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Influence of Decreasing Supplementation to Transformation of Chemical Forms of Ni, Zn and Cu During Composting of Sewage Sludge

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Abstract: This paper pertains to the influence of decreasing supplementation to transformation of chemical forms of Ni, Zn and Cu during composting of sewage sludge. A universally used supplementing material constituting a rich source of organic carbon is straw. Addition of straw to sewage sludge is aimed at increasing C/N proportion up to at least 15 due to the risk of formation of toxic forms of nitrogen, concentration of which in sewage sludge is exceptionally high. We have presented in this paper the results of speciation research of three elements applying Tessier's sequential extraction. It's been proven that decreasing of straw share in the composted mixture with sewage sludge down to the level of C/N value below the admissible value, has a beneficial effect on the allocation of tested heavy metals towards the forms that are permanently bound in compost matrix. A systematic increase of organic (IV) and residual (V) fractions share and decrease of mobile forms of heavy metals content in bioavailable fractions i.e. ion-exchange (I) and carbonate (II) has been ascertained.

Keywords: sewage, composting, heavy metals, speciation

1. Introduction

Composting of sewage sludge originating from municipal sewage treatment, is a universally applied method of biological transformation of biodegradable waste that assures obtaining of highly fertile material i.e. compost (Curtis & Claassen 2009, Carrizo et al. 2015). Due to high content of macroelements, mainly organic carbon, nitrogen and phosphorus, mechanically dehydrated sewage sludge makes a valuable raw material for production of compost, which complies with the requirements set for soil improvers and materials substituting soil in growing media (Pinasseau et al. 2018, Regulation EU 2019). High concentration of total nitrogen in mechanically dehydrated sewage sludge generally falling into 2-7% DM (dry matter) interval (Sidełko et al. 2010, Świerczek et al. 2018) and its high humidity amounting to 85-75% (Kacprzak et al. 2017) causes that composting of sewage sludge requires use, at the compost mass formation stage, of a supplement featuring high concentration of organic carbon and low nitrogen content (Hamoda et al. 1998, Zhang et al. 2010, Doublet et al. 2010). The optimum C/N ratio at the compost formation commencement point should fall, according to various sources, within 20-30 interval (Sidełko et al. 2011, 2017) whereas value of this parameter in dehydrated sewage sludge generally does not exceed 7 (Kacprzak et al. 2017). The required C/N ratio originates, among other things, from a hazard of generation of nitrogen toxic forms – NH_3, in conditions of increased organic nitrogen concentration in sewage sludge (Gonzalez et al. 2019).

Supplementation, being an essential operation for sewage sludge composting, was a theme of many studies associated with use of various organic materials i.e. sewage sludge with green waste originating from park and garden upkeep activities, where the proportion of both components was 1:1.2 w/w (C/N = 28.9) (Gonzalez et al. 2019), sewage sludge with wood shavings and mature compost in proportion of 1:0.5:0.17 w/w (C/N = 19.3) (Zheng et al. 2018), sewage sludge with maize straw 1:1.9 w/w (C/N = 30) (Głąb et al. 2018) and sewage sludge with maize stark – 5.7% moisture content 1:0.17 w/w (C/N = 18.2) (Li et al. 2017). Research works on sewage sludge composting with supplementation below C/N = 15 proportion. due to a possibility of generation NH_3, having unfavourable impact on the kinetics of organic matter decomposition, are, however, rare. Test results for compost samples performed in laboratory conditions using a reactor of approximately 1.3 m^3 volume and proportion between dehydrated sewage sludge and barley straw being 1:0.3 respectively (C/N = 15) obtained by Kulikowska & Sindrewicz (2018), did not confirm any unfavourable impact of the increased volume of sewage sludge in composted mass on the process course. Also results of our own earlier research work on composting of sewage sludge using a dynamic reactor in industrial conditions, taking into account observed compost temperatures and timing of its maintenance as well as the change of organic substance content and C/N parameter, have indicated correct course of composting at relatively low C/N value amounting to 10 (Sidełko et al. 2020).

The research work initiated in 2018 pertained to evaluation of composting process in conditions of increased concentration of sewage sludge in its mixture with straw and comprised an analysis of change of selected physico-chemical indicators values, including also heavy metals. Total heavy metals content in compost qualified as soil improver makes, in accordance with applicable regulations, a criterion admitting compost for use in agriculture (Regulation EU 2019). The Regulation of the European Parliament and of the Council (EU) 2019/1009 of 5 June 2019 strictly defines the admissible levels of total Ni, Hg, Cr (VI), Pb and As (inorg) in organic fertiliser content. However, the recommended total copper content must not exceed 300 mg/kg DM and zinc 800 mg/kg DM.

The total heavy metals content does not allow to asses the level of hazards that the natural environment would be exposed to, which may constitute the application of composted sewage sludge to the soil. In order to determine bioavailability. mobility and reactivity of trace elements, analytical procedures based on sequential extraction are used (Janowska & Szymański 2009, Zhu 2014). The most frequently applied method is the sequential extraction developed by Tessier et al. (Zhu 2014). It consists in separation of five fractions i.e. ion-exchange, associated with carbonates and Fe/Mn oxides, with organic substance and residual fraction. It's been proved that metals in form of ion-exchange and water-soluble compounds can migrate and be accumulated in plant tissues (Zhu 2014).

The objective of our research work was to determine Ni, Zn and Cu amounts bound in specific chemical forms, also in form of complexes that are hardly available for microorganisms. It is considered that bonding of heavy metals in the solid phase during composting e.g. in humic compounds structure and argillaceous minerals compared to other chemical forms is exceptionally strong (Szymański et al. 2005). Thus the risk of liberation of heavy metals to the ground and their bioaccumulation in cultivated plants is reduced.

