

**Speciation of trace metals (Cu, Zn, Ni, Fe and Mn) during rotary drum composting of paper mill sludge**Utpal Ghosh¹, Jayeeta Hazarika², Ajay S Kalamdhad¹, Meena Khwairakpam¹ and Jiwan Singh^{3*}¹Department of Civil Engineering, Indian Institute of Technology Guwahati, Guwahati-781039, Assam, INDIA²Department of Civil Engineering, National Institute of Technology, Meghalaya, Bijnai Complex, Laitumkhrah, Shillong-793003, Meghalaya, INDIA³Department of Environmental Science, Babasaheb Bhimrao Ambedkar University, Lucknow-226025, INDIA**ARTICLE INFO****Received: 13 Jan 2018****Revised: 12 Feb 2018****Accepted: 01 Mar 2018****Key words:** PPMS, rotary drum composting, heavy metals, chemical speciation.**ABSTRACT**

Chemical speciation is a very proficient tool for assessing bioavailability of heavy metals. Thus, this study was carried out to detect the variation in distribution of different fractions (Exchangeable, Carbonate, Reducible, Oxidizable and Residual) of Cu, Fe, Ni, Zn and Mn and also to assess the influence of physico-chemical parameters on chemical speciation during rotary drum composting of primary paper mill sludge (PPMS). Residual fraction was established as the most dominant fraction during speciation. Concentration of Ni was found to be mostly associated with residual fraction. However, though concentration of Cu was not found very high but its bioavailability was prominent. The current study reveals that cow dung addition in optimization can influence the physico-chemical parameters, which in turn decreases the bioavailable fraction (exchangeable and carbonate) of heavy metals during composting.

1) INTRODUCTION

The use of paper by a society is often marked as a yardstick of its development. Rapid industrialization is responsible for the boost in paper consumption and global consumption of paper is projected to increase by around 500 million tonnes per annum in 2025 [1]. Accumulative surge of about 77% is expected by the year 2020 [2]. The burgeoning of paper industry results in huge amount of sludge, making sludge management, a serious concern as paper production approximately generates 40-50 kg of dry sludge per tonne product each day [3]. Currently about 69% of total sludge produced goes for landfilling, whereas 21% is incinerated, 8% is landspread and further 8% is divulged to various other alternatives [4]. High organic content of the sludge renders composting as the most suitable recycling option. Composting also removes the short-term phytotoxic effects [5], increases cation exchange capacity and nutrient retention efficiency of soil and kills the potential pathogens [6]. The high heavy metal concentration of PPMS is a serious concern as it can cause severe environmental issues through land application [7]. Though heavy metals are consumed by plants in micro quantities for metabolism, metal concentration exceeding the optimum becomes toxic to plants. Heavy metals present in soil can affect the metabolism, which in turn hinders the growth and morphology of soil microbes and loss of soil fertility as a consequence [8]. Total concentration of heavy metals is a very reliable overall pollution indicator but it cannot provide insights into the bioavailability of metals as it is a self-driven process influenced by explicit combinations of chemical, biological and environmental parameters. The parameter

includes soil properties such as pH, organic matter content, redox potential, cation exchange capacity, sulphate, carbonate, hydroxide, soil texture and clay content. Evaluation of the chemical speciation of heavy metals in composts facilitates information on its bioavailability and hence determines the suitability for land application [9].

Chemical speciation identifies and quantifies different species, forms or phases present in a material [10]. Chemical speciation is therefore, a very reliable technique for determining the chemical forms in which the metals are present [11]. Assessment of compost maturity is also very important from the context of agronomic use of compost. Study of identification and evolution of the chemical, structural composition and functional properties is the best method available till now for assessing compost maturity.

The objective of this work is therefore to determine the total concentration of trace metals (Cu, Fe, Ni, Cr, Zn and Mn) and evaluate the contribution and variation of different fractions through sequential extraction procedure and assess the effect of physico-chemical parameters on metal speciation during composting of PPMS.

2) MATERIALS AND METHODS**2.1 Raw materials collection**

* Corresponding Author: **Dr. Jiwan Singh**

Email address: jiwansingh95@gmail.com

Primary Paper mill sludge (PPMS), cow dung and saw dust were mixed in five different ratios for preparing the feed material for rotary drum composting. PPMS was collected from Nagaon Paper Mill, a unit of Hindustan Paper Corporation situated at Nagaon in the state of Assam, India at a distance of around 70 km from Indian Institute of Technology, Guwahati. For treating the wastewater, the mill is using a combination of clariflocculator and aerated lagoon for primary treatment and secondary treatment respectively. The sludge produced from clariflocculator is dewatered using filter press and disposed. The dewatered sludge from filter press was collected for composting instead of mere disposal. However, saw dust was brought from saw mill whereas cow dung was collected from dairy farms nearby the campus and accordingly all the three constituent materials were characterised as per Table 1. Lumps of PPMS were broken to apparently 1 cm particle size to facilitate better aeration. The proportions of raw materials (in kg) is given as follows: Trial 1 (T1): PPMS-150, cow dung-0, sawdust-0; Trial 2 (T2): PPMS-120, cow dung-15, sawdust-15; Trial 3 (T3): PPMS-105, cow dung-30, sawdust-15; Trial 4 (T4): PPMS-90, cow dung-45, sawdust-15; Trial 5 (T5): PPMS-75, cow dung-60, sawdust-15. The constituent materials are therefore uniformly mixed and afterwards fed into the reactor.

