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Recycling of gas-to-liquid sludge as a potential organic amendment: Effect on soil and cotton properties under hyperarid conditions

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Abstract:	Gas-to-liquid (GTL) sludge is a specific wastewater treatment by-product, which is generated during the industrial process of natural gas conversion to transportation fuels. This least studied sludge is pathogen-free and rich in organic carbon and plant nutrients. Therefore, it can be reused for soil enhancement as a sustainable management strategy to mitigate landfill gas emissions. In this field study, we compared the performance of soil treatments with GTL sludge to the more conventional chemical fertilizers and cow manure compost for the cultivation of cotton under hyperarid conditions. After a complete growing season, GTL sludge application resulted in the enhancement of soil properties and plant growth compared to conventional inputs. As such, there was a significant dose-dependent increase of soil organic matter (4.01% and 4.54%), phosphorus (534 and 1090 mg kg-1), and cumulative lint yield (4.68 and 5.67 t ha-1) for GTL sludge application rates of 1.5% and 3%, respectively. The produced fiber quality was adequate for an upland cotton variety (Gossypium hirsutum var. MAY 344) and appeared more dependent on the prevailing climate conditions than soil treatments. On the other hand, the adverse effects generally related to industrial sludge reuse were not significant and did not affect the designed agro-environmental system. Accordingly, plants grown on GTL sludge-amended soils showed lower antioxidant activity despite significant salinity increase. In addition, the concentrations of detected heavy metals in soil were within the standards' limits, which did not pose environmental issues under the described experimental conditions. Leachate analysis revealed no risks for groundwater contamination with phytotoxic metals, which were mostly retained by the soil matrix. Therefore, recycling GTL sludge as an organic amendment can be a sustainable solution to improve soil quality and lower carbon footprint. To reduce any environmental concerns, an application rate of 1.5% could be provisionally recommended since a two-fold increase in sludge dose did not result in a significant yield improvement.
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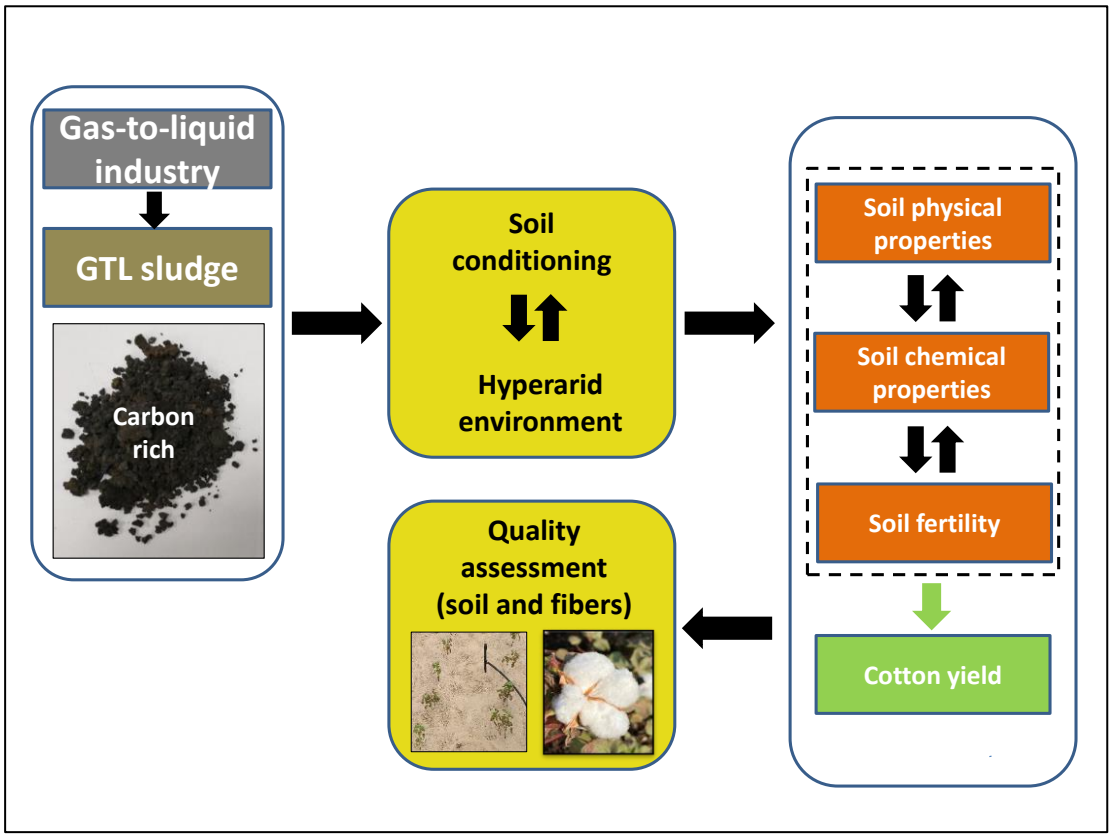

Highlights

- Gas-to-liquid sludge is a carbon-rich by-product of GTL wastewater treatment
- GTL sludge land application mitigates the risks of landfill gas emissions
- GTL sludge improves soil and plant properties under hyperarid conditions

Recycling of gas-to-liquid sludge as a potential organic amendment: Effect on soil and cotton properties under hyperarid conditions

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Abstract

Gas-to-liquid (GTL) sludge is a specific wastewater treatment by-product, which is generated during the industrial process of natural gas conversion to transportation fuels. This least studied sludge is pathogen-free and rich in organic carbon and plant nutrients. Therefore, it can be reused for soil enhancement as a sustainable management strategy to mitigate landfill gas emissions. In this field study, we compared the performance of soil treatments with GTL sludge to the more conventional chemical fertilizers and cow manure compost for the cultivation of cotton under hyperarid conditions. After a complete growing season, GTL sludge application resulted in the enhancement of soil properties and plant growth compared to conventional inputs. As such, there was a significant dose-dependent increase of soil organic matter (4.01% and 4.54%), phosphorus (534 and 1090 mg kg^{-1}), and cumulative lint yield (4.68 and 5.67 t ha^{-1}) for GTL sludge application rates of 1.5% and 3%, respectively. The produced fiber quality was adequate for an upland cotton variety (*Gossypium hirsutum* var. MAY 344) and appeared more dependent on the prevailing climate conditions than soil treatments. On the other hand, the adverse effects generally related to industrial sludge reuse were not significant and did not affect the designed agro-environmental system. Accordingly, plants grown on GTL sludge-amended soils showed lower antioxidant activity despite significant salinity increase. In addition, the concentrations of detected heavy metals in soil were within the standards' limits, which did not pose environmental issues under the described experimental conditions. Leachate analysis revealed no risks for groundwater contamination with phytotoxic metals, which were mostly retained by the soil matrix. Therefore, recycling GTL sludge as an organic amendment can be a sustainable solution to improve soil quality and lower carbon footprint. To reduce any environmental concerns, an application rate of 1.5% could be provisionally recommended since a two-fold increase in sludge dose did not result in a significant yield improvement.

Keywords: Gas-to-liquid sludge; hyperarid conditions; soil enhancement; cotton growth; heavy metals

1. Introduction

As the global population grows, the demand and consumption of agricultural goods such as food, feed and fiber crops increase, and the use of agricultural inputs for the production of these goods follows a similar trend (Gianico et al., 2021). Chemical fertilizers are the most consumed inputs in intensive agricultural systems, with significant amounts of energy required for their synthesis as well (Neves et al., 2020). In addition, the repetitive or excessive applications of chemical fertilizers can affect the human health and environment (Nayana and Ritu, 2017). Alternatively, organic fertilization traces back to ancient Greek civilization and have historically involved a variety of more sustainable/renewable materials that range from fresh or dried plant residues to animal manures and litters to agro-food by-products (Das and Jana, 2003; Antonkiewicz and Łabętowicz, 2016; Kumar et al., 2017).

