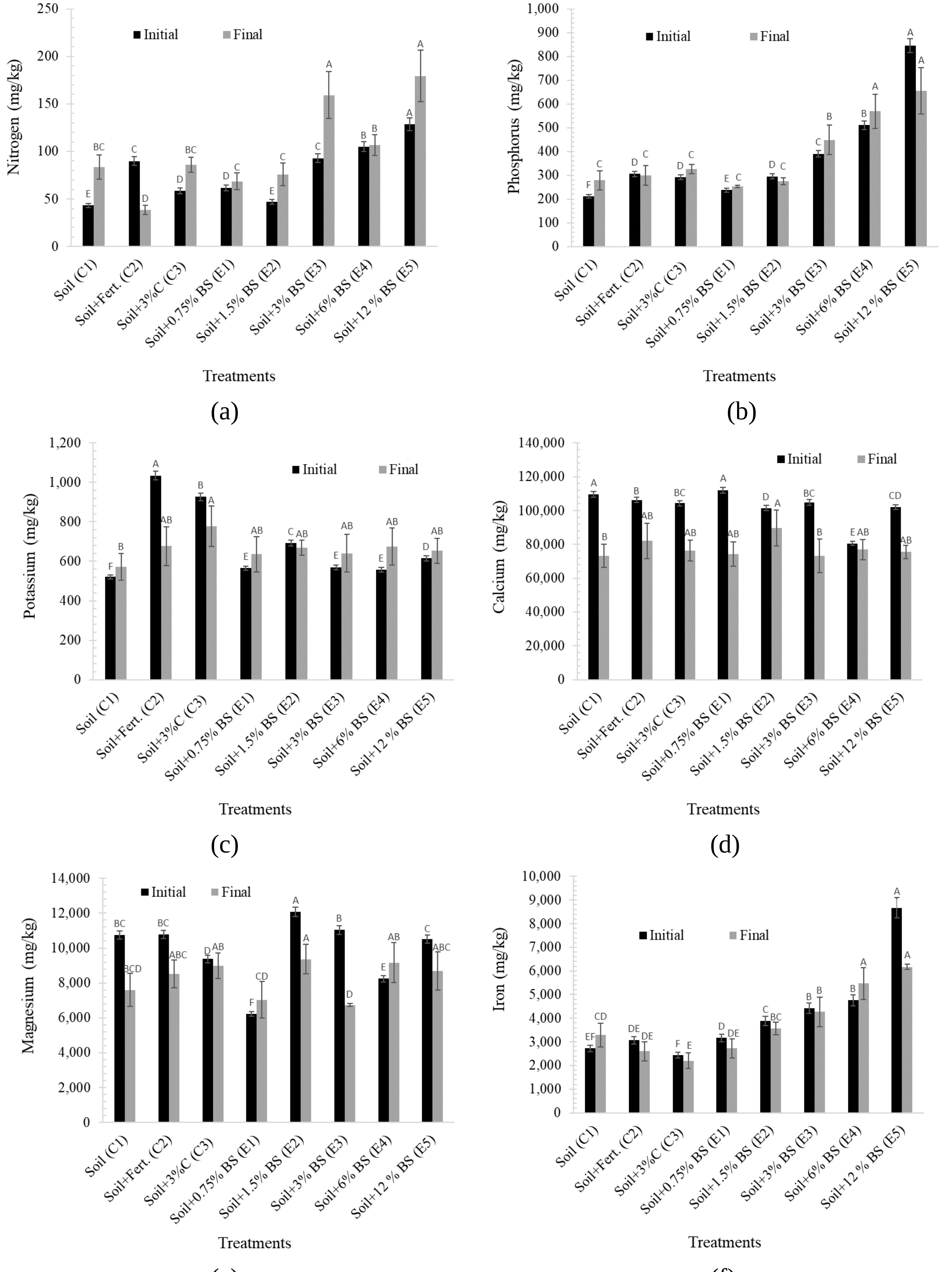


Figure 2. Particle size distribution of the different treatments before planting.

3.2. Soil elemental composition

Figure 3 shows the absolute concentrations of 6 selected elements in soil samples from the different treatments determined through ICP-OES at the initial- and final-growth stages. The average of the spatial variation of the selected elements in the top and bottom layers of the pots in the different treatments at the final-growth stage is shown in Figure 4. The selected elements were chosen based on their relative abundance in the biosludge or soil. The 6 elements in question are primary macronutrients, N, P, K, secondary macronutrients, Ca and Mg, and the micronutrient, Fe, which shows a relatively high concentration in the biosludge (see supplementary section, Table A2). The absolute concentrations of all elements analyzed in the biosludge, soil, soil-biosludge and soil-compost mixtures at the initial- and final-growth stages are shown in the supplementary section (Tables A2 and A3). The semi-quantitative XRF analysis of the elemental composition of the biosludge and compost used, and the soils in the different treatments at the initial- and final-growth stages is also shown in the supplementary section (Table A4). It can be seen from Table A2 in the supplementary section that the concentrations of all applicable elements are within the regulatory limits prescribed by the Gulf Cooperation Countries (GCC) standard for sewage fertilizer and the United States Environmental Protection Agency (US EPA) 40 CFR Part 503 guideline on biosludge (US EPA, 1993). This makes the biosludge safe for land application based on the elements identified in the standards.

