

Effect of sewage sludge, sewage sludge compost and sewage sludge biochar on heavy metal and its fractions

ABSTRACT

Aim: The study was conducted to evaluate the effect of sewage sludge (SS), sewage sludge compost (SSC) and sewage sludge biochar (sewchar, SC) on the heavy metal content and its fractions.

Study design: The preparation of sewage sludge compost, sewchar and incubation study were done in completely randomized block design.

Place and duration of study: Department of Soil science and agricultural chemistry, College of agriculture, Vellayani, Thiruvananthapuram, India.

Methodology: The experiment studied the effect of composting and pyrolysis on the total, available and heavy metal fractions. Sewage sludge compost was prepared by using sewage sludge, sawdust and zeolite in the ratio 50: 30: 20 and for the adjustment of pH 2.5 kg flyash was used. Sewchar was prepared through the process of slow pyrolysis at a temperature of 400°C for 2 hours using the muffle furnace. An incubation experiment was conducted by using different ratios of sewage sludge biochar, sewage sludge and sewage sludge compost and maintained at field capacity for 180 days.

Results: Experimental results showed that conversion of sewage sludge to sewchar causes the enrichment of nutrients (except N) and heavy metals (Cd and Cr) in them. Total Cd and Cr in the sewchar increased during the pyrolysis process and a reduction during the composting process was observed. Composting and pyrolysis decreased the available Cd and Cr content in the incubation soil compared to the sewage sludge amended soil. Fractionation studies also showed that there was a reduction in the unstable fractions of Cd and Cr as the incubation days progressed from 0 to 180 days. Composting and pyrolysis can be considered as an effective way to decrease the availability of heavy metals as it can convert the unstable fractions of heavy metals to stable fractions.

Key words; *Heavy metals, sewage sludge, biochar, fractionation*

1. INTRODUCTION

Rapid population expansion, industrialisation and urbanisation have resulted in massive sewage sludge creation in India's main cities, with an annual sludge production of 277 million tonnes [1]. Sewage sludge is an inevitable byproduct from the waste water treatment process contain organic compounds, macro and micro nutrients, pathogens and microorganisms. Improper handling of the sewage sludge creates more serious problem as it may cause secondary pollution, especially the heavy metals in sewage sludge can create soil and ground water pollution [2]. Land application, landfill disposal and incineration are methods for disposing of sewage sludge. Reusing sewage sludge in agriculture is the most cost-effective method of recycling organic matter and plant nutrients into the soil for crop production. The use of sewage sludge has an impact on the environment since it can lead to the accumulation of hazardous heavy metals and the presence of harmful microbes. Sewage sludge can be used as a nutrient source for plants and crops if the heavy metals are effectively stabilized and further studies are required in this field. The availability of heavy metals present in the sewage sludge can be minimised by converting it into compost and biochar.

By adding different additives, sewage sludge composting can reduce the amount of available heavy metals [3]. The breakdown of insoluble carbonates, the adsorption of heavy metals by bulking agents or the creation of organo-metallic compounds during the composting process could all be contributing factors to the decrease in the bioavailability of heavy metals. Sewage sludge can be composted using a variety of additions, including sawdust, zeolite, coal flyash, coirpith, and lime [4].

Composting of sewage sludge using bulking agents, heavy metal adsorbent (zeolite) and liming materials (lime and flyash) reduces the mobile (exchangeable) heavy metal fraction and increases the stable (residual) fraction [5].

Pyrolysis has recently undergone a viable strategy for the sustainable treatment of sewage sludges [6]. According to Jin et al. (2016), pyrolysis reduces the amount of sludge, eliminates any pathogens or parasites, converts organic materials into bioenergy and immobilises metals in a charred carbonaceous solid residue. By using the pyrolysis method, metals in sewage sludge can be significantly changed from weakly bound forms to more stable states (in oxidisable and residual forms) [7]. This reduces the environmental risk associated with applying sludge biochars to land.