PPMS composting was performed in a rotary drum composter of 500 L capacity as described by [12]. Proper degradation and stabilization of PPMS was the key parameter in fixing the duration of composting as 20 days. Grab sampling method was followed for collecting samples from various positions i.e. top, bottom and middle layer of the drum. Samples were collected in triplicates at interval of every 4 days till the end of composting process. Collected samples were then mixed to get a homogenized mixture and further milled and sieved through 0.2 mm sieve after drying for 24 h at 105°C.

2.2 Physico-chemical analysis and metal fractionation

Aqueous extract of the sample was used to measure pH and moisture content was determined by drying the sample at 105°C for 24 h. Sequential extraction of the heavy metals in the compost samples has been applied according to the method described [13]. Five fractions were obtained: (i) Exchangeable fraction (F1) (ii) Carbonate fraction (F2) (iii) Reducible fraction (F3) (iv) Organically bound fraction (F4) and (v) Residual fraction (F5). The sequential extraction was performed with oven pre-dried sample of 1 g in centrifuge tubes to minimize losses. In every step of extraction, the samples were centrifuged at 10000 rpm for 5 min and then the supernatant liquid was separated with a pipette and made up to the required volume and stored for further analysis in AAS. The pellet was washed with 20 mL of Milli Q water through shaking for 15 min followed by centrifugation. All the extractions were performed in triplicates and the mean value has been presented with standard deviation. F1 was extracted with 8 mL of 1 M MgCl₂ (pH 7) after continuous agitation for 1 h at room temperature. F2 was leached out from the residue of previous step with 8 mL of NaOAc (pH adjusted to 5 with conc. HoAc) after continuous agitation of 5 h at room temperature. 20 mL of 0.04 M NH₂OH HCl in 25% (v/v) HOAc was added to the residue of the above step and agitated for 6 h at 96°C to extract F3. To extract F4, 3 mL of 0.02 M HNO₃ and 5 mL of 30% H₂O₂ (pH 2, adjusted with conc. HNO₃) was added to the residue from F3 step and heated at 85°C for 2 h with occasional agitation, after that a second 3 mL aliquot of 30% H₂O₂ was added and heated at 85°C for 3 h with occasional agitation. After cooling, 5 mL of 3.2 M

NH₄OAc in 20% (v/v) HNO₃ was added; diluted to 20 mL; agitated for 30 min and centrifuged. 10 mL of H₂SO₄ and HClO₄ (5:1) mixture was added to the residue from F4 step and it was digested in a Block Digester (Pelican Equipments, Chennai India) at 300°C for 2 h to extract F5 fraction.

According to the extraction procedure, total concentration of F1, F2, F3 and F4 represents the concentration of mobile fraction [14]. A ratio (Bioavailable Fraction) was calculated from the ratio of mobile concentration to total concentration to assess the bioavailability of heavy metals.

$$\text{Bioavailable Fraction (BF)} = \frac{F1+F2+F3+F4}{F1+F2+F3+F4+F5}$$

3) RESULTS AND DISCUSSION

3.1 Physico-Chemical Parameters

The temperature profile throughout rotary drum composting PPMS is shown in Fig. 1(a). Trials 4 and 5 with maximum amount of cow dung, showed rapid increase in temperature due to efficient microbial activity, whereas trials with comparatively less cattle manure attained maximum temperature at a later stage during composting. A maximum temperature of 44.8°C was recorded for trial 4, however absence of microbial activity in trial 1 prevented rise in temperature. It is believed that a temperature between 52 to 60°C is enough to continue thermophilic activity [15]. [16] reported a maximum temperature of 61.4°C in rotary drum composting of vegetable waste. Thus, the maximum temperature in PPMS composting depicts that the material is resistant to degradation but the initial sharp increase in temperature can be credited to optimum addition of cattle manure which propagated high microbial activity. The prolonged consistent temperature from middle to final days depicts that the effect of the cattle manure is minimized, but biodegradable organic was still present in the sludge which is being used by the microorganisms at a slower rate. This shows that the material is hard to degrade due to presence of lignocellulosic materials.

pH is one of the important environmental factors influencing composting. The optimum pH for the sustainability and the degradation efficiency of the microorganisms is in the range of 7-8 [17]. A maximum increase in the pH profile from 7.6 to 8.1 was observed in trial 4 during composting, however trial 1 depicted no change in pH as shown in Fig. 1(b). During the composting process, regular turning was ensured to provide sufficient aeration and therefore leading to release of CO₂, as a result of which increase in pH was observed and further the development of anaerobic condition was prevented [18].