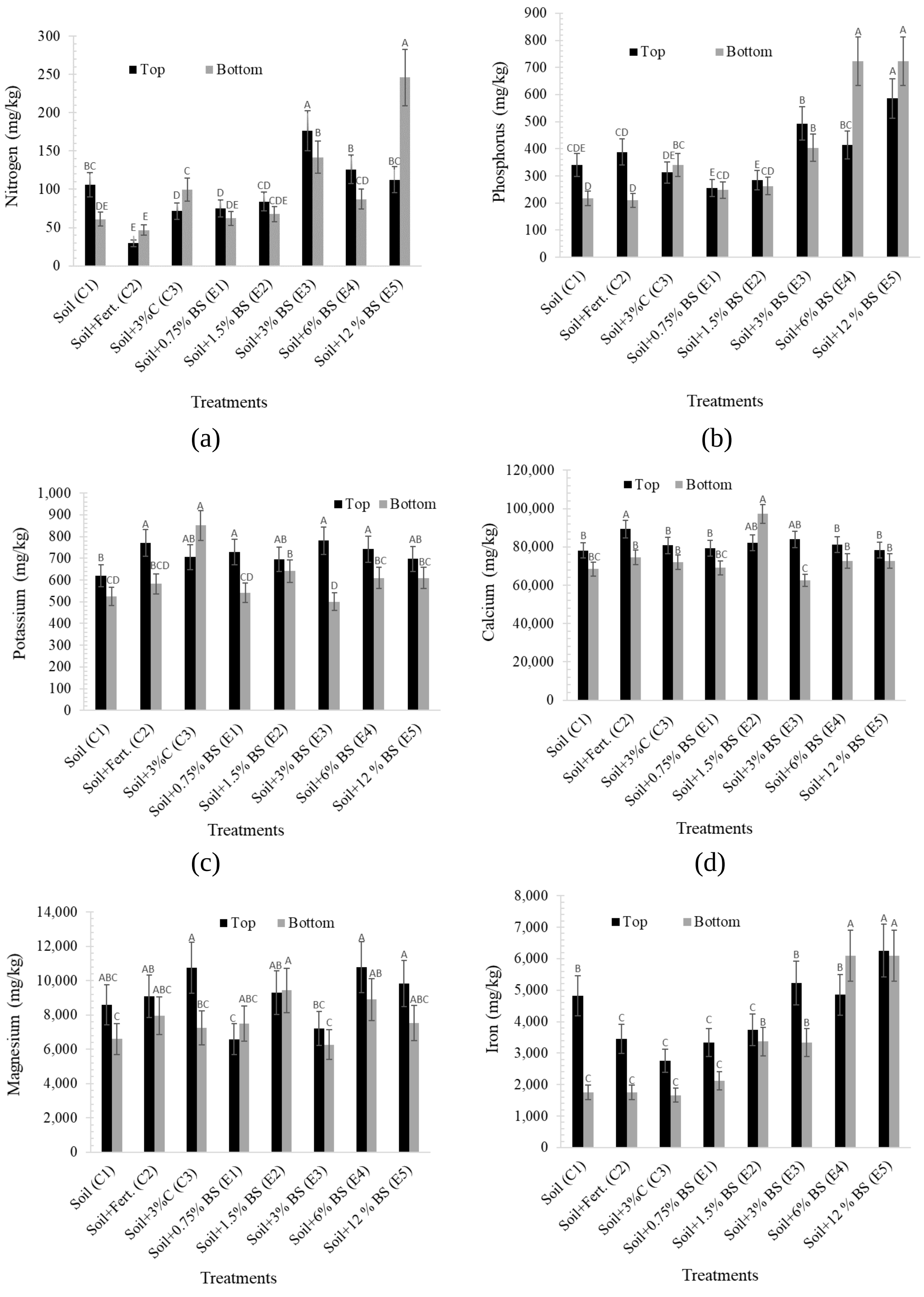


(a) (b)

(c) (d)

(e) (f)

Figure 3. Absolute concentrations of selected elements at the initial (before planting) and final-growth stages. *Note: BS – Biosludge, C – Compost, Fert. – Fertilizer (NPK + Urea). Treatments not sharing a letter (from the Duncan's multiple range test) above the columns are significantly different from each other.*



(a) (b)

(c) (d)

(e) (f)

Figure 4. Absolute concentrations of selected elements in the top and bottom layers at the final-growth stage. *Note: BS – Biosludge, C – Compost, Fert. – Fertilizer (NPK + Urea). Treatments not sharing a letter (from the Duncan's multiple range test) above the columns are significantly different from each other.*

There was a general increase in the total N between the initial and final stages in all treatments except the soil + fertilizer (C2) treatment even though the initial samples were collected before fertilizer application in the treatment (Figure 3a). This confirms previous observations on the quick loss of nutrients from inorganic fertilizers from the soil compared to the slow-release of nutrients by organic fertilizers (compost and biosludge) (Sullivan et al., 2015). Phosphorus also showed a similar trend as its concentration increased between the initial and final stages in the majority of the treatments. There was a significant loss of K from the soil + fertilizer treatment and a milder loss in the soil + compost treatment between the initial and final stages. This contrasts with a general slight increase in K concentration in the biosludge treatments in line with the aforementioned nutrient loss observation (Figure 3c). All the same, the observed increases in N, P and K concentration may be due to the high intrinsic variability of soil properties over space (horizontally and vertically) and time. Calcium concentrations decreased between the initial and final stages in all treatments. The concentrations of Mg and Fe decreased in six out of eight treatments between the initial and final growth stages (Figures 3e and 3f).