Sewage sludge, its compost and biochar contain various kinds of heavy metals, which are non-biodegradable. Their mobility and bioavailability within a particular medium are critical factors to consider when evaluating their environmental impact. However, their chemical speciation in sludge, compost and biochar can also be used to accomplish this [8].

The amount of pollution is indicated by the total concentrations of heavy metals. However, the total concentration of heavy metals provides us very little about the forms that these metals can take or about how mobile and bioavailable they can be in the environment [9]. The overall heavy metal content of sewage sludge, measured on a dry weight basis, ranges from 0.5-2.0%, yet it can occasionally reach 4%, particularly for metals like Cu and Zn [10]. The process of recognising and measuring the various phases or forms that a substance has is known as chemical speciation [11].

[12] identified five fractions that heavy metals such as exchangeable fraction impacted by the process of sorption and desorption. The carbonate fraction is impacted by pH variations, whereas the reducible fraction, which is made up of iron and manganese oxides and is thermodynamically stable in anoxic environments and the organic fraction degrade and release soluble metals when exposed to oxidising conditions. Residual fractions made up of main and secondary minerals that have the potential to contain metals in their crystal structures. In this study the effect of composting and pyrolysis of sewage sludge is compared on the basis of immobilization of heavy metals by conducting an incubation experiment for 180 days.

2. MATERIALS AND METHODS

The study involved the use of sewage sludge, sewage sludge compost and sewchar. Sewage sludge used for the present study was collected from waste water treatment plant Muttathara, Thiruvananthapuram, Kerala. Composite samples of sewage sludge was collected from the drying yard of treatment plant. Sewage sludge compost was prepared by mixing sewage sludge with sawdust and zeolite in the ratio 50:30:20 added 2.5 kg of fly ash for the adjustment of pH and composted for 60 days. Sewchar was prepared using muffle furnace. Air dried, 2mm sieved sewage sludge was placed in the crucibles and placed in the muffle furnace and pyrolyzed at 400°C for 2 hours. After the completion of pyrolysis, muffle furnace was allowed to cool over night. Sewchar so produced were collected and stored in air tight container for further chemical analysis.

The air dried sewage sludge, sewage sludge compost and sewchar were separately crushed and sieved through 2mm sieve for laboratory analysis and use in the incubation experiment.

In order to evaluate the impact of sewage sludge, its compost and biochar on total and available heavy metals and heavy metal fractions an incubation study was carried out under laboratory conditions for 180 days. Five kg of 2 mm sieved, air-dried soil samples were placed in a pot, given various treatments and left in the laboratory for 180 days. The treatment details: T₁ - Absolute control (5 kg soil alone), T₂ - 5kg soil + 12.5 g sewchar, T₃- 5kg soil + 25 g sewchar, T₄ - 5kg soil+ 37.5 g sewchar, T₅- 5kg soil + 50 g sewchar, T₆- 5kg soil + 50 g sewage sludge compost, T₇- 5kg soil + 50 g sewage sludge , T₈- 5kg soil + 50 g FYM

pH and electrical conductivity were measured using an aqueous extract of dried materials in distilled water (1:5 w/v), using a CyberScan PC510 pH meter and a Systronics MK509 conductivity meter, respectively. Using the vario EL cube elemental analyser, the weight loss upon ignition of the dried materials was used to determine the total organic carbon. Kjeldahl method was used for the determination of total N, P by colorimetry and K by flame photometry. Using an inductively coupled

plasma-optical emission spectrophotometer (ICP-OES, Optima 8000), the elements Cd and Cr were analysed. The procedure for sequential extraction of heavy metals are given in table 1.