Variation in moisture content depicted moisture loss in all the trials, however a maximum of 23.2% was observed in trial 4 whereas minimum was 3.4% in trial 1 as shown in Fig. 1(c), which represents maximum degradation of organic matter in trial 4. During composting, degradation can be evaluated through indirect measure by estimating loss in moisture resulting due to the heat generated during the process [19]. Maintaining an optimal moisture content during composting is important, insufficient moisture content will hinder the biological process and produce a physically stable but biologically unstable compost, whereas if the moisture content is high it will inhibit the aeration process by clogging the pore [20].

Organic matter (OM) degradation and conversion to humic substances during composting, has innate relationship with

availability of metal ions. Metal ions form complexes with the humic substance, which can buffer the pH and will serve as a source of nutrients for plants [21]. The scenario of reduction of OM is presented in Fig. 1d, wherein OM was reduced in all the trials. In trial 4, a maximum reduction of 16.8% was observed, while minimum reduction of 1.9% of OM was observed in trial 1. Higher OM loss in trial 4 was due to higher microbial activity during composting. [7] has reported a decrease of 32.6% in OM during rotary drum composting of water hyacinth. The less OM reduction in PPMS composting can be attributed to presence of lignocellulosic matters.

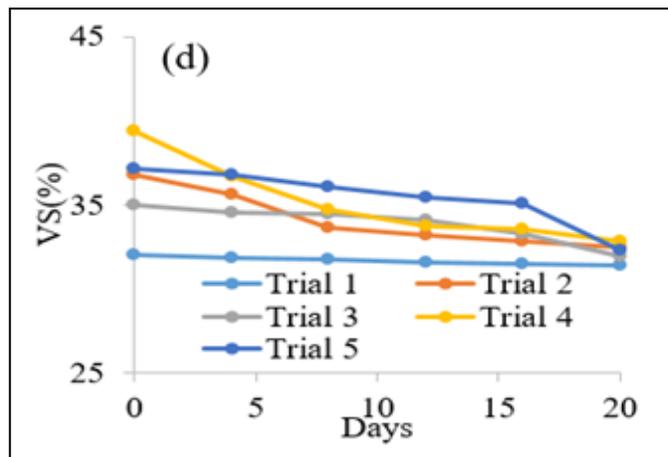
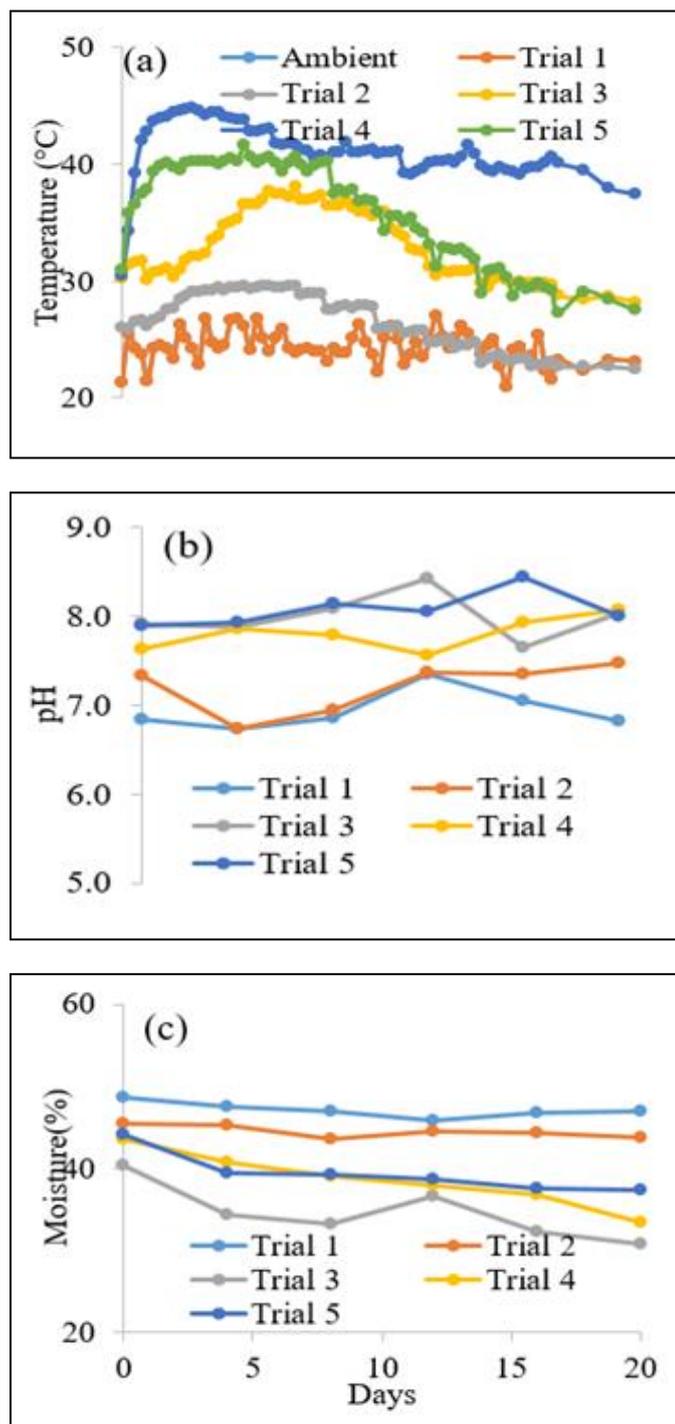