Since organic matter is a major component of soil structural stability and fertility (Zoghlami et al., 2020; Gerke, 2022), cropland restoration efforts have induced shortages in traditional inputs such as plant litter and animal manure. This has made the need of searching for organic materials from non-conventional sources very high (Hamdi et al., 2019). For instance, sludge is the semi-solid organic by-product of the wastewater treatment process (Mohajerani et al., 2019). Depending on the origin and composition of the raw wastewater, sludge can be high in organic carbon, plant nutrients, and beneficial microorganisms (Fytili and Zabaniotou, 2008; Zoghlami et al., 2016; Mokni-Tlili et al., 2020). In this regard, millions of metric tons of urban and industrial sludges generated worldwide may be subjected to land application in the most cost-effective way (Rosazlin et al., 2015; Hamdi et al., 2019; Kogbara et al., al., 2020a; 2020b). In addition, the agricultural recycling of these organic wastes is a sustainable solution to prevent open dumping and the subsequent greenhouse gas emissions (Faubert et al., 2019).

It has generally been shown that the improvement of soil properties following sludge application influences positively the growth and yield of agricultural plants. Several studies reported that the increase of plant performance because of sludge application often exceeded that of well-managed fertilized controls (Kogbara et al., 2020a; Kogbara et al., 2020b; Ali et al., 2021). While the majority of published data and reuse guidelines refer to urban sewage sludge as the main wastewater by-product suitable for land application, the agricultural valorization of industrial sludge is far from common. This is because it is less abundant and generally associated to heavy chemical contamination and potential environmental concerns (Mantis et al., 2005; Bin Anwar et

al., 2018). For instance, raw wastewaters and the subsequent sludge generated during styrene–butadiene rubber production (Paśmionka et al., 2022), nitrocellulose manufacturing (Ziganshina et al., 2016), and textile dyeing process (Gottlieb et al., 2003) are highly toxic and can cause serious risks to biota. Therefore, the available literature on industrial sludge reuse in agriculture has mostly focused on biomass-based sludges from food processing and paper mill industries (Mahapatra et al., 2013; Faubert et al., 2019).

Gas-to-liquid (GTL) sludge is one of the least studied industrial sludges worldwide because it is a by-product of wastewater treatment generated during the specific process of natural gas conversion into liquid transportation fuels (Surkatti et al., 2020). Australia and Qatar are presently the main producing and exporting countries of liquefied natural gas (87.6 and 77.4 million metric tons, respectively) followed by the United States, which has an annual capacity of 73.9 million metric tons (Statista, 2022). Because of the abundance of alcohol, ketones, and organic acids, GTL wastewater has a high chemical oxygen demand (COD) and total organic carbon (TOC) content (Surkatti et al., 2020). Once treated and stabilized, the final dry GTL sludge is dark brown to black in color, has an earthy smell and substantial amounts of organic matter (OM) and nutrients (Mabrouk et al., 2023). GTL activities in Qatar produce around 15-18 tonnes of dry sludge daily, resulting in an annual amount of approximately 6000 tonnes. This dry sludge is considered a solid waste and, therefore, is mostly landfilled (Kogbara et al., 2020a). As an industrial sludge deriving exclusively from GTL processes, the major difference with urban sewage sludge resides in the absence of human/animal gastrointestinal pathogens and antibiotic-resistant bacteria as well as residual pharmaceuticals (Zoghlami et al., 2016; Kogbara et al., 2020b; Mokni-Tlili et al., 2022).

To the best of our knowledge, the current research team is the first-ever to have carried out GTL sludge recycling trials as a soil amendment starting from 2016. Therefore, the only available experimental data have been published by Kogbara et al. (2020a; 2020b). It consisted of field studies using various application rates of GTL sludge to grow fodder crops during the cool season of Qatar that roughly extends from November through April. According to the first study, GTL sludge added to an agricultural soil in the range of 0.75% to 3% improved soil physical properties and fertility, which positively influenced alfalfa growth and yield (Kogbara et al., 2020a). The concentration of chemicals found in the leachate and plant biomass grown on sludge-amended soils was either below or similar to the levels observed in control treatments (chemical fertilizer or compost). However, lower biomass yields were noticed when GTL sludge application rates

exceeded 6% with significant decrease at 12%. In a second study, Kogbara et al. (2020b) found that increasing GTL sludge application rates proportionally improved the volume of different soil pore types, especially micropores (up to 56% of cumulative porosity). Consequently, soil water retention was enhanced and positively influenced buffer grass growth especially at GTL sludge doses of 1.5% and 3%.

Soils in arid regions (Vertisols) are generally degraded, poor in OM content, and have a low water-holding capacity (Ahmedna et al., 2016). Qatar, like many hot desert countries, also experiences no rainfall and extreme temperatures during the extended dry season (Ben Hassen et al., 2020). For all these reasons, conventional agricultural activities heavily rely on the usage of water and agrochemicals. In addition, there is a low social motivation and negative public perception on the agricultural reuse of treated wastewater and sludge, especially for growing food crops (Dare et al., 2017; Dare and Mohtar, 2018). Therefore, this field study explored the agricultural recycling of abundant GTL sludge as a sustainable management strategy to mitigate the environmental impacts of current open dumping. Besides, the rationale behind the choice of cultivating cotton resides in its tolerance to extreme conditions and the general social acceptance of growing non-food crops using unconventional inputs (Dare et al., 2017; Khor and Feike, 2017).

2. Materials and methods

2.1. Site Description

The field study was carried out at an Agricultural Research Station located in Northern Qatar (25.82191°N, 51.33107°E). The region has a hot desert climate with long summers extending from May to October, characterized by intense heat waves, high evapotranspiration rates, and fluctuating relative humidity (Issaka et al., 2017; Karanisa et al., 2021). During this season, diurnal temperatures often rise above 40 °C. The mean annual rainfall is about 80 mm and is mostly received during the relatively cooler season (November through April). Therefore, irrigation is required to ensure crop production under field conditions. In this study, cultivation trials were performed in irrigated lysimeters (2.8×2.8×1 m), which are open semi-controlled structures that allow for water movement monitoring. These lysimeters were filled with a typical agricultural soil classified as Vertisol of sandy loam texture with the following soil particle content: sand (62%), silt (29%), and clay (9%). The physico-chemical properties were determined based on Standard Analytical Australasian Methods as described by Rayment and Lyons (2010). Metal

concentrations were determined following Methods 3050B and 6010C (USEPA, 1996; 2007). Fecal coliforms were estimated according to SMEWW-9221 (Lipps et al., 2018). As shown in Table 1, the soil had a low organic matter and nitrogen content, relatively high salinity (2.5 dS m^{-1}), and very high calcium and sodium concentrations, typical characteristics of arid soils. Cotton (*Gossypium hirsutum* L., variety MAY 344) was chosen as the test fiber crop for field trials. According to the producer's catalogue, MAY 344 has been developed in Turkey and consists of an early maturing cotton variety that has a wide boll structure, strong tolerance to drought as well as to *Verticillium* spotting and *Fusarium* wilt diseases, and generates high volume of profitability (Gubre, 2022).