The decreases of the cations, Ca and Mg, in the treatments over time is probably because considerable amounts of the elements are taken up by alfalfa (Schrenk and Silker, 1950) and due to their relatively higher rate of leaching from the soil. A relatively higher leaching rate may also account for the aforementioned loss of K from the soil + compost treatment. The exact mechanism for losses in Fe concentration over time is however unclear as only small amounts showed up in the leachate and the plant aboveground biomass. These are discussed subsequently in the sections on leachate concentrations and plant performance parameters. Nevertheless, below ground biomass (roots) was not analyzed in this work and previous studies have shown that Fe compounds could be deposited on plant roots in soils with elevated Fe concentrations

(Batty and Younger, 2003; Peña-Olmos et al., 2014). The Duncan multiple range test indicated that all six selected elements showed the same level of significance at the initial stage ($p < 0.0001$). However, at the final stage, N, P and Fe indicated the most significant differences ($p < 0.0001$) between treatments. There were also significant differences in Mg concentrations among treatments ($p = 0.013$) but K ($p = 0.26$) and Ca ($p = 0.25$) concentrations were however not significant (see range of letters on columns in Figure 3a – 3f). Hence, N, P, Fe and Mg are likely to affect differences in plant productivity. Among all six selected elements, only Ca ($p < 0.0001$) and Mg ($p = 0.001$) showed significant differences in concentrations between the initial and final growth stages probably due to the aforementioned reason.

Furthermore, there were significant differences between the concentrations of the metals, K, Ca, Mg and Fe in the top and bottom layers of the pots in the different treatments as the metals resided more in the top than bottom layers in at least 6 out of 8 treatments. However, differences in N and P concentrations between the top and bottom layers were not significant ($p > 0.80$), and a few treatments showed higher concentrations in the bottom than top layer. Phosphorus tended to reside more in the bottom than top layer in the 6 and 12% biosludge treatments. Interestingly, N, P and K resided more in the bottom than top layer in the soil + 3% compost treatment.

3.3 Soil mineralogical composition

The mineralogical composition of the biosludge, compost and the different treatments is shown in Table 2. The biosludge is mainly amorphous and contains calcite, quartz and dolomite. The major minerals in the soil and soil-biosludge mixtures are calcite, quartz, dolomite, muscovite (mica), albite (Na-feldspar), and clay minerals such as kaolinite and palygorskite. The systematic change of mineral weight percentage with increasing biosludge content at the initial stage is not apparent because all treatments contained various amounts of amorphous materials. Hence, the analysis at the final growth stage focused on selected treatments, namely, soil, and soil with 3, 6 and 12% biosludge contents (Table 2).

Generally, the mineralogical compositions do not show significant variations between the initial and final growth stages and between treatments at both growth stages, in the treatments analyzed, at the 5% probability level. A similar observation was made in a previous study (Bakker et al., 2018). The only exception was albite (i.e. Na-feldspar), which showed a significant decrease ($p = 0.049$) between the initial and final growth stages. This is likely due to the well-documented chemical weathering of albite to kaolinite enabled by the production of organic acids from biosludge decomposition (Sokolova, 2013). This is supported by an increase in kaolinite content in the mixtures with higher biosludge content (Table 2).

Table 2. Mineralogical composition of the biosludge, compost, soil, soil-biosludge and soil-compost treatments before planting