Fig. 1 (a,b,c,d) Variation in physico-chemical parameters during rotary drum composting

3.2 Speciation of Heavy Metals

3.2.1 Copper

Copper was mainly bound to oxidizable and residual fraction in the all the trials. This is in accordance with [10] and [22]. The variation of different fractions has been detailed in Table 2. F1 in trial 1 and 3 was found to increase slightly, while it decreased in trial 2 and 5. F1 was not detected in trial 4. F2 increased in all the trials except trial 5, where it decreased from 15.5 to 11.1%. The F3 fraction decreased in trial 1 and 3, on the other hand it increased in trial 2 and 4 and was not detected in trial 5. F4 increased in trial 3, in rest of the trials it got reduced. Maximum decrease was observed in trial 5. F5 remained almost constant in trial 4, whereas it increased in the remaining trials. [23] has also reported same trend during composting of water hyacinth. An increase in F5 was also reported by [25]. [14] also observed an increase in F1, F2 and F3 fraction during composting of sewage sludge. The oxidizable fraction converted to residual fraction in trial 1 and 5. The BF for Cu decreased in trial 1 and 5 from 0.76 and 0.49 to 0.65 and 0.32 respectively. In all other trials it was unchanged during composting. The reduction bioavailability is due to formation of Cu ion complexes with two or more organic functional groups mainly carboxylic, carbonyl and phenolic, so that the ion is immobilized in a rigid inner-sphere complex [23].

3.2.2 Iron

The distribution of different fractions of Fe has been detailed in Fig. 2. F3 and F5 fractions were predominant in PPMS compost. F1, F3 and F5 fraction decreased in trial 1, F2 remained unchanged and F5 got increased. F1 fraction was found to be increasing in trial 2, 3, 4 and 5. F2 in trial 5 and F4 in trial 3 stayed unchanged during composting. F2 increased in trial 2 and 3, whereas a decrease was observed in trial 4. In trial 1 and 5, F5 got increased but the value of F4 got decreased. F5 fraction was decreased in trial 2, 3 and 4. An increase in F5 fraction was also observed by [23]. The BF decreased in trial 1 and 5 from 0.52 and 0.47 to 0.48 and 0.43 respectively, but it increased in trial 2, 3 and 4 from 0.44, 0.47 and 0.49 to 0.46, 0.48 and 0.51. The decrease in BF can be attributed to the formation of Fe complex with humic substances [23].

3.2.3 Nickel

Nickel was mainly associated with residual fraction. The different fractions during speciation are shown in Table 3. F1, F2 and F3 were found to increase in trial 5 from 9.7, 3.4, 13.7

to 10.5, 4.7 and 15.1% respectively. F4 was detected in trial 3 which decreased from 8.4 to 7.6%. The total concentration was associated with F5 in trial 1, 2 and 4, while in trial 3 an increase was observed. In trial 5, F5 fraction decreased from 76.4 to 73.3%. [14] reported an increase in F1, F2 and F3 fraction during composting of sewage sludge. [23] also observed a high percentage of Ni to be associated. Due to absence of F1 and F2 fractions, there is a less possibility of environmental toxicity of Ni. BF was not observed in trial 1, 2 and 4, in trial 3 it decreased from 0.08 to 0.07. In trial 5, BF increased from 0.26 to 0.3. From these results it can be said that excessive amount of cow dung is making the Ni more mobile.

3.2.4 Zinc

The sharing of different fractions of Zn during rotary drum composting is shown in Fig.3. Though the total concentration of Zn was high but F1 fraction was low in all the trials. Residual fraction dominated in all the trials. Not much change was observed in F1 fraction in all the trials except trial 5 where it reduced from 9.6 to 6.4%. [23] also observed a reduction in F1 fraction during water hyacinth composting. This decrease is may be due to formation of Zn complexes with organic matter in the course of composting. F2 and F3 decreases in trial 1, 2 and 3 but got increased in trial 5. A consistent increase was observed for F4 in all the trials, whereas F5 fraction reduced in trial 2, 4 and 5. In trial 1 and 3, F5 increased from 49.2 and 51.8 to 51.8 and 54.4% respectively. Decrease in F5 fraction was observed as F5 converted to F4 during composting. [26] has reported the transformation of F5 to F4 during sewage sludge composting. Very little variation in BF was observed in all the trials for Zn. But the high BF can cause phytotoxicity [23].