2.2. Soil treatments

In this study, GTL sludge was the main biosolids (BS) to investigate the agricultural aptitude for soil improvement under extreme arid conditions. Accordingly, the aerobically digested sludge was obtained from a local gas-to-liquid industrial water processing plant and had 5–10% moisture content. Cow manure compost (CMC) was chosen as the reference organic amendment and was obtained from the composting unit of a large local dairy Company. The used sludge had higher contents of OM, N and P than CMC, and was pathogen-free since it did not originate from excreta. Because it had an industrial origin, GTL sludge contained substantial amounts of trace elements with particularly high Fe and Zn concentrations compared to CMC and the agricultural soil (Table 1).

Soil treatments consisted of amendments with GTL sludge and CMC as biofertilizers. Gas-to-liquid sludge was applied to soil at two rates (w/w): 1.5% (BS 1.5%) and 3% (BS 3%) whereas the cow manure compost was applied at 1.5% dosage (CMC 1.5%). Both organic materials were first evenly spread onto the soil surface then incorporated by hand hoeing into the top layer (0-10 cm) prior to cottonseed sowing. A chemical fertilization (CF) treatment was also carried out as the conventional practice usually adopted by local farmers for crop management. To this end, N, P, and K were fractioned and supplied according to the fertilization calendar recommended for cotton cultivation. All soil treatments (BS 1.5%, BS 3%, CMC 1.5%, and CF) were performed in triplicate in a completely randomized design. Therefore, 12 lysimeters were used for this field study.

2.3. Cultivation process

Cottonseeds were sown around mid-March 2021 in 3 cm holes dug at 60 cm between rows and 30 cm between plants within the row. As previously mentioned, the used cotton variety was

G. hirsutum L. var. MAY 344 (MAY Seed Company, Adana, Turkey). The full cultivation cycle lasted until the end of December of the same year. Throughout this period, plants were irrigated at set periods of two to three times per week depending on climate conditions. Each lysimeter received water from four corner bubblers connected to a groundwater source at the station. No significant rainfall was ever recorded during the course of cotton cultivation. As cotton is susceptible to a range of destructive pests, chemical treatment against mealybug insects was carried out 1, 5, 7, and 8 months after planting. In total, seven cotton picks (harvest campaigns) were carried out manually for all treatments at 149, 199, 212, 235, 247, 254 and 284 days after seed sowing. Cumulative lint yield (seed-free fibers) was then extrapolated to tonnes per hectare for each treatment. At the final cotton harvest date, leaf and soil samples were taken to investigate the overall effect of different treatments on their properties.

2.4. Soil parameters

Soil samples were taken from the top (0-30 cm) and bottom (30-60 cm) profiles in each replicate lysimeter at the end of the cultivation process. Soil physico-chemical parameters were investigated using air-dried soil samples sieved through 2 mm mesh and stored at 4 °C in the dark. Soil pH and electrical conductivity (EC) were measured in soil-water slurry (1:5). A TOC analyzer (Skalar Primacs MCS - Formacs CA16, The Netherlands) was used to estimate the organic matter content. Total Kjeldahl nitrogen was determined according to Bremner and Mulvaney (1982). Extractable phosphorus was measured by $NaHCO_3$-ascorbic acid method (Olsen et al., 1954). Anions and cations in soil were extracted by shaking for 24 hours in deionized water, filtered through 0.45 µm then analyzed using a 850 Professional Ion Chromatography (Metrohm, Switzerland). For heavy metal analysis, 0.25 g of soil sample were first added to digestion tubes along with 6 mL nitric acid, 2 mL hydrochloric acid, and 2 mL hydrofluoric acid then microwave digested (MARS 6, CEM Corporation, USA). Trace element concentration was determined by ICP-OES (Optima 7300 DV, PerkinElmer, USA). Simultaneously, leachate samples were also analyzed for heavy metals drained throughout the whole soil profile in each lysimeter. Collected leachates were first filtered through 0.45 µm to eliminate solid particles then stored at 4 °C in the dark before analysis. The analysis of dissolved metals was determined using an iCAP 6000 Series ICP-OES (Thermo Scientific, USA) after dilution with 2% nitric acid solution.

2.5. Plant parameters

2.4.1. Major elements in leaves

Fresh leaf samples were taken randomly from each replicate lysimeter and dried at 70 °C for 48 hours. Then, 0.5 g of dry ground leaf material was sonicated in 50 mL deionized water. The suspension was further mechanically shaken for 20 min, centrifuged then elemental analysis was carried out using a 850 Professional Ion Chromatography (Metrohm, Switzerland). A FlashSmart Analyzer (Thermo Fisher Scientific, USA) using dynamic flash combustion of the dried leaf material helped to determine the C.H.N composition.

2.4.2. ABTS antioxidant assay

To determine the oxidative stress that might be caused to plants by soil treatments, the ABTS (2,2-azinobis (3-ethylbenzothiazoline-6-sulfonic acid)) assay (aka trolox equivalent antioxidant capacity, TEAC) was performed according to SIGMA-ALDRICH Antioxidant Assay Kit (Merck, USA). Fresh cotton leaves were first grinded using liquid nitrogen then 0.1 g of the sample was extracted using 0.5 mL of buffer solution. After centrifugation for 15 min, the supernatant was collected and stored at -20 °C prior to testing. The final reaction solution contained 10 µL leaf extract mixed with 20 µL myoglobin working solution and 150 µL ABTS substrate. After incubation for 45 min in the dark, 150 µL of stop solution was added and samples were run into Agilent BioTek Synergy H4 Hybrid Microplate Reader at 405 nm. Antioxidant activity in leaves was expressed as mmol TEAC g^{-1}.

2.4.3. Fiber yield and quality

Total lint yield (seed-free fibers) was estimated from the cumulative seed cotton yield on the basis of 60:40 seed:fiber ratio as described by Boykin et al. (2010). Seeds in raw cotton were manually removed then a composite lint sample from each replicate treatment was analyzed for fiber quality. A High Volume Instrument (HVI) (USTER® HVI 900A, USA) allowed to determine the following fiber parameters: micronaire, reflectance, length, maturity, and strength (Paudel et al., 2013).

2.5. Statistical analysis

The effect of the four soil treatments on plant properties was analyzed by ANOVA with a post hoc Duncan's multiple range test ($P \leq 0.05$) for triplicate mean separation (STATISTICA 8.0, StatSoft Inc., Tulsa, USA). The variation of soil parameters was assessed for both sampling depths (0-30 and 30-60 cm) at $P \leq 0.05$.

3. Results and discussion

3.1. Soil fertility parameters

Organic matter is commonly considered as the most important component of soil fertility due to its role in improving soil physico-chemical and biological properties as well as plant nutrition (Kominko et al., 2017). Chemical fertilizers may sustain crop production by directly supplying available mineral forms of macro- and micro-nutrients but would never result in soil OM increase and quality restoration (Hammad et al., 2020). This was evidenced in this study as both organic materials increased soil organic matter content significantly as compared to chemical fertilization (Fig. 1A). Besides, organic matter was higher in topsoil (0-30 cm) than in subsoil (30-60 cm) due to the surface incorporation of cow manure compost and GTL sludge. This depth-dependent decreasing pattern was also applicable for most of analyzed parameters. On the other hand, there was no significant difference in topsoil organic matter content between biowaste-treated soils. Numerically, BS 3% ranked highest followed by CMC 1.5% and then BS 1.5% with OM levels of 4.54%, 4.37% and 4.01% respectively. These results are in line with previous studies where adding sludge increased soil organic matter content in a dose-dependent manner (Navas et al., 1998; Hamdi et al., 2019; Hechmi et al., 2020). However, the absence of significance in terms of soil OM content indicates that the extreme weather conditions coupled to microbial activities and cotton growth stabilized the carbon stock in soil towards the end of the cultivation process (Lal, 2009).