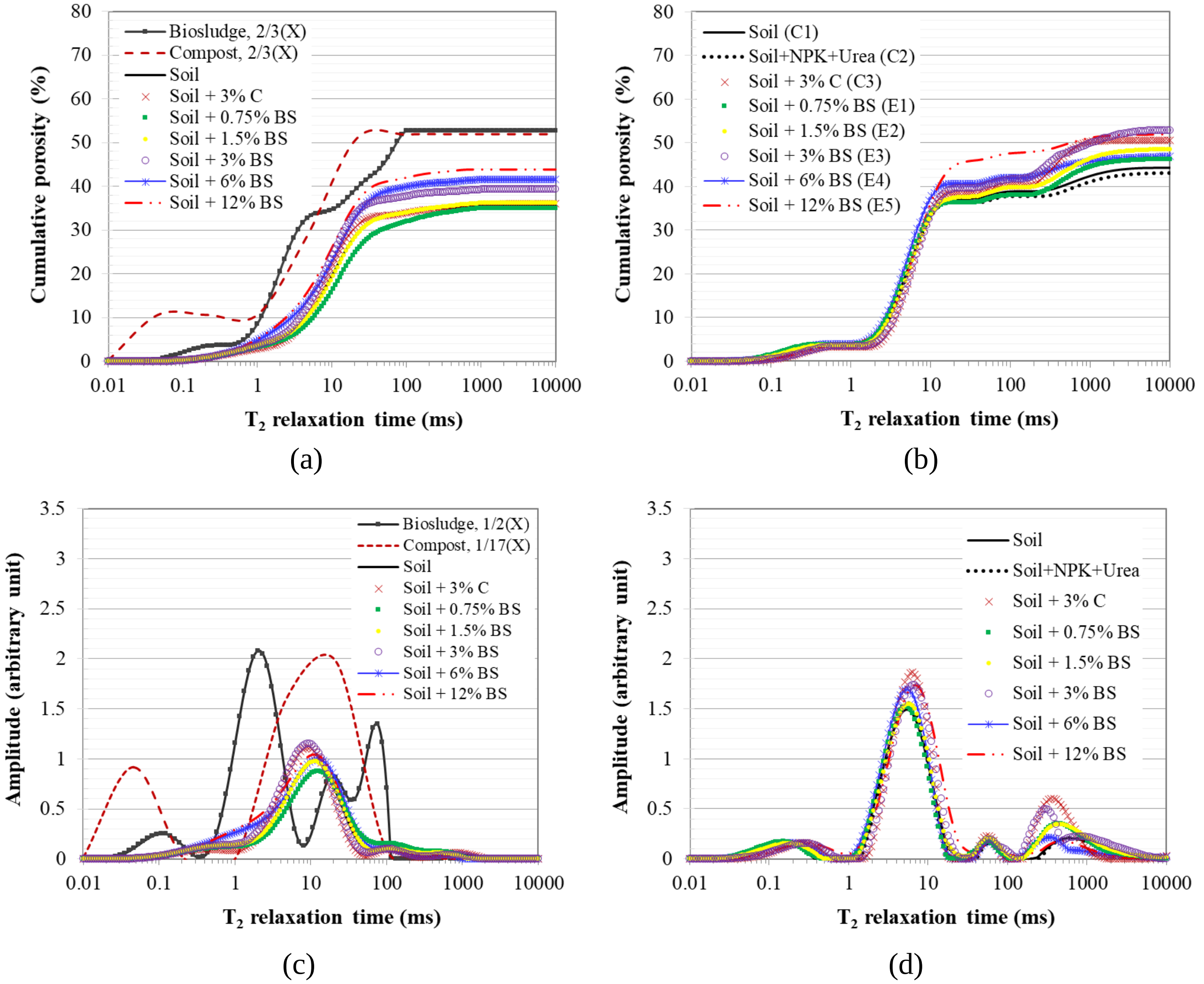
Mineral	Growth Stage	Biosludge (BS)	Compost (C)	Soil only (C1)	Soil + 3% C (C3)	Soil + 0.75% BS (E1)	Soil + 1.5% BS (E2)	Soil + 3% BS (E3)	Soil + 6% BS (E4)	Soil + 12 % BS (E5)
		Percent by weight								
Calcite ($CaCO_3$)	Initial	69	16	20	18	24.8	40	24	47	21
	Final			18	-	-	-	27	31	21
Quartz (SiO_2)	Initial	6	77	57	37	54.4	40	50	30	57
	Final			60	-	-	-	53	53	54
Dolomite [$CaMg(CO_3)_2$]	Initial	25	-	8.6	5.9	5.1	4.5	9.0	6.0	8.2
	Final			7.8	-	-	-	9.2	6.7	6.1
Muscovite [$KAl_2(AlSi_3O_{10})(F,OH)_2$]	Initial	-	-	1.3	1.4	2	1.45	1.5	3.4	3.1
	Final			3.3	-	-	-	3.7	4.1	3.4
Albite ($NaAlSi_3O_8$)	Initial	-	-	10.8	32	12.2	13	14	10	9.3
	Final			7.8	-	-	-	3.3	1.5	6.1
Kaolinite [$Al_2Si_2O_5(OH)_4$]	Initial	-	-	0.8	4.8	0.64	0.39	0.5	0.4	0.6
	Final			0.5	-	-	-	1.4	1.9	2.3
Palygorskite [$(Mg.Al)_2Si_4O_{10}(OH).4(H_2O)$]	Initial	-	-	1.5	0.9	0.84	0.65	0.64	3	0.85
	Final			2.3	-	-	-	1.5	2.0	6.7
Sylvite (KCl)	Initial	-	7	-	-	-	-	-	-	-
	Final			-	-	-	-	-	-	-

Note: Samples in all treatments included various amount of amorphous materials, which cannot be identified by XRD. Thus, the weight percentage here only shows mineral contents (i.e., the crystalline portion) of the sample. The Soil + Fertilizer treatment (C2) is not included as it is similar to C1 at the initial stage (before fertilizer application) and was not among the selected treatments (C1, E3, E4 and E5) analyzed at the final growth stage.

3.4. Porosity and pore size distribution

The NMR porosity and pore size distribution (using a proxy - the T_2 distribution) analyses of the treatments at the initial and final growth stages is shown in Figure 5. It is common practice to use T_2 as a proxy for pore size instead of converting it to actual pore size since relaxation times are impacted by paramagnetic species such as Fe (Kogbara et al., 2015). The spatial variability of the aforementioned pore structure parameters at the final growth stage is shown in Figure 6. In the T_2 distribution graphs, the peaks represent pores of different sizes, while the amplitudes of the peaks denote the volumetric abundance of each pore type. The values for T_2 relaxation times are related to pore sizes (diameter) – shorter times for smaller pores and longer times for larger pores. The boundary conditions for different pore systems vary between publications. The threshold T_2 relaxation time separating micropores and mesopores was found between 10 and 30 ms for soil samples with various textures and organic matter content (Jaeger et al., 2009). A T_2 relaxation time > 300 ms is reported to represent macroporosity (Bayer et al., 2010). Thus, Figures 5 and 6 show that the treatments had far more micropores than meso- and macro-pores.