Table. 1 Initial characterization of the waste materials

Parameter	Waste Material (Kg)		
	PPMS	Cattle Manure	Saw Dust
pH	7.3±0.1	6.8±0.1	6.1±0.1
Moisture Content (%)	46±0.8	86.4±0.9	9.6±0.5
Volatile solids (%)	40.2±0.5	72.1±0.2	79.3±0.2
Nutrients (g/kg)			
Na	2.94±0.43	1.08±0.01	2.5±0.05
K	0.46±0.04	0.7±0.01	0.96±0.02
Ca	31.56±8.914	2.620±0.02	9.89±0.03
Total heavy metal concentration(mg/kg)			
Cd	1600.5±161.5	59.95±0.5	52.63±1.2
Cu	43.67±1.53	29.5±0.7	44.8±0.5
Fe	3666.67±813.9	2735.3±11.3	1860.8±2.8
Ni	89.5±5.6	267.5±1.5	239.9±2.1
Pb	320±25.9	1000±5	847.55±5.5
Cr	220.8±35.8	93.27±0.5	121.45±0.5
Zn	23125.9±1071	124.45±2.25	188.4±1.95
Hg	1883.3±125.8	143.45±2.5	195±2.45
Mn	378.67±14.98	173±3.5	532.5±1.5

3.2.5 Manganese

During rotary drum composting of PPMS, F3 and F5 fractions were predominant. The distribution of different fractions has been detailed in Fig.4. Concentration of F1 fraction was high in trial 4 comparing to other trials. In trial 2 and 5, initially F1 was detected but rapidly reduced during the process to become not detectable. The reduction in F1 fraction is due to the increase in pH value during the composting process. In trial 3, F1 concentration didn't changed much, but in trial 4 it was found to increase. F1 and F2 were not observed in trial 1. In trial 3 and 4, a very little decrease in F2 was seen while in trial 5 it was not observed. F3 concentration increased from 46.9 to 55.6% in trial 1, in all other trials it remained unchanged. In trial 1, it seems that the F5 fraction is being converted to F3. F4 fraction was not detected in trial 2 and 5, while in trial 1, F4 concentration was high and it was observed to decrease from 22.6 to 18.6% during the course of composting. A decrease was observed in F5 fraction in trial 1 and 4, while increase in concentration was observed in rest trials with maximum increase in trial 5, from 44.4 to 54.6% in the course of composting. Similar result was also observed by [23] during water hyacinth composting. BF of Mn increased in trial 1 and 4 from 0.7 and 0.78 to 0.74 and 0.8 respectively, whereas it decreased in trial 2, 3 and 5 from 0.43, 0.63 and 0.56 to 0.33, 0.58 and 0.45 respectively. This high bioavailability can cause phytotoxicity. This rise in pH can decrease Mn mobility by precipitate formation, thereby increasing the number of adsorption sites and decreasing the competition of H⁺ for adsorption, followed by increase in metal stability with humic substances [23].

4) CONCLUSION

Chemical speciation is very beneficial in assessing bioavailability of heavy metals during rotary drum composting of PPMS. F5 was the predominant fraction for all the heavy metal and increased during PPMS composting, except for Mn, where most of the concentrations were bound to F3 fraction. F1 and F2 fraction was only detected for Ni in trial 5. It seems that addition of cow dung in excess quantity is converting these metals to mobile fraction. Bioavailable fraction was quite high for Cu and Zn. A reduction in BF was observed during composting in all the trials. It can be concluded that cow dung addition in optimum quantity and change in physico-chemical parameters is influential on the distribution of various fractions of heavy metals during rotary drum composting of PPMS.

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Table. 2 Variation of different fraction of Cu during composting

Days	Trial 1					Trial 2				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	3.86±0.06	4.97±0.11	3.69±0.16	12.07±0.08	7.64±0.36	3.39±0.24	4.22±0.21	2.27±0.08	10.39±0.21	11.44±0.12
4	3.67±0.29	4.67±0.28	3.27±0.08	9.4±0.15	8.9±0.66	3.15±0.19	4.25±0.2	2.97±0.21	11.99±0.16	14.14±0.16
8	3.09±0.23	5.09±0.17	3.5±0.05	7.45±0.22	8.54±0.24	2.84±0.19	4.5±0.05	2.24±0.08	10.5±0.05	10.97±0.16
12	2.94±0.12	5.49±0.16	3.2±0.09	11.09±0.19	8.6±0.27	2.04±0.06	4.75±0.18	2.65±0.14	11.47±0.15	11.8±0.37
16	3.44±0.13	5.35±0.15	3.12±0.08	8.7±0.11	8.64±0.12	3.55±0.23	4.57±0.03	2.62±0.21	9.17±0.03	10.54±0.42
20	3.89±0.11	5.59±0.18	2.97±0.24	8.22±0.06	10.84±0.56	2.22±0.03	5.2±0.33	2.44±0.11	9.85±0.15	11.8±0.63