Primary macronutrients (NPK) are structural units essential for any higher plant to complete its life cycle and play a very important role in improving crop yield and quality (Maathuis, 2009). In particular, nitrogen is considered an essential parameter in the assessment of biosolids quality (Jiang et al., 2019). As shown in Figure 1B, there was no significant difference of soil N among the treatments for both soil depths at the end of the cultivation process. Chemical fertilization showed the highest N level in topsoil profile (0.92%) since it was directly supplied under mineral form in three fractions. At an equivalent application rate of 1.5%, GTL sludge resulted in higher N content (0.86%) than CMC (0.56%). Sludge is known to contain more N than animal manure (Antil et al., 2011); the used GTL sludge had eventually two-fold higher N content than CMC as shown in Table 1. This was reflected by enhanced N levels in leaves of cotton plants grown on sludge-amended soils at the end of cultivation cycle (Fig. 2B). As such, leaves of plants

grown on sludge-amended soils had the highest N content (2.8% and 2.9% for BS 1.5% and BS 3%, respectively).

The second essential macronutrient for plants is phosphorus (Jiang et al., 2019). Analytical data showed that adding GTL sludge at 3% resulted in the highest topsoil available P content (1089 mg/kg), which is almost 8-fold higher than in CF and CMC 1.5% treatments (Fig. 1C). This is explained by the very high concentration of P in GTL sludge, which was 8100 mg kg^{-1} as compared to CMC (1600 mg kg^{-1}) (Table 1). Phosphorus in cotton leaves varied proportionally with soil P, resulting in highest values in presence of 3% GTL sludge (Fig. 2C). High concentrations of P in biosolids were previously reported in many studies but the GTL sludge has particularly an excessive content (Hamdi et al., 2019; Zhang et al., 2019). Iqbal et al. (2020) reported that P plays a pivotal role in cotton by enhancing the reproductive growth and yield formation. It has been documented that sludge-amended soils have extractable P concentrations that are at least ten times higher than the suggested upper limit for agricultural soils (McBride, 2022). This is very important for the productivity of P-demanding crops like cotton (Qureshi et al., 2012) but may pose environmental risks mainly related to surface runoff (Doydora et al., 2020). Accordingly, periodical leachate analysis from all lysimeters did not reveal detectable P concentrations under the described experimental conditions testifying to its low vertical mobility (Shen et al., 2011).

Potassium is the third listed essential macronutrient to plants. Compared to P, K is relatively a mobile element in the soil (Lalitha and Dhakshinamoorthy, 2014). Unlike other nutrients, the potassium found in manure and biosolids becomes quickly available to the plant after soil application since it is not bound to organic elements and is not a constituent of biomolecules (Sardans and Peñuelas, 2015). As illustrated in Figure 1D, there was a significant difference in K concentration (0-30 cm) between conventional treatments and GTL sludge-treated soils. Despite the significant 10-fold higher available K concentration in CMC (16 g kg^{-1}) compared to GTL sludge, content in soil at the end of cotton cycle was in the following order: BS 1.5% > BS 3% > CMC 1.5%. A similar translocation pattern was also observed in cotton leaves (Fig. 2D), indicating a close relationship between plant mineral nutrition and soil fertility (Ordoñez et al., 2009).

3.2. Soil quality parameters

3.2.1. Changes in pH and salinity

As shown in Figure 3, there was no significant difference between pH values in the top soil layer (0-30 cm) for all treatments in contrast to bottom layers (30-60 cm). However, the average

pH in the whole soil profile for each treatment showed neutral pH variations of 7.4 to 7.6. Gas-to-liquid sludge has a slightly lower pH compared to the original agricultural soil (Table 1), and the sludge dose effect was obvious especially at the subsoil level (7.23 for BS 3%). In general, the degradation of organic matter has been shown to decrease soil pH in the long term (Hamdi et al., 2007; Skowrońska et al., 2020). Buchanan and Ippolito (2021) also observed a similar trend of pH decrease with biosolids application rate. This decrease has been linked to the aerobic biodegradation of organic carbon, which leads to the formation of carbonic acid (H_2CO_3) in the soil solution (Stevenson, 1994; Hamdi et al., 2007; Hechmi et al., 2021). However, the field experimental conditions characterized by only one application of organic materials coupled to the buffer capacity of the agricultural soil due to high Ca content (3.9 g kg^{-1}, Table 1) were likely to have all participated in keeping organic-amended soils within a neutral pH range (Hamdi et al., 2019; Kang et al., 2024)

Soil EC has been recognized in the scientific literature as an index of soil salinity (Rashidi and Seilsepour, 2011). As shown in Figure 4, there was a significant difference in salinity between the soils amended with CF and CMC compared to GTL sludge for the upper layer (0-30 cm). However, EC values were lower in the bottom layer (30-60 cm) for all treatments. This is due mainly to the surface incorporation of organic materials and chemical fertilizers, where soluble ions are loaded and active mineralization occurs (Vilkienė et al., 2016). GTL sludge addition at both rates significantly increased EC values compared to CMC 1.5% and CF treatments. These outcomes corroborate with those observed by Hamdi et al. (2019) and Hechmi et al. (2020) where sludge addition resulted in a consistent dose-dependent increase of EC with respect to control treatments. Besides, topsoil EC determined in soil:water suspension (1:5) of BS 1.5% treatment already exceeded the soil salinization reference value of 4 dS m^{-1} for saturated paste extracts (USDA-NRCS, 2014). This indicates that the actual salinity of the GTL sludge-amended soils (ECe) should be significantly higher than those reported in this study because positive linear relationship connects both EC measurements (Kargas et al., 2020).

3.2.2. Heavy metal contamination

Trace elements have historically been the most addressed contaminants in biosolids and the major concern that restricts their agricultural valorization especially when growing food crops (Geng et al., 2020). As such, quality guidelines on the safe reuse of sludge ubiquitously include limits for heavy metals (Hudcová et al., 2019). According to USEPA (1980), recommendations for

sludge agricultural reuse can be less stringent with non-food-chain crops such as forest nurseries, energy biomass trees, and fiber crops. On the other hand, the accumulation of heavy metals in amended soils has been shown to mainly depend on metal content in the organic amendment and agricultural soil, as well as the applied dose and frequency of applications (Gu et al., 2013; Hamdi et al., 2019; Hechmi et al., 2020). The concentration of phytotoxic metals found in different soil treatments is presented in Figure 5. Other metals of environmental concern such as As, Co, Cd, and Ni were not detected. As for the rest of soil parameters, Pb, V and Cr concentrations in the topsoil layer were higher than in subsoil.

Overall, soil treatments did not significantly increase the content of detected phytotoxic metals with respect to the original soil under the described experimental conditions except for Cr (Fig. 5C). The latter had already higher concentration in GTL sludge (74 mg kg^{-1}) and CMC (55.4 mg kg^{-1}) than the used agricultural soil. However, detected Pb, V and Cr content in soil did not exceed the limits set by several international guidelines (USEPA, 1994; Hudcová et al., 2019). In addition, neutral pH values and high OM content in CMC- and GTL sludge-amended soils would limit the bioavailability of some metals/metalloids in the soil solution (Smith, 1994; Hou et al., 2019). Among all analyzed trace elements (As, Pb, Co, Cd, Cr, Cu, Fe, Mn, Mo, Ni, V, and Zn), only zinc and vanadium were detected in leachates from different soil treatments as shown in Figure 6. While Zn is considered a highly mobile and less hazardous oligo-element, water-dissolved V concentrations were low (0.33-0.55 mg L^{-1}) and did not show significant differences among the treatments (Fig. 6B). This indicates that all soil treatments had some intrinsic retention capability enhanced by the addition of the organic materials, in particular cow manure that had higher V content than sludge (Baldi et al., 2021). Vanadium is known to have a strong sorption onto the soil particles (Larsson et al., 2013). On the other hand, the presence of high Ca content in soil, a characteristic of Qatar aridisols, results in the formation of low soluble calcium orthovanadate ($Ca_3(VO_4)_2$) (Hobson et al., 2018).