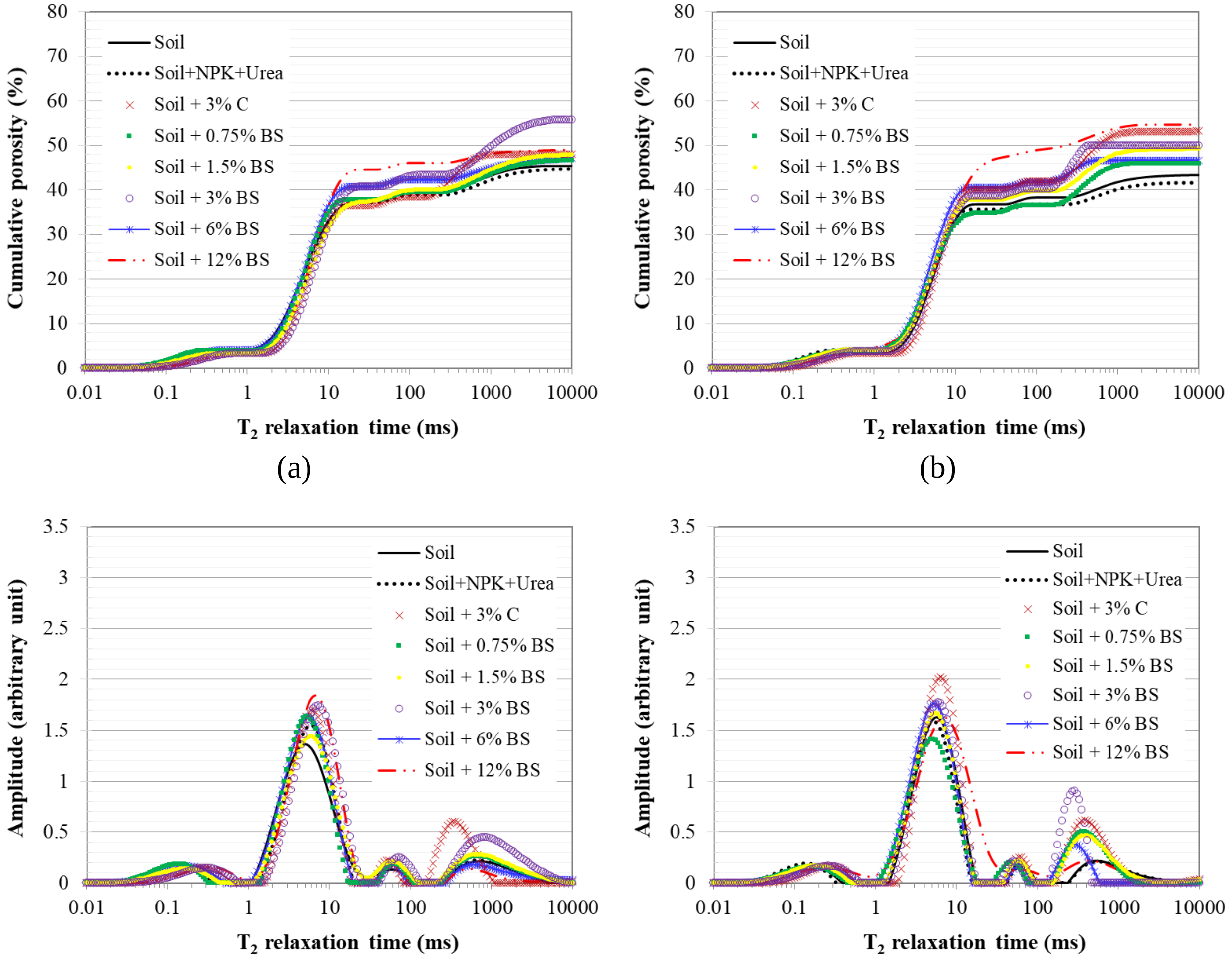
The addition of biosludge and compost to the soil significantly increased the total (cumulative) porosity of the soil ($p < 0.01$) between the initial and final growth stages (Figure 5). Over time, growing roots naturally increase porosity at the root-soil interface by reducing root-soil contact as roots grow and decay (Bodner et al., 2014). However, there was no significant difference ($p = 0.08$) in the total porosity due to differences in biosludge content at the 5% probability level. The average total porosity in the top and bottom layers were similar. However, there were apparently higher micro- and macro-porosity in the bottom than the top layer at the final-growth stage (Figure 6). It is likely that the pressure from the repeated manual micro-head irrigation method used may compact the top layer and reduce porosity.



(a) (b)

(c) (d)

Figure 5. NMR cumulative porosity and T_2 distribution of the different treatments, respectively, at the (a) and (c) initial- (before planting), and (b) and (d) final-growth stages. *Note: BS – Biosludge, C – Compost. Treatment C2 is not shown in (a) as it is similar to C1 at the initial stage. The T_2 distribution (proxy for pore size distribution) data for biosludge and compost were reduced two and seventeen times, respectively, while the cumulative porosity data were reduced one and half times to enable plotting on the same scale with the different treatments. The T_2 log-mean values are shown in the supplementary section (Table A5).*



(a) (b)

(c) (d)

Figure 6. Spatial variation of the NMR cumulative porosity and T_2 distribution, respectively, in the (a) and (c) top, and (b) and (d) bottom layers, at the final-growth stage.

Note: BS – Biosludge, C – Compost.

The T_2 distributions of the treatments before planting show that ≥ 1.5% biosludge content, as well as compost content, caused a noticeable increase in the microporosity of the soil (Figure 5d). There was also a considerable increase in the volumetric abundance of the different pore types between the initial and final growth stages. These can possibly improve the water holding capacity of the soil as microporosity retains water required for plant growth. In particular, the soil-compost treatment and soil with 0.75 - 3% biosludge contents showed significantly ($p = 0.0002$) higher volumes of macropores compared to other treatments at the final growth stage.