Days	Trial 3					Trial 4				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	3.85±0.44	5.04±0.23	6.09±0.71	6.1±0.66	10.2±1.58	0±0	2.49±0.31	3.84±0.23	15.42±0.21	21.44±0.82
4	3.5±0.5	4.47±0.39	6.04±0.31	11.69±0.66	10.84±0.31	0±0	2.89±0.26	4.14±0.44	13.7±0.33	17.64±2.62
8	4.17±0.48	4.8±0.66	5.2±0.4	13.24±0.47	9.84±0.71	0.27±0.08	2.44±0.66	3.47±0.71	15.49±0.26	13.9±0.61
12	4.3±0.15	4.35±0.4	4.99±0.72	15.72±0.62	12±0.92	1.04±0.19	2.8±0.96	3.75±0.71	16.3±0.56	15.4±0.56
16	3.95±0.36	6.1±0.71	4.77±0.21	6.09±1.21	9.1±0.53	0.98±0.54	4.1±0.35	4.34±0.61	14.14±0.43	22.54±0.97
20	5.28±1.74	5.82±0.44	5.39±0.36	7.22±0.56	12.04±2.45	0±0	4.04±0.32	4.5±0.37	15.94±0.21	23.7±0.73

Days	Trial 5				
	F1	F2	F3	F4	F5
0	1.52±0.25	3.04±0.21	ND	5.12±0.15	9.9±0.35
4	0.79±0.12	3.62±0.38	ND	5.8±0.14	10.5±0.2
8	1.3±0.14	2.72±0.18	ND	6.34±0.26	5.07±0.16
12	1.02±0.21	3.14±0.21	ND	2.84±0.13	8.17±0.06
16	0.74±0.08	2.64±0.29	ND	3.62±0.36	8.14±0.06
20	0.94±0.13	2.19±0.11	ND	3.25±0.14	13.24±0.73

(ND= not detected)

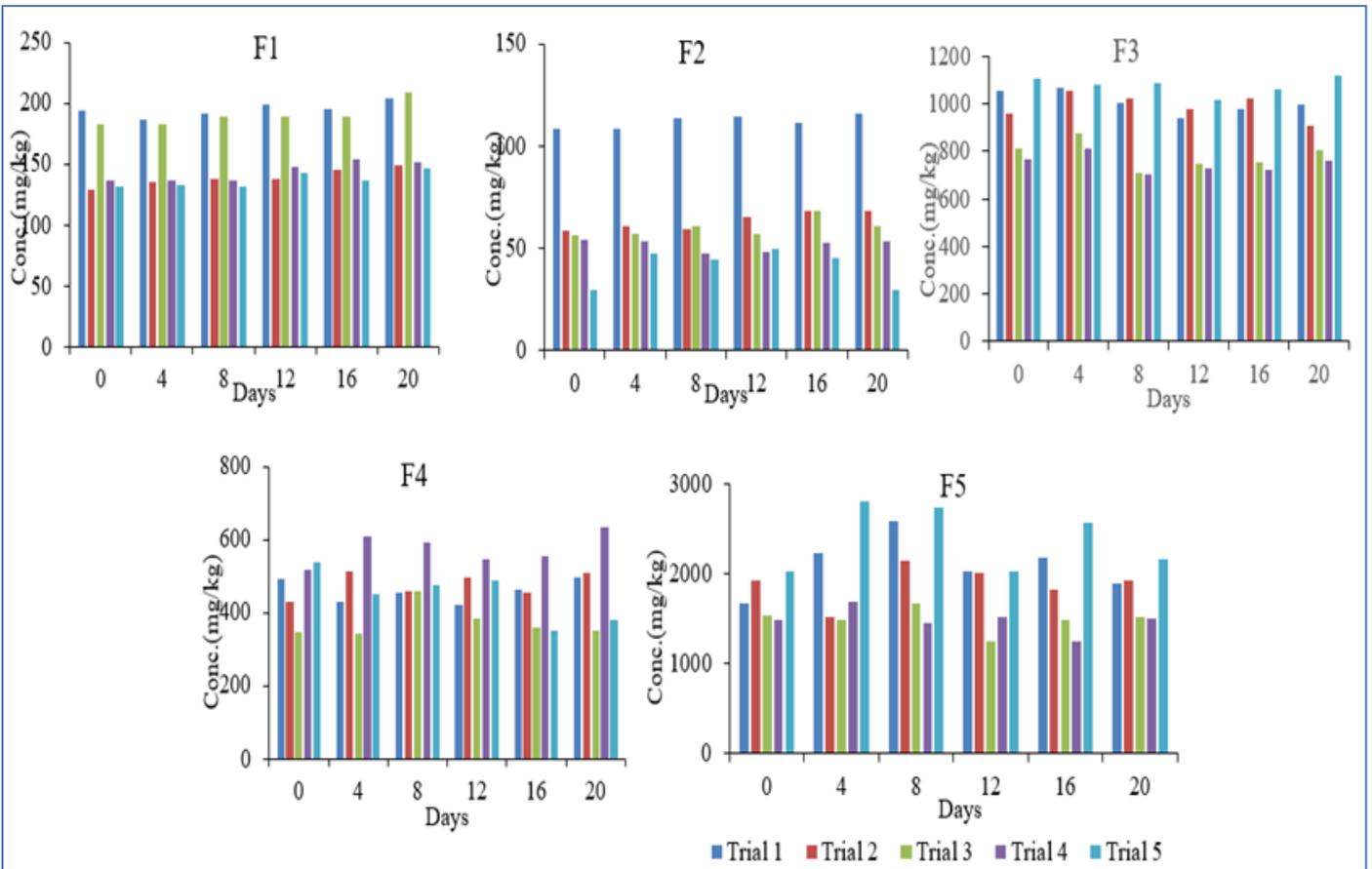


Fig. 2 Speciation of Fe in trial 1, 2, 3, 4 and 5 during the composting process.