3.3. Production parameters

3.3.1. Cotton yield

The cumulative cotton yield obtained over seven harvesting campaigns for each treatment was expressed in tonnes per hectare (t ha^{-1}) to allow for comparison with field data reported in literature. In this study, hand harvesting was performed each time cotton bolls had been fully opened at 70-80% (Meng et al., 2021). In this regard, all treatments almost showed the same

physiological behavior in terms of cotton fiber development and maturation dates independently of the soil treatment. As illustrated in Figure 7A, the cumulative seed cotton yield was significantly higher when using GTL sludge compared to chemical fertilizer and animal manure. Total seed cotton production rates were approximately 14 t ha^{-1} for BS 3% followed by BS 1.5% (11.7 t ha^{-1}), which represents a yield increase of 56.2% and 47% compared to regular CF treatment, respectively. The lowest cumulative yield was significantly observed in presence of cow manure compost (3.43 t ha^{-1}). Consequently, the total lint (pure fiber) yield followed the same linear variation as indicated by Boykin et al. (2010), and was therefore the highest for BS 3% (5.66 t ha^{-1}) (Fig. 7B). This also shows that all the observed physico-chemical changes in GTL sludge-amended soils ultimately resulted in a positive dose-dependent enhancement of cotton growth. Seilsepour and Rashidi (2011) linked the increase of lint yield to the concentration of N in cotton leaves at harvest time, which was the case for the current study (Fig. 2B). In addition, the presence of significant amounts of microelements in GTL sludge (Ca, Fe and Zn) was likely linked to cotton yield improvement as they are essential elements for some plant physiological functions and resistance to environmental stressors (Yaseen et al., 2013; Rutkowska et al., 2014). In fact, post-harvest field observations revealed a striking visual difference in plant biomass development between treatments. In general, cotton plants grown on CF and CMC-treated soils showed visible signs of exhaustion as compared to healthier sludge-treated plants (Fig. 8). This was also evidenced by significantly higher plant ABTS antioxidant activities with respect to sludge-amended soils (0.43, 0.45, 0.28, and 0.16 mmol TEAC g^{-1} for CF, CMC 1.5%, B 1.5%, and BS 3%, respectively). Previous cropping trials under similar experimental conditions reported that the same GTL sludge outperformed CF and green waste compost in terms of alfalfa and buffel grass growth (Kogbara et al., 2020a; 2020b).

The reported cumulative yields were nonetheless exaggerated if compared to maximum reported lint yield of 2.1 t ha^{-1} achieved in China (Jabran et al., 2020). When cotton is handpicked at large-field scale, it requires multi plucking in two to three cycles (Deshmukh and Mohanty, 2016). In this study, seven successive harvesting campaigns were technically possible because of the relatively small surface area of the experimental site. In addition, it is likely that extreme temperatures coupled to full irrigation regime resulted in short boll maturation cycles. Snowden et al. (2013) obtained the highest lint yield when cotton plants were fully irrigated during a hot and dry year in Texas (1.5 t ha^{-1} for total water amount of 684 mm). The current region of study is by

far among the hottest and driest in the world (Karanisa et al., 2021). Therefore, a total water amount of 1100 mm was needed to maintain cotton plants throughout the whole cultivation cycle.

3.3.2. Fiber quality

Cotton fiber quality has a huge impact on the efficiency of processing, the quality of the yarn produced, and the value of the textiles and end-products when brought to the market (CottonWorks, 2023). Even if cotton yield can be enhanced through adapted agricultural practices, no after-harvest mechanisms are available to either growers or processors that can improve intrinsic fiber quality (Bradow and Davidonis, 2000). To categorize cotton fiber quality, several parameters have often been considered such as fiber length, fineness, moisture regain/content, tensile strength, and whiteness. The cultivated cotton species was *Gossypium hirsutum* var. MAY 344, which is an upland cotton originating from Central America and Mexico. In contrast to soil properties and lint yield, there was no significant variability in the addressed quality parameters with soil treatments (Table 2). For instance, Seilsepour and Rashidi (2011) did not observe any effect of N significant increase in soil and leaves on cotton quality as well. This suggests that the cotton variety and climate conditions are the major factors responsible for fiber quality while soil conditions only influence cotton yield (Seilsepour and Rashidi, 2011; Zafar et al., 2022). Fiber color is assessed with a cotton colorimeter and expressed in degrees of reflectance. It usually varies from 50 to 85 units in correlation to fiber whiteness (CottonWorks, 2023). As shown in Table 2, reflectance values varied between 77.6 and 79.6, which indicates a shiny white color cotton. The length of fiber in all treatments averaged between 27.68- 28.63 mm. This is considered a short staple fiber characterizing upland cotton according to Hasan (2022). This trait is due to high temperatures that prompted cellulose synthesis and hence fiber elongation and maturity (Loka and Oosterhuis, 2010). Likewise, physiological responses to extreme temperatures greatly affect cotton fiber strength (Jiang et al., 2006). The fiber strength in all treatments is considered average since values varied between 26 and 28 g tex^{-1} (Hasan, 2022). In addition to the effect of extreme heat waves and occasional high relative humidity that coincided with cotton fiber development and maturation period (Hsieh et al., 2000), the chosen cotton variety and the manual ginning practiced to separate lint from seeds may have all influenced the fiber strength properties observed in this study.

Micronaire is one of the most important indicators of cotton quality. It is a measure of the air permeability of compressed cotton fibers and often used as an indication of fiber fineness and

maturity in HVI testing of quality traits (USDA-AMS, 2013). Marketers and producers of cotton prefer a micronaire range of 3.8 to 4.5. Thicker or thinner fibers present issues in spinning as well as uniform dyeing of yarn (Hake et al., 1990). In this study, the micronaire averaged between 4.36-4.62, showing an acceptable fiber fineness and correlates therefore with the observed high maturity rates (0.86-0.95). This corroborates with a previous study where different cotton varieties grown in Pakistan (dry and warm climate) showed micronaire values between 4.3 and 4.5 (Khatri et al., 2018). Compared to water-stressed cotton plants, Snowden et al. (2013) observed an increase of micronaire (up to 4.91) when plants were fully irrigated during a hot and dry year. In this study, the highest micronaire value of 4.62 in treatment BS 3% was likely due to a greater soil water retention in relation to the increase of organic matter content up to 4.54% (Lal, 2020).

4. Conclusions

To our best of knowledge, this is the very first reported data on the agricultural reuse of GTL sludge to grow fiber crops as an alternative option to uncontrolled landfilling. One of the main outcomes was the successful enhancement of soil properties and subsequently cotton production under the hyperarid conditions of the Arabian Desert. More precisely, only one sludge application of 1.5% and 3% resulted in the significant improvement of soil carbon stock and plant growth compared to the more conventional practices. Although heavy metal contamination was not significant, soil salinization should be closely monitored if repetitive applications would occur on the same soil using crops that are sensitive to saline conditions. To reduce possible environmental impacts, a GTL sludge application rate of 1.5% would be provisionally recommended since cotton yield did not significantly increase when sludge dose was doubled.