Table 1. Sequential extraction procedure

Fractions	Reagents
Exchangeable (F ₁)	1 mol/L Mg Cl ₂ 8 ml, shake for 1 h
Carbonate (F ₂)	1 mol/L NaAc 8ml, shake for 8 h
Reducible (F ₃)	0.04mol/L NH ₂ OH.HCl 20 ml, in a bath of 96 °C, shake for 6 h
Oxidizable (F ₄)	30 % H ₂ O ₂ , heating at 85 °C mix intermittently for 3 hr
Residual (F ₅)	HCl + HNO ₃ +HF, digest

3. Results and discussion

As shown in table 2 the sewage sludge used in this study had a pH of 5.36 and organic carbon content of 17.03 % . It was rich in plant nutrients N (1.68 %), P (7.73 %) and K (1.2 %). The sludge contained heavy metals Cr (90.74 mg kg⁻¹) and Cd (8.43 mg kg⁻¹). When the sewage sludge is used for the preparation of sewage sludge compost, the total nutrient content and the total heavy metal decreased. This reduction in may be due to the dilution effect, offered by the bulking agents sawdust as suggested by [8]. The total nitrogen content was found to decreased and the increase in other elements was observed when the sewage sludge is pyrolyzed at a temperature of 400°C. Similar results were obtained by [13]. The decline in the total nitrogen content is due to the massive loss of NO₃-N and NH₄ N during the pyrolysis process. The decomposition of NO₃ and NH₄ salt originally present in the sewage sludge may cause a decline the content of NO₃-N and NH₄-N leading to the decline in the total N content of biochar. The increase in P content of biochar may be due to the fact that P contained in the sewage sludge is mainly composed of thermostable phosphate minerals and these minerals are difficult to decompose. They become more crystallized during the pyrolysis process [14]. K content in the sewage sludge was enriched with the pyrolysis process. This may be due to the enrichment process as suggested by [15]. The heavy metals such as Cd and Cr increased after pyrolysis process. The Cd and Cr content in sewage sludge biochars were essentially higher than those in the raw sewage sludge, this might be due to the less loss of heavy metals during pyrolysis than their weight loss from organic components, which led to the heavy metals' enrichment in the biochar matrix [16].

Table 2. Characterization of sewage sludge, sewage sludge compost and sewage sludge biochar

Parameters	Sewage sludge	Sewage sludge compost	Sewage sludge biochar
pH	5.36	7.07	6.20
EC (dS m ⁻¹)	8.08	5.30	2.25
OC (%)	17.03	13.51	5.28
N (%)	1.68	1.60	0.92
P (%)	7.73	1.24	7.80
K (%)	1.20	0.29	1.60
Cd (mg kg ⁻¹)	8.43	5.41	10.80
Cr (mg kg ⁻¹)	90.74	47	113.20

3.1 Total and available heavy metals

Total cadmium content of soil was significantly influenced by the application of sewchar, sewage sludge compost and sewage sludge treatments and the data are shown in the table 3. Among the treatments the highest total cadmium was observed in different sewchar treatments compared to SSC, SS whereas, Cd was not detected in FYM applied treatment. The total cadmium content in soil ranged between 3.45 (T₇) and 4.81 mg kg⁻¹ (T₅). The highest value of 4.81 mg kg⁻¹ was recorded in T₅ (S + 50 g SC) and the least value of 3.45 mg kg⁻¹ was recorded in T₇ (S + 50 g SS).

Available cadmium content of the soil varied between 83 (T₂) and 178 µg kg⁻¹ (T₇) due to the application of different treatments (Table 2). The highest value of 178 µg kg⁻¹ was recorded in T₇ (S + 50g SS). As the sewage sludge application rate increased from 12.5 g to 50 g an increase in the available and total cadmium content of the soil was observed. Cadmium content was not detected in the T₁ (absolute control) and T₈ (S + 50 g FYM).

Application of different treatments such as sewage sludge, sewage sludge compost and sewage sludge significantly affected the total chromium content of the soil (Table 4). The value varied between 31.11 (T₂) and 35.31 mg kg⁻¹ (T₅). The highest value for total chromium (35.31 mg kg⁻¹) was observed with T₅ on the 90th day and the least value was with T₂ (31.11 mg kg⁻¹) on the 0th day. While considering the different treatments, treatment receiving sewage sludge @ 50 g (T₅) registered the highest value during the entire period of incubation, which was on par with T₄ (S + 37.5 g SC) followed by T₇ (S + 50 g SS), T₆ (S + 50 g SSC), T₃ (S + 25 g SC) and T₂ (S + 12.5 g SC). T₂ registered the least value for total chromium throughout the period of incubation. Total chromium was not detected in T₁ (absolute control) and T₈ (S + 50 g FYM).