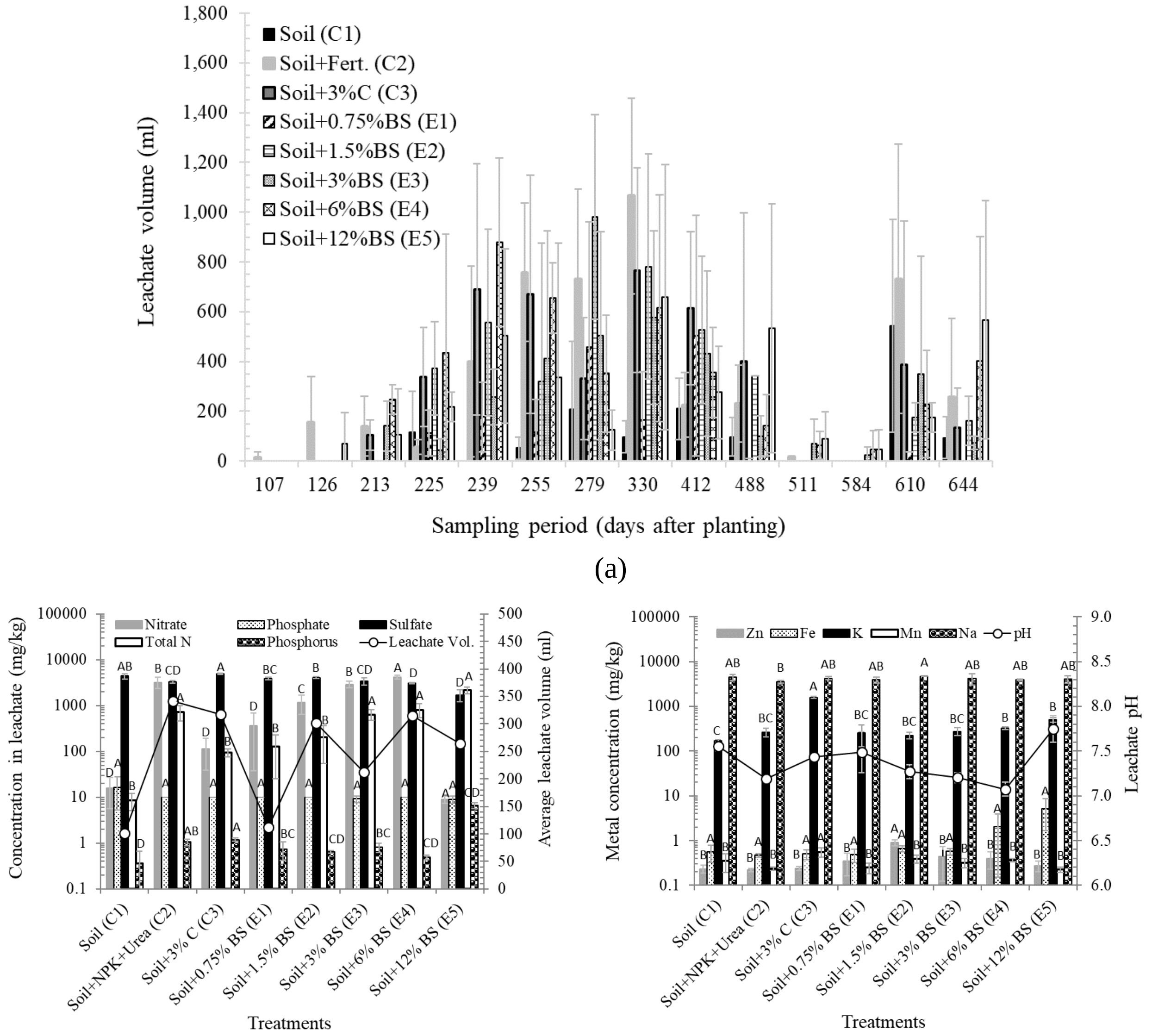
This increased macroporosity and the resulting better aeration might enhance alfalfa growth. Macroporosity controls rapid drainage of excess water after irrigation and circulation of oxygen to roots. It also has some useful effect on root penetration (Pagliai and Vignozzi, 2006).

3.6 Leachate concentrations

Figure 7 shows the evolution of leachate formation in the pots, the leachate pH and concentrations of key anions and cations. The average leachate volume in each treatment is shown on the secondary axis in Figure 7b. Figure 7b and 7c contain only selected elements (Zn, Fe, K, Mn, Na, N and P) and anions (NO_3^-, PO_4^{3-}, SO_4^{2-}). The selection is based on primary nutrients and key elements with much higher concentration in biosludge than soil, which can possibly pollute ground water (see Table A2 in the supplementary section). The leachate concentrations of all other elements not included in Figure 7 is shown in the supplementary section (Table A6).

Leachate formation was initiated earlier in the fertilizer and compost controls and ≥ 3% biosludge treatments compared to the soil and lower biosludge treatments (Figure 7a). This may result from differences in pore structure characteristics of the different treatments, including parameters such as pore network connectivity and tortuosity, which affect the flow of water through porous media (Kogbara et al., 2014; Smet et al., 2018), but is beyond the scope of this work. However, on the average, mixtures with ≥ 1.5% biosludge and compost contents resulted in more leachate than the soil and 0.75% biosludge mixtures (Figure 7b). This correlates with higher total porosities recorded in the former than the latter (see Figure 5b). The addition of biosludge was expected to improve water retention rather than release. However, it is well documented that legumes such as alfalfa increase soil permeability (Soong and Yap, 1976).

Thus, the interaction of decomposable organic matter from the plant and the added biosludge could accelerate percolation and lessen water retention. The leachate pH of the different treatments was similar, falling within a narrow range of 7.0 - 7.8 (Figure 7c).



(a)

(b)