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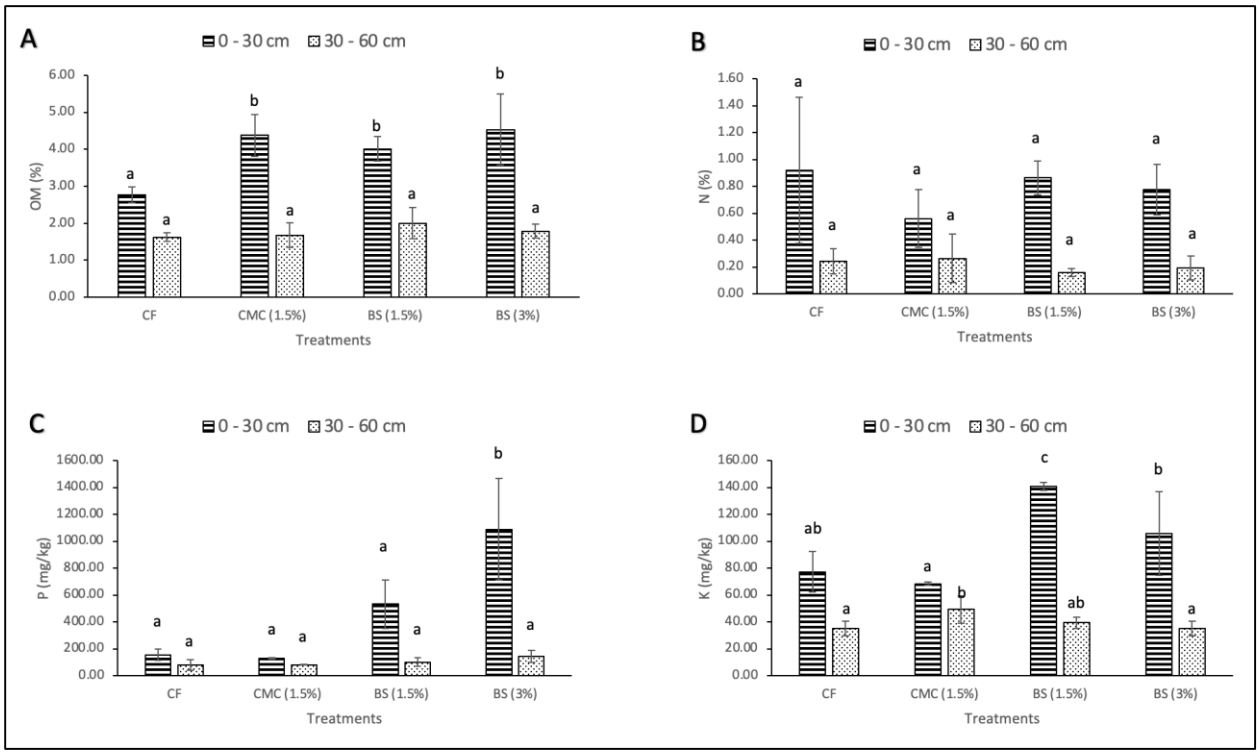

Figure 1. Soil organic matter (A), nitrogen (B), phosphorus (C), and potassium (D) at the final cotton harvest time. CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). For each soil depth, treatment means with the same small letters are not statistically different at $P \leq 0.05$.

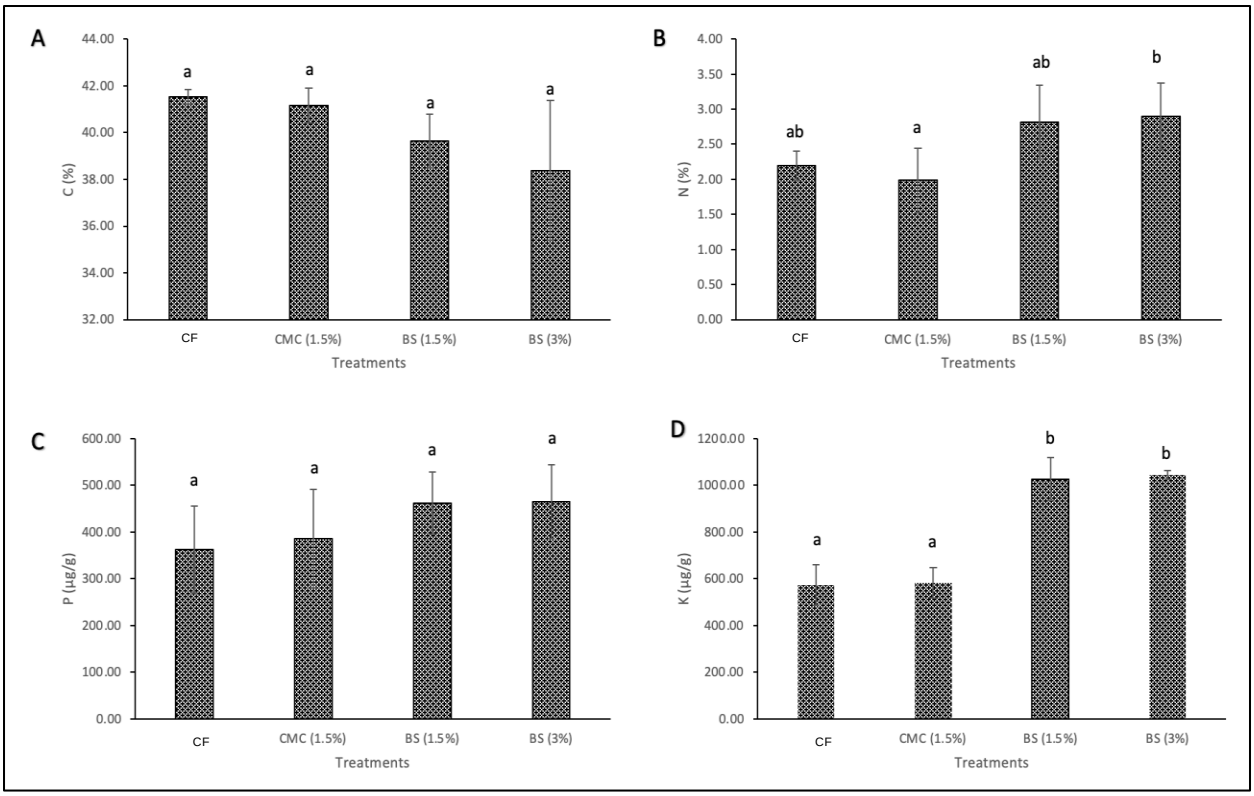

Figure 2. Elemental composition of cotton leaves at the final cotton harvest time. Carbon (A), nitrogen (B), phosphorus (C), and potassium (D). CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). For each element, treatment means with the same small letters are not statistically different at $P \leq 0.05$.

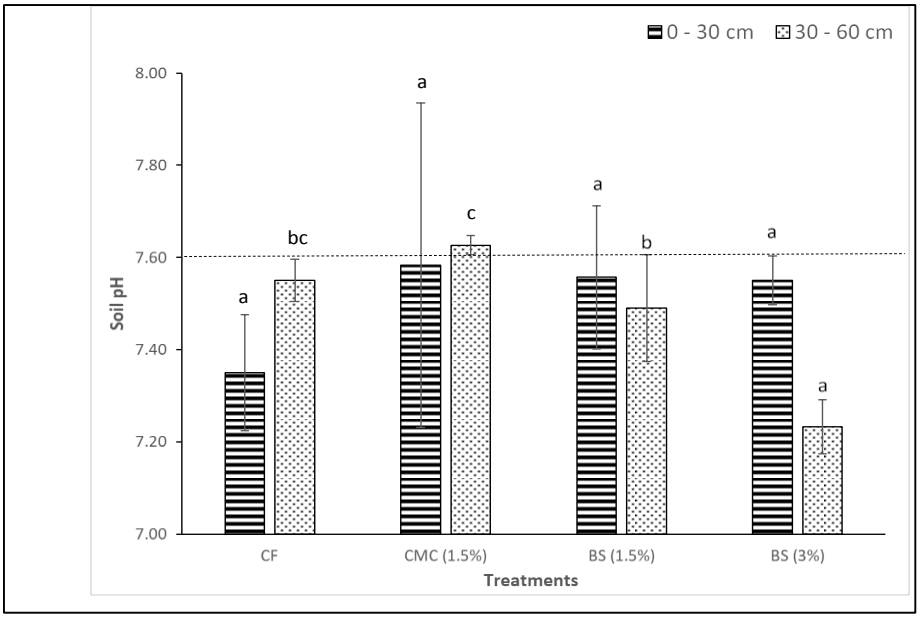

Figure 3. Soil pH at the final cotton harvest time. CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). Dashed line represents pH value of the original soil. For each soil depth, treatment means with the same small letters are not statistically different at $P \leq 0.05$.