The available chromium content varied between 2.24 (T₂) and 7.08 mg kg⁻¹ (T₇) during the entire incubation period. Among the various treatments the highest value was recorded by sewage sludge (T₇), followed by sewage sludge compost (T₆) and sewage sludge (T₅). The least value was observed in T₂ (S + 12.5 g SC) during the entire period of incubation. With respect to different rates of sewage sludge application the chromium content was found to be increased as the rate increased from 12.5 g to 50 g. In treatments T₁ (absolute control) and T₈ (S + 50 g FYM) the chromium content was not detected.

The total heavy metal content in the sewage sludge applied treatment was higher compared to the other treatments. From the table 2 it was clear that total heavy metal content in the biochar was higher than the sewage sludge indicating that the pyrolysis intensified the enrichment. The higher thermo-stability of the heavy metals compared to other sewage sludge compositions may be the cause of this enrichment. A significant amount of the heavy metals in the sewage sludge remained in the biochars due to the fact that the mineral salts and hydroxide generally converted into oxide or sulphides with better thermo-stability during reductive pyrolytic conditions. The heavy metals can also exist in the sewage sludge as various mineral salts (carbonate, sulphate, chlorate, phosphate, etc.), sulphides, hydroxide, oxide and clathrate. Furthermore, because heavy metals have variable boiling and decomposition temperatures, the enrichment degree changed with heavy metals, which may be related to the previously described heavy metal speciation and their corresponding amounts [17].

According to the present study, the order of available heavy metals in the soil was SS > SSC > SC. By adding biochar to soil, the amount of negative charges present on the soil's surface increases, decrease zeta potential and boost cation exchange capacity [17]. This encourages positively charged heavy metals to be attracted to the soil through electrostatic forces. Because its surface contains many functional groups, such as OH and COO, biochar forms compounds with heavy metals that decrease their availability [18]. These elements' bioavailable concentrations in biochars were found to be lower than in sewage sludge, suggesting that the pyrolysis process may inhibit the release of these elements in the DTPA extractant. The low bio-available amounts of these metals in biochars may be explained by the absorption of flourishing pore structure, high BET surface area of biochar on these metals, and the creation of organometallic complex [19].

Table 3. Effect of treatments on total and available cadmium content of soil

Treatments	0 th day		90 th day		180 th day	
	Total Cd (mg kg ⁻¹)	Available Cd (µg kg ⁻¹)	Total Cd (mg kg ⁻¹)	Available Cd (µg kg ⁻¹)	Total Cd (mg kg ⁻¹)	Available Cd (µg kg ⁻¹)

T ₁ - absolute control	ND	ND	ND	ND	ND	ND
T ₂ - 5 kg S + 12.5 g SC	4.43 ^c	83 ^d	4.50 ^b	118 ^f	4.44 ^b	113 ^f
T ₃ - 5 kg S + 25 g SC	4.69 ^b	119 ^c	4.72 ^{ab}	131 ^e	4.70 ^a	118 ^e
T ₄ - 5 kg S + 37.5 g SC	4.73 ^{ab}	122 ^c	4.77 ^a	139 ^d	4.74 ^a	132 ^d
T ₅ - 5 kg S + 50 g SC	4.79 ^a	132 ^b	4.83 ^a	146 ^c	4.81 ^a	141 ^c
T ₆ - 5kg S + 50 g SSC	3.77 ^d	137 ^a	3.85 ^c	169 ^b	3.80 ^c	151 ^b
T ₇ - 5 kg S + 50 g SS	3.45 ^e	141 ^a	3.51 ^d	178 ^a	3.50 ^d	161 ^a
T ₈ - 5 kg S + 50 g FYM	ND	ND	ND	ND	ND	ND
SEm (±)	0.023	1.364	0.078	1.776	0.079	0.993
CD (0.05)	0.069	4.09	0.233	5.323	0.238	2.977