Table. 3 Variation of different fraction of Ni during composting

Days	Trial 1					Trial 2				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	ND	ND	ND	ND	51.24±1.78	ND	ND	ND	ND	16.4±1.66
4	ND	ND	ND	ND	58.4±1.82	ND	ND	ND	ND	14.04±0.67
8	ND	ND	ND	ND	61.44±1.66	ND	ND	ND	ND	21.64±2.81
12	ND	ND	ND	ND	58.34±2.03	ND	ND	ND	ND	21±2.89
16	ND	ND	ND	ND	64.87±4.79	ND	ND	ND	ND	15.24±2.12
20	ND	ND	ND	ND	71.74±0.98	ND	ND	ND	ND	19.7±0.44

Days	Trial 3					Trial 4				
	F1	F2	F3	F4	F5	F1	F2	F3	F4	F5
0	ND	ND	ND	7.24±0.21	78.57±2.05	ND	ND	ND	ND	82±7.01
4	ND	ND	ND	9.72±1.29	74.1±2.49	ND	ND	ND	ND	73.2±1.38
8	ND	ND	ND	5.3±1.71	86.34±4.9	ND	ND	ND	ND	76.67±4.46
12	ND	ND	ND	10.2±1.23	72.17±1.16	ND	ND	ND	ND	83.2±5.6
16	ND	ND	ND	9.49±0.38	74.44±1.82	ND	ND	ND	ND	84.2±2.26
20	ND	ND	ND	6.59±0.28	79.2±0.9	ND	ND	ND	ND	88.8±1.67

Days	Trial 5				
	F1	F2	F3	F4	F5
0	5.4±0.01	1.9±0.91	5.8±0.38	ND	42.3±1.06
4	4.95±0.31	5.79±0.39	6.09±0.6	ND	42.07±0.91
8	5.59±0.48	3.92±0.51	5.42±0.66	ND	45.8±0.27
12	5.45±0.14	6.82±0.08	6.59±0.12	ND	42.87±1.98
16	4.42±0.56	4.14±0.25	5.79±0.44	ND	37.34±1.39
20	6.09±0.35	2.72±1.22	6.49±0.62	ND	42.9±1.65