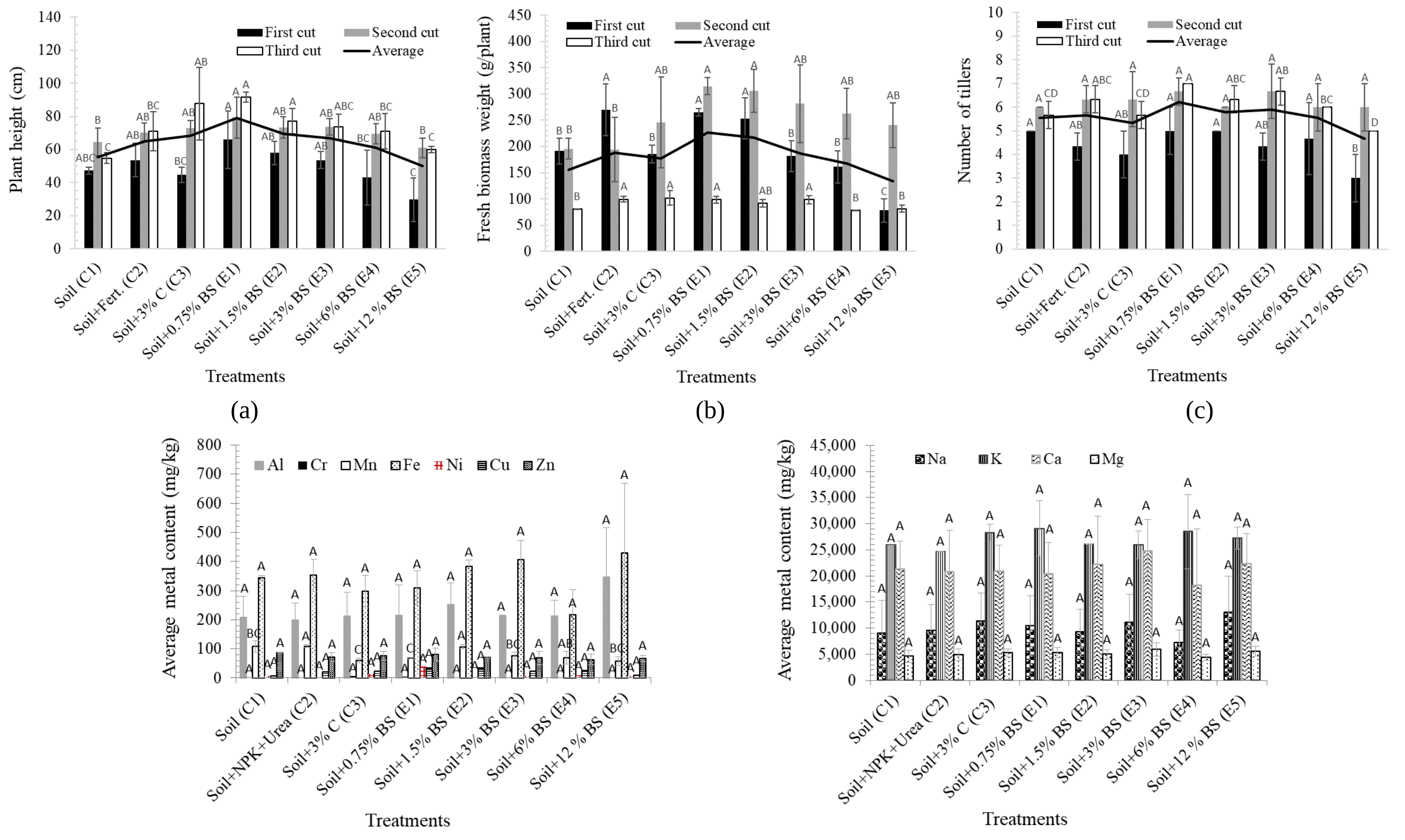
(c)

Figure 7. Leachate parameters in terms of, (a) leachate volume, (b) average leachate volume and concentrations of anions and cations, and (c) leachate pH and concentrations of metals. *Note: BS – Biosludge, C – Compost. Treatments not sharing a letter (from the Duncan's multiple range test) above the columns in Fig. 7 (b & c) are significantly different from each other.*

The leachate concentrations of SO_4^{2-} and PO_4^{3-} were generally lower in the biosludge mixtures than in the soil and soil-fertilizer controls. The NO_3^- and total N concentrations were however up to two orders of magnitude higher in most of the biosludge mixtures than the controls. Albeit, the NO_3^- concentration in the soil-fertilizer treatment far exceeded most of the other treatments. The leachate concentration of P in ≤ 6% biosludge mixtures was similar to the soil only treatment and less than the soil-fertilizer and soil-compost mixtures (Figure 7b). The leachate concentrations of Zn, Fe, Mn and Na in the biosludge treatments were generally similar to the controls. Although for Fe, this applies to mixtures with ≤ 3% biosludge content as the 6 and 12% biosludge treatments showed roughly four to eight times higher concentration than the soil-only control (C1). Nevertheless, as noted in Section 3.2, the ≤ 5 mg/kg leachate concentrations were quite low (Figure 7c). In contrast, much higher K concentrations were leached from the biosludge treatments compared to the soil-only and soil fertilizer controls. However, these were far less than the leachate concentration in the soil-compost mixture, which may be responsible for the significant loss of K from the soil compared to other treatments as noted in Section 3.2. Overall, the anions/cations that showed high leaching potential in the biosludge mixtures exceeding 1,000 mg/kg in some treatments were total N / NO_3^-, SO_4^{2-} and Na (Figure 7). Other species such as Cl^-, Ca and Mg also demonstrated high leaching potential (Table A6 in the supplementary section). These mostly came from the irrigation water and background concentrations in the soil and biosludge. However, the leachate concentrations in question were generally similar to the soil only and soil-fertilizer controls, which rules out the possibility of the biosludge polluting ground water.

3.7 Plant performance parameters

Figure 8 shows the plant height, aboveground fresh weight biomass, number of tillers and metal contents (average of the three cuts) of the plant biomass. The plant height, aboveground biomass and the number of tillers generally decreased with increasing biosludge content. There were comparable or better plant height, aboveground biomass and number of tillers with biosludge content ranging from 0.75 – 3% compared to the fertilizer and compost controls (Figure 8a – 8c). Two-way ANOVA showed significant differences in the plant height, fresh weight biomass and number of tillers due to the different treatments ($p < 0.0001$, but for number of tillers, $p = 0.0045$) at different harvest periods ($p < 0.0001$). There was no significant difference in the fresh weight biomass ($p = 0.06$) and the number of tillers ($p = 0.33$) among treatments with 0.75 – 3% biosludge at the 5% probability level. Differences in plant height were however significant ($p = 0.03$). The optimum alfalfa growth parameters here compare favorably with published values for soils in neighboring Saudi Arabia in the absence of similar published data for Qatari soils (Daur et al., 2018). There were generally no significant differences in the concentrations of metals and macro elements in the plant biomass among the different treatments, except for Mn ($p = 0.006$) (Figure 8d and 8e).