S- soil, SC- sewchar, SSC-sewage sludge compost, SS- sewage sludge, ND- not detectable

Table 4. Effect of treatments on total and available chromium content of soil

Treatments	0 th day		90 th day		180 th day	
	Total Cr (mg kg ⁻¹)	Available Cr (mg kg ⁻¹)	Total Cr (mg kg ⁻¹)	Available Cr (mg kg ⁻¹)	Total Cr (mg kg ⁻¹)	Available Cr (mg kg ⁻¹)
T ₁ - absolute control	ND	ND	ND	ND	ND	ND
T ₂ - 5 kg S + 12.5 g SC	31.11 ^c	2.24 ^e	31.29 ^c	2.51 ^e	31.19 ^c	3.21 ^f
T ₃ - 5 kg S + 25 g SC	31.78 ^{bc}	2.39 ^e	32.04 ^{bc}	2.62 ^e	32.03 ^{bc}	3.29 ^e
T ₄ - 5 kg S + 37.5 g SC	34.64 ^a	3.55 ^d	34.92 ^a	3.82 ^d	34.98 ^a	4.35 ^d
T ₅ - 5 kg S + 50 g SC	35.03 ^a	3.95 ^c	35.31 ^a	4.35 ^c	35.17 ^a	4.55 ^c
T ₆ - 5kg S + 50 g SSC	32.70 ^b	5.25 ^b	32.94 ^b	5.59 ^b	32.85 ^b	6.03 ^b
T ₇ - 5 kg S + 50 g SS	32.88 ^b	6.77 ^a	33.25 ^b	7.08 ^a	32.30 ^{bc}	6.14 ^a
T ₈ - 5 kg S + 50 g FYM	ND	ND	ND	ND	ND	ND
SEm (±)	0.437	0.077	0.526	0.090	0.537	0.019
CD (0.05)	1.31	0.231	1.576	0.270	1.609	0.058

S- soil, SC- sewchar, SSC-sewage sludge compost, SS- sewage sludge, ND- not detectable.

3.2 Heavy metal fractions

The exchangeable, reducible, oxidisable and residual fractions of cadmium were analysed during the incubation period and the results are discussed here (Fig 1-4). Among the different fractions of cadmium the highest amount was found in residual fraction followed by oxidisable, exchangeable and reducible fraction. As the incubation days progressed from 0 to 180 days there was a slight decline in exchangeable and reducible cadmium content and a slight increase in the oxidisable and residual fraction was noticed in all the treatments. Among the different treatments the exchangeable and reducible cadmium content was found to be highest in sewage sludge received treatment and residual and oxidisable cadmium content was found to be highest in sewchar received treatment. Carbonate fraction of cadmium was not detected in any of the treatments. Fractions of cadmium were not detected in absolute control and FYM received treatments.

When comparing the different fractions of chromium, the residual fraction accounts the highest followed by oxidisable and reducible fraction (Fig 5-7). The exchangeable fraction was detected only in sewage sludge compost and sewage sludge applied treatments. Oxidisable, reducible and residual fractions of chromium showed a slight increase as the incubation days progressed from 0 to 180 days. Exchangeable and reducible fraction was found to be the highest in SS where as oxidisable fraction in SSC and residual fraction in 50 g SC.

Generally, there was a slight decline in the unstable fractions of heavy metals such as exchangeable and carbonate fraction of heavy metals during the incubation period. This may be due to the reason that after pyrolysis, the majority of heavy metals were present in residual and oxidizable forms as suggested by [20]. The results of this experiment showed that more immobile speciations formed as a result of the temperature rise during pyrolysis. The oxidisable (F3) and residual (F4) fractions increased significantly during the pyrolysis of sludge to biochar, corresponding well with the change in pH of the biochars with increasing pyrolysis temperature, while the bioavailable category (F1

+ F2) declined significantly. The pH became more alkaline due to metal oxides and mineral residues as the ash content fixed in the biochars during pyrolysis [20].