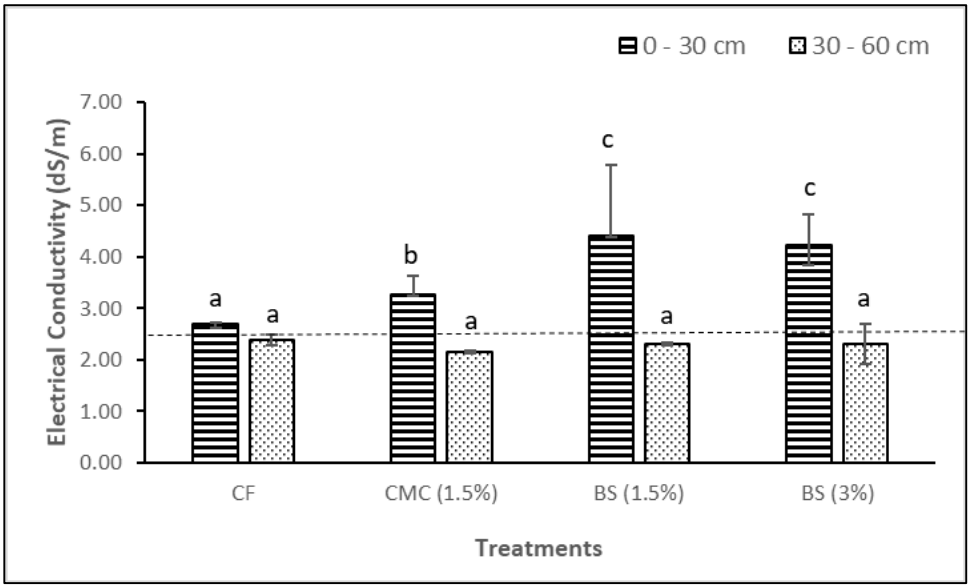

Figure 4. Soil EC at the final cotton harvest time. CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). Dashed line represents EC value of the original soil. For each soil depth, treatment means with the same small letters are not statistically different at $P \leq 0.05$.

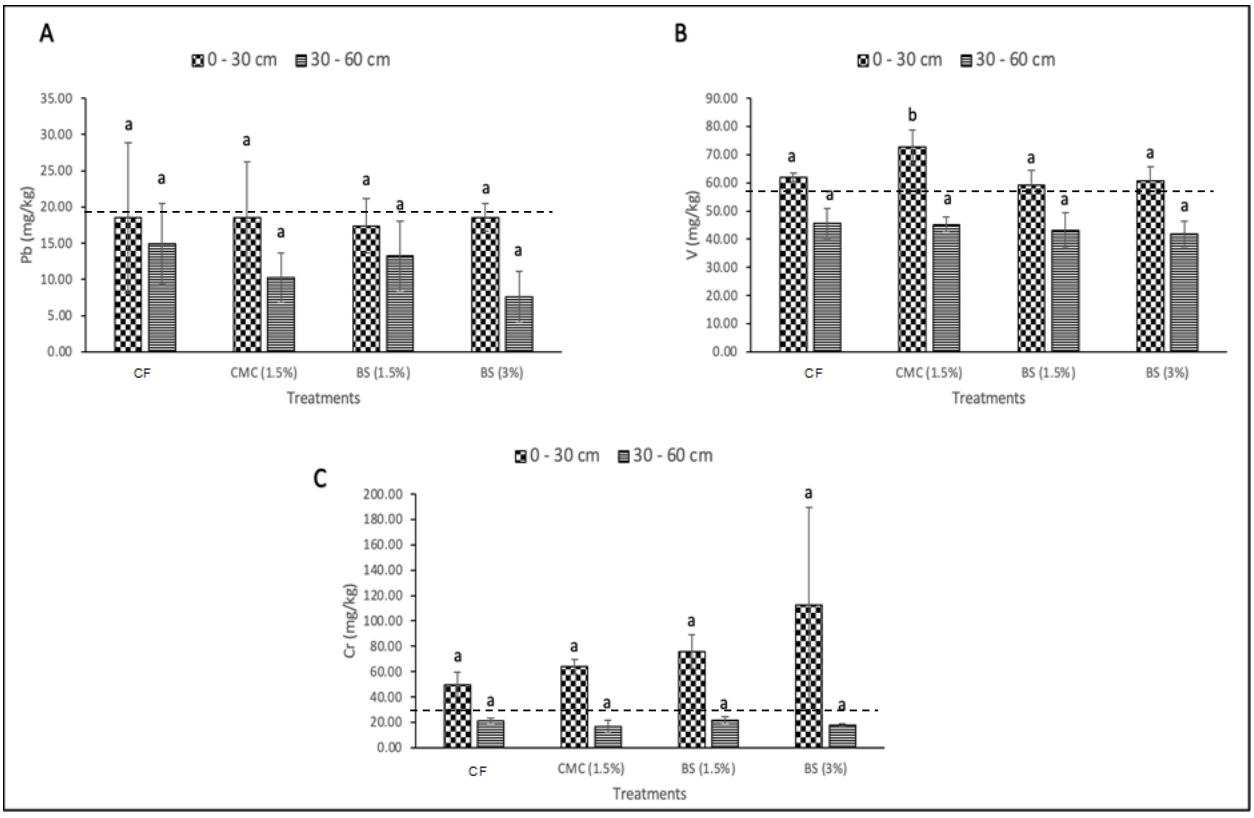

Figure 5. Phytotoxic metals in soil at the final cotton harvest time. Pb (A), Vanadium (B), and Chromium (C). CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). Dashed line represents the concentration of each element in the original soil. For each soil depth, treatment means with the same small letters are not statistically different at $P \leq 0.05$.