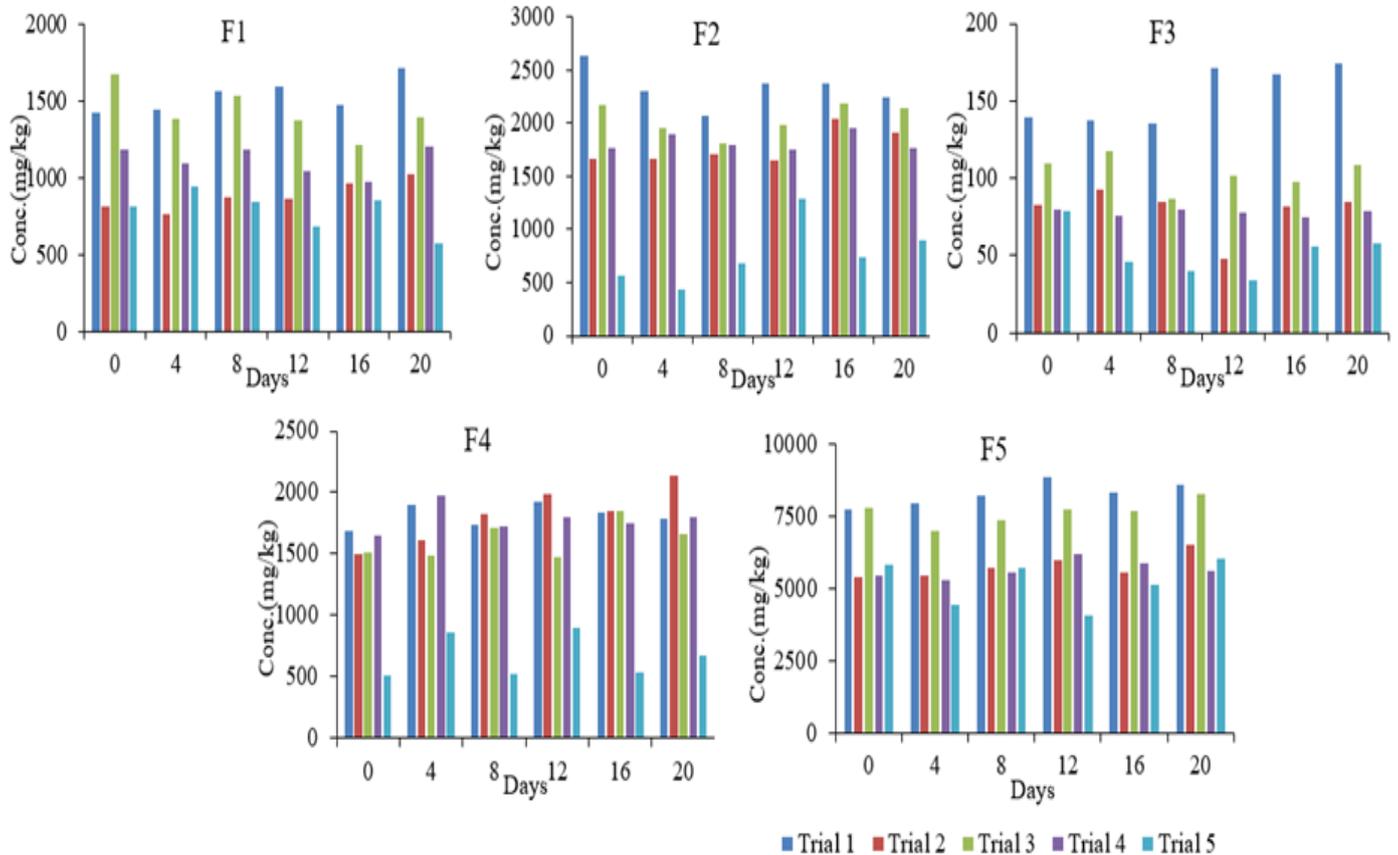


Fig. 3 Speciation of Zn in trial 1, 2, 3, 4 and 5 during the composting process.

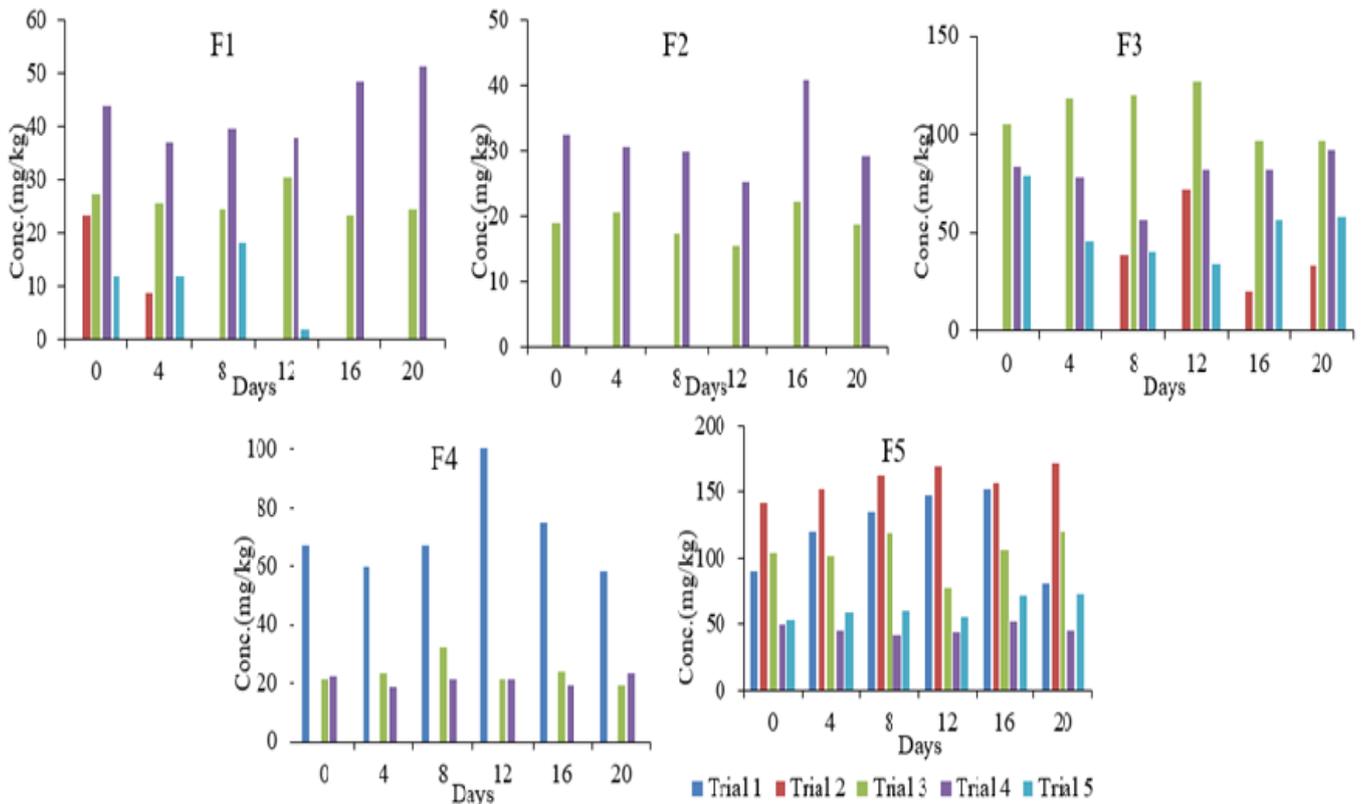


Fig. 4 Speciation of Mn in trial 1, 2, 3, 4 and 5 during the composting process.

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