Three distinct mechanisms exist for the decrease of readily available portions of heavy metals are adsorption, biological transformation and precipitation. According [21] the inorganic components of biochar such as carbonates, phosphates and oxides can cause heavy metals to precipitate. Decrease in the unstable fractions of heavy metals are thought to be caused by the various functional groups, such as carboxyl, phenolic, hydroxyl, etc., on the surface of porous biochar that can adsorb heavy metals by coordination and chelation [22]. According to [23] microbial biochar decomposition may lessen oxidisable heavy metals.

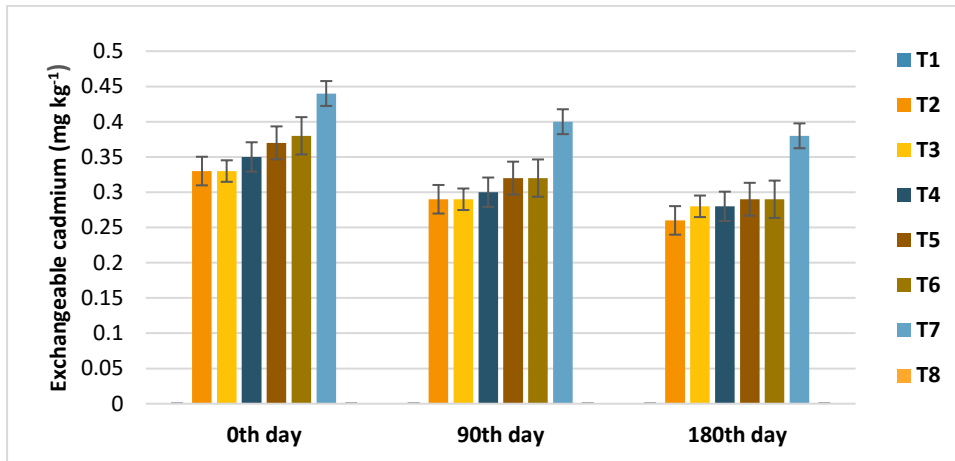


Fig.1 Effect of treatments on exchangeable cadmium of soil