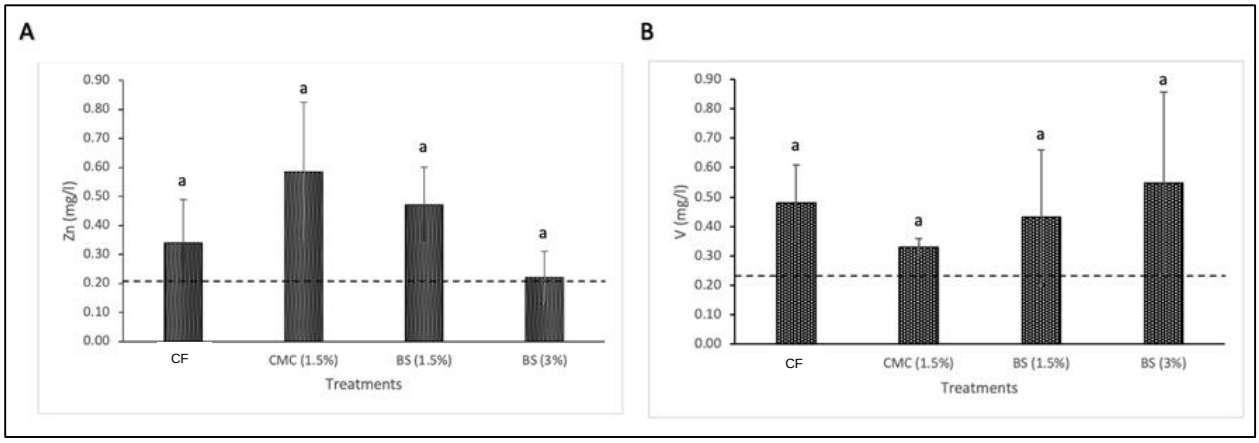

Figure 6. Zinc (A) and vanadium (D) concentrations in leachate samples at the final cotton harvest time. CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). Dashed line represents concentration in leachates from the original soil. Treatment means with the same small letters are not statistically different at $P \leq 0.05$.

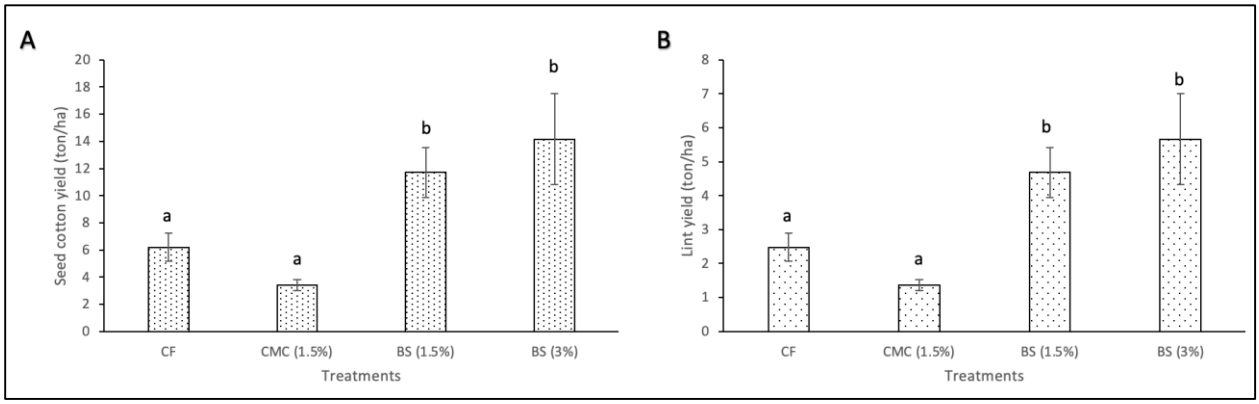

Figure 7. Cumulative seed cotton yield (A), and lint yield (B). CF (chemical fertilizer), CMC 1.5% (cow manure compost added at 1.5%), BS 1.5% (GTL sludge added at 1.5%), and BS 3% (GTL sludge added at 3%). Treatment means with the same small letters are not statistically different at $P \leq 0.05$.

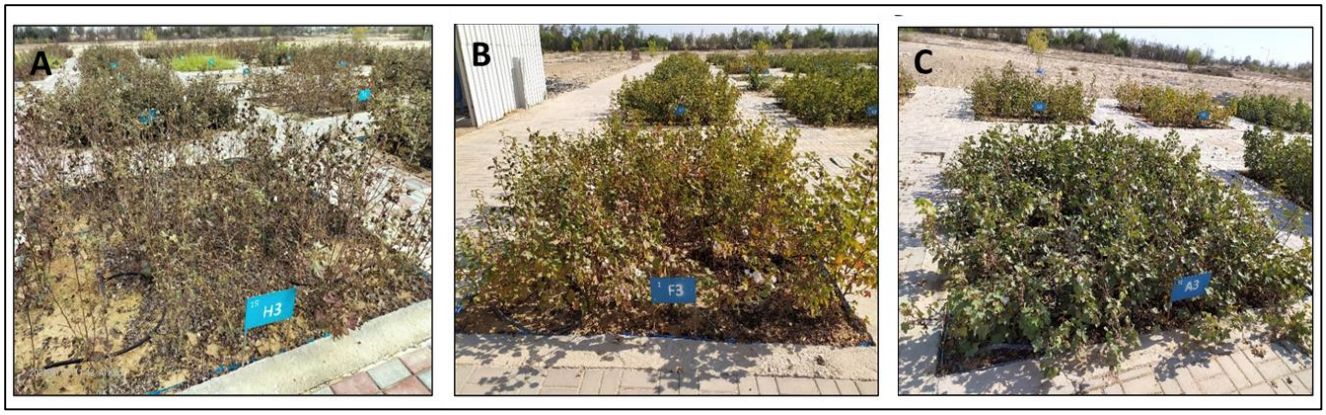

Figure 8. Cotton plant appearance at post-harvest time in different treatments. A: CF; B: CMC 1.5%; C: BS 1.5%.

List of Tables

Table 1. Physico-chemical and microbial properties of experimental soil, GTL sludge, and cow manure compost

Parameters	Soil	GTL Sludge	Cow manure compost
pH	7.61	6.47	8.67
EC (dS m^{-1})	2.5	3.85	3.24
OM, % (w/w)	1.86	68	34.16
N, % (w/w)	0.03	6.1	2.99
P (mg kg^{-1})	14.1	8100	1600
K (mg kg^{-1})	144	1600	16000
Na (mg kg^{-1})	580	2250	1898
Ca (mg kg^{-1})	3908	4600	924
Mg (mg kg^{-1})	277	972	112
Cl (mg kg^{-1})	363	1520	1837
Cr (mg kg^{-1})	30.2	74	55.4
Cu (mg kg^{-1})	45.3	49.1	52.5
Mo (mg kg^{-1})	3.02	84.8	6.93
Pb (mg kg^{-1})	18.6	18.7	7.09
V (mg kg^{-1})	57	< 0.5	32.35
Zn (mg kg^{-1})	14.34	660	250.7
Fe (mg kg^{-1})	14875	39900	1244
Mn (mg kg^{-1})	205.2	1109	175.3
Total bacteria (mg kg^{-1})	8.3×10^{3}	1.12×10^{8}	4.05×10^{6}
Fungi (mg kg^{-1})	9.43×10^{2}	7.71×10^{6}	7.35×10^{3}
Total coliforms (mg kg^{-1})	nd	nd	3.59×10^{6}
Fecal coliforms (mg kg^{-1})	nd	nd	2.26×10^{5}

nd: not detected

Table 2. Quality parameters of the produced cotton fibers

	Micronaire	Length (mm)	Reflectance	Maturity (%)	Strength (g tex^{-1})
CF	4.56 ± 0.4^{a}	28.63 ± 0.3^{c}	78.23 ± 0.7^{a}	0.92 ± 0.03^{ab}	27.8 ± 0.5^{a}
CMC 1.5%	4.36 ± 0.1^{a}	28.46 ± 0.3^{bc}	79.63 ± 0.5^{a}	0.91 ± 0.01^{a}	26.6 ± 0.9^{a}
BS 1.5%	4.47 ± 0.6^{a}	27.88 ± 0.3^{ab}	79.07 ± 0.3^{a}	0.92 ± 0.02^{ab}	27.7 ± 0.7^{a}
BS 3%	4.62 ± 0.3^{a}	27.68 ± 0.5^{a}	77.63 ± 0.9^{a}	0.94 ± 0.01^{b}	27.0 ± 0.2^{a}

*Treatment means with the same small letters are not statistically different at $P \leq 0.05$