(a) (b) (c)

(d) (e)

Figure 8. Plant growth performance of the different treatments over time in terms of the (a) plant height, (b) aboveground fresh biomass weight, (c) number of tillers, (d) average content of metals, and (e) average content of macro elements. *Note: BS – Biosludge, C – Compost, Fert. – Fertilizer (NPK + Urea). The metal concentrations in (d) and (e) are the means and standard deviations of the first, second and third cuts of the plants. Treatments not sharing a letter (from the Duncan's multiple range test) above the columns are significantly different from each other.*

The variables that significantly affected plant growth performance based on the regression modeling are summarized in Table A7 in the supplementary section. The equations for the models are shown beneath Table A7. The regression modeling showed that the plant height was mostly affected by the total porosity, leachate concentrations of Cl^- and P, the biomass Ca content and the soil clay content, with the leachate P concentration being the most influential variable. Phosphorus has previously been identified as the most required nutrient for alfalfa production, thus, the leachate concentration is likely to affect plant growth. The roles of Cl^- in plants include photosynthesis, osmotic adjustment and suppression of plant disease. Calcium is a component of plant cell walls and regulates cell wall construction (McCauley et al., 2011). The influence of soil porosity was discussed previously in Section 3.5.

The fresh weight biomass is mostly affected by the soil Fe content, the leachate concentrations of NO_3^-, PO_4^{3-} and Mg, and the soil's silt content, with soil Fe content being the most influential (see Table A7). As mentioned in Section 3.2, due to the relatively high Fe concentration in the soil, Fe compounds could be deposited on plant roots, and some taken up into plant tissues (see Figure 8d). This can in turn significantly reduce growth by causing iron toxicity within the plants and/or impede the uptake of nutrient(s) by the plants, resulting in nutrient deficiency. Nutrients that may be involved include PO_4^{3-} and metals (Mg, K, Ca and Zn) known to be absorbed by $Fe(OH)_3$ deposits on roots (Batty and Younger, 2003). Magnesium is important as it is necessary for the synthesis of chlorophyll and photosynthesis (McCauley et al., 2011). The importance of N/ NO_3^- and P/PO_4^{3-} have been discussed previously.

The number of tillers is mostly affected by the concentrations of Al, N and Zn in the soil, biomass Mn content, and soil clay content, which is identified as the most influential variable. This makes sense as the capacity to hold plant nutrients is in the order, clay > silt > sand and three of the other four influential variables are contained in the soil. Manganese is the only metal that differed significantly among the treatments in the plant biomass. Its biomass concentration was around half that of soil (see Figure 8d and Table A2). The amounts in the biomass are around the average reported being essential for alfalfa growth (Schrenk and Silker, 1950). A similar situation applies to the soil and biomass Zn contents.

It should be noted that pot experiments have several limitations, including the use of limited soil volume and the inability to use the natural soil profile. These make soil structural features (e.g., pre-existing biopores), temperature, aeration, and soil-water relations somewhat different from what obtains in the field. These parameters strongly influence root structure, physiology, and interactions in the root zone (Passioura, 2006). Other limitations include the use of optimum watering, leading to better solubility of nutrients, which enhances the effects of nutrients compared to field conditions. Hence, the results here serve as a background for field experiments and may not be directly transferred for practice. Future research will include experiments in 1 m deep lysimeters with much soil aerated well enough, making hypoxia unlikely. Field experiments, which use the natural soil profile and considers the limitations mentioned above, will also be conducted.

4. Conclusions

This work investigated the effect of GTL biosludge on soil properties and alfalfa yields in an arid soil. Alfalfa was grown for a year period in semi-controlled pots containing soil, soil-fertilizer/compost and soil plus five biosludge contents, namely, 0.75, 1.5, 3, 6 and 12%. The results demonstrate that the biosludge improved the total porosity and the volumetric abundance of different pore types in the soil, which in turn affected plant performance, especially the plant height. Alfalfa yield in terms of plant height, fresh biomass weight and the number of tillers decreased with increasing biosludge content. Treatments with 0.75, 1.5 and 3% biosludge content showed comparable or better plant yield compared to the soil-only, soil-fertilizer and soil-compost controls. The 6% biosludge content produced slightly lower yields compared to the soil-fertilizer and soil-compost controls but better than the soil-only control. The 12% biosludge content showed the worst yields.

Regression modeling indicated key variables that influenced plant performance parameters. Leachate concentrations of P, soil Fe concentration and soil clay content were identified as the most influential variables for plant height, fresh biomass weight and the number of tillers, respectively. Some other key variables include soil total porosity and silt content, leachate concentrations of Cl^-, NO_3^-, PO_4^{3-} and Mg, soil concentrations of Al, N and Zn, and biomass concentrations of Ca and Mn. The concentration of chemical species in the leachate and plant biomass of biosludge treatments were either lower or similar to the fertilizer and compost controls. Hence, the results suggest that GTL biosludge could potentially enhance arid soil properties and improve alfalfa yields. Work is in progress to conduct lysimeter and field experiments to better ascertain the effects of GTL biosludge on soil properties and alfalfa yields.

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Appendix A. Supplementary data

Supplementary data to this article is provided in a separate document.

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