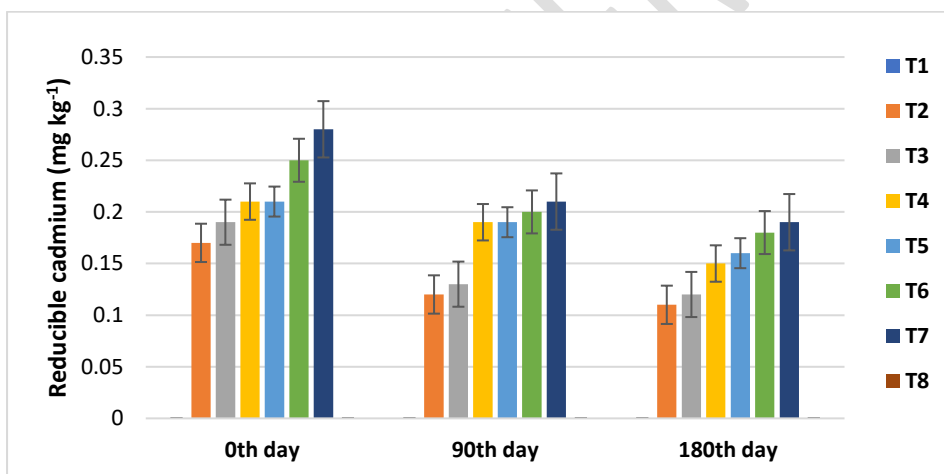


Fig. 2 Effect of treatments on reducible cadmium of soil

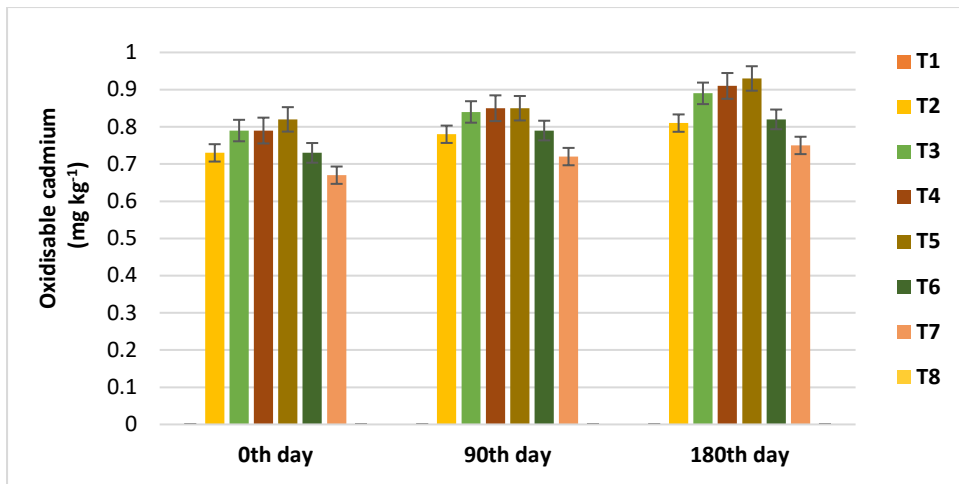


Fig.3 Effect of treatments on oxidizable cadmium of soil

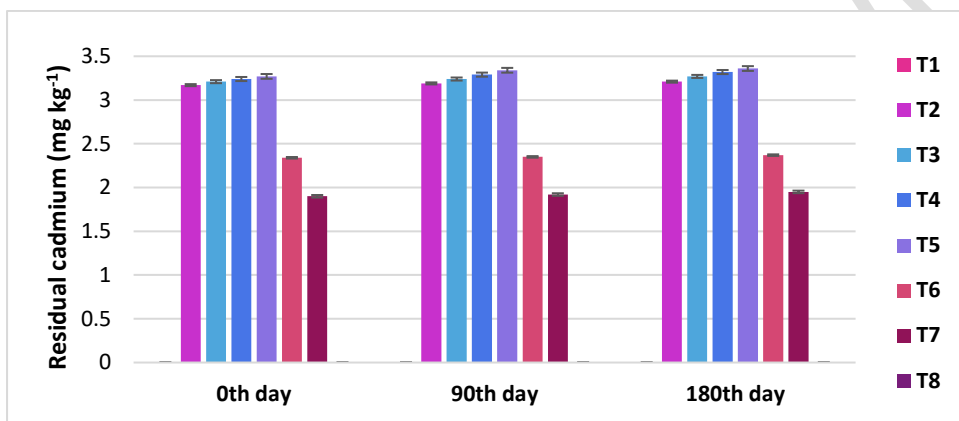


Fig.4 Effect of treatments on residual cadmium of soil

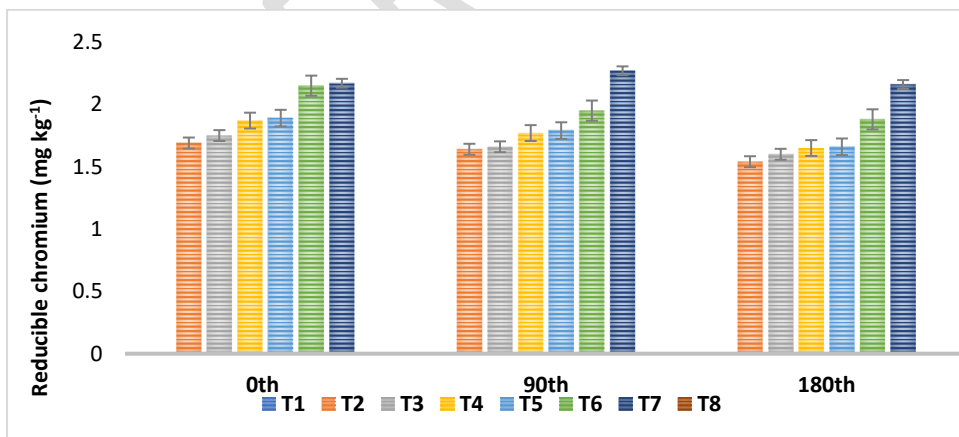


Fig.5 Effect of treatment on reducible chromium of soil

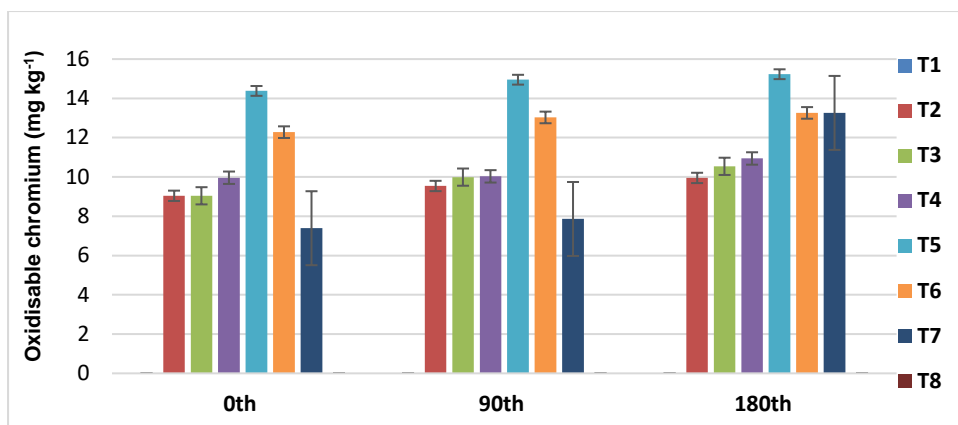


Fig. 6 Effect of treatments on oxidisable chromium of soil

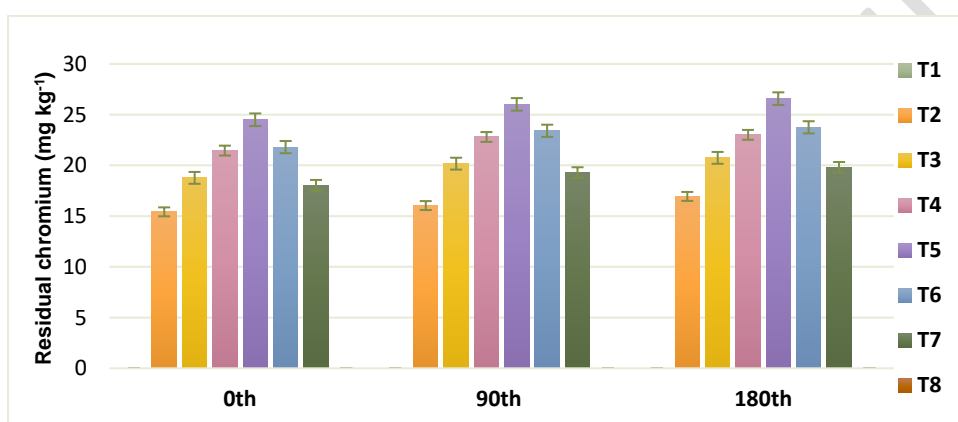


Fig. 7 Effect of treatments on residual chromium

4. CONCLUSION

Based on the results it can be concluded that composting and pyrolysis can effectively immobilize the heavy metals. The conversion of sewage sludge to sewage sludge compost and sewage sludge biochar decreases the available fraction of heavy metals and causes the conversion of unstable fractions of heavy metals to stable fractions. Results from the incubation revealed that even though the enrichment of total heavy metals is happening during the pyrolysis but the availability of heavy metals was lower than the sewage sludge and sewage sludge compost. Pyrolysis process increased the total P and K content and a decline in total N content compared to sewage sludge. Hence the application of sewage sludge biochar is safer than sewage sludge.

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