2. Materials and Methods

Field research was performed at Goleniów wastewater treatment plant withing a project financed under EU South Baltic programme (STEP 2018).

Before modification of the composting technique. which took place in October 2020, processing of sewage sludge originating from municipal wastewater treatment consisted in its mechanical dehydration and then composting with straw, wood chips and mature compost (inoculum) added in mass proportion of – 4:1:0.5:0.5. Composting was performed in roofed windrows 70 m in length and trapezoid cross-section dimensions being 3m – bottom base width and 1.5 m – the height. The windrows were periodically mechanically overturned at a rate of two overturns per week during the first three weeks of composting and, in the subsequent weeks, once per week on average. Composting operation comprising an intense phase and maturing lasted 4-5 months depending on the external conditions.

Two windrows differing in proportion of particular components being respectively: windrow no 1 (series 1) – 4:1:1 and windrow no 2 (series 2) – 8:1:2 (sewage sludge : straw : wood chips and inoculum) were earmarked for the research. From each windrow compost samples weighing approximately 1 kg were taken; apart from the selected physico-chemical indicators heavy metals were determined by application of a flame method using AAS atomic absorption spectrometer. The analysis of heavy metals fractions based on the multistage sequential extraction was performed using the Tessier's method (Table 1).

Table 1. Analytical procedure

Step	Fraction	Extractant	Extraction conditions	
			temperature	time
I	FR I exchangeable	10 cm^3 1 M CH_3COONH_4 pH = 7	20°C	1h
II	FR II carbonate	20 cm^3 1M CH_3COONa. pH = 5	20°C	5h
III	FR III bound with Mn and Fe oxides	20 cm^3 0.04 M $NH_2OH{\cdot}HCl$ w 25% (v/v) CH_3COOH	95°C	5h
IV	FR IV organics and sulphides	a) 5 cm^3 0.02 M HNO_3 + 5 cm^3 30% H_2O_2 . pH = 2 b) 5 cm^3 30% H_2O_2 . pH = 2 c) 10 cm^3 3.2M CH_3COONH_4 w 20% (v/v) HNO_3	a) 85°C b) 85°C c) 20°C	2h 3h 0.5h
V	FR V residue	5 cm^3 65% HNO_3 + 1 cm^3 30% H_2O_2 + 1 cm^3 75% $HClO_4$	Microwave mineralization	

*Szymański et al. 2005

3. Results and discussion

Generally, total concentrations of tested heavy metals increased in both series during the composting (Table 2-4).

Table 2. Average Ni content in fractions

Composting time day	Average Ni content in fractions. [mg/kg d.m]					
	FRI	FRII	FRIII	FRIV	FRV	sum
Windrow no. 1 – 4:1:1 (sewage sludge/straw/wood chips and innoculum)						
3	0.83	0.38	0.50	2.50	9.30	13.51
10	0.30	0.50	0.38	3.13	13.95	18.26
16	0.30	0.25	0.38	3.00	12.33	16.26
24	0.30	0.25	0.25	3.25	12.45	16.50
29	0.23	0.25	0.25	3.13	18.62	22.48
48	0.23	0.25	0.13	3.88	19.78	24.27
62	0.15	0.25	0.00	3.88	9.80	14.08
83	0.23	0.50	0.00	4.38	10.90	16.01
111	0.15	0.50	0.00	4.50	10.10	15.25
133	0.15	0.50	0.00	5.13	16.23	22.01
Windrow no. 2 – 8:1:2 (sewage sludge/straw/wood chips and innoculum)						
3	0.68	0.38	0.88	2.38	8.45	12.77
10	0.15	0.25	0.50	3.38	8.73	13.01
16	0.30	0.00	0.50	3.00	9.95	13.75
24	0.15	0.13	0.50	3.13	15.35	19.26
29	0.15	0.00	0.25	3.38	16.73	20.51
48	0.23	0.25	0.00	3.25	4.73	8.46
62	0.15	0.00	0.00	3.50	12.85	16.50
83	0.15	0.25	0.00	3.13	9.73	13.26
111	0.15	0.25	0.00	3.25	12.10	15.75
133	0.15	0.13	0.00	3.38	13.35	17.01
Sewage sludge	2.03	2.63	1.25	1.50	3.10	10.51

Table 3. Average Zn content in fractions

Composting time day	Average Zn content in fractions. [mg/kg d.m]					
	FRI	FRII	FRIII	FRIV	FRV	sum
Windrow no. 1 – 4:1:1 (sewage sludge/straw/wood chips and innoculum)						
3	14.25	124.88	162.88	90.75	48.75	441.51
10	14.93	117.00	178.50	94.38	70.45	475.26
16	16.65	115.63	183.88	97.13	69.48	482.77
24	19.43	112.38	183.63	110.50	58.58	484.52
29	14.10	109.38	205.38	106.38	74.09	509.33
48	11.93	92.13	213.25	136.50	78.70	532.51
62	9.98	82.75	208.75	167.75	79.48	548.71
83	10.80	69.25	198.63	170.00	84.83	533.51
111	10.28	62.75	195.38	170.38	123.23	562.02
133	10.43	65.25	194.38	177.13	107.83	555.02
Windrow no. 2 – 8:1:2 (sewage sludge/straw/wood chips and innoculum)						
3	16.58	117.00	160.00	62.88	49.05	405.51
10	10.28	90.75	181.88	101.75	47.10	431.76
16	13.58	102.00	177.13	85.50	60.80	439.01
24	14.40	82.00	152.63	85.88	43.60	378.51
29	14.10	88.50	186.88	94.25	39.53	423.26
48	16.20	68.50	148.50	105.50	55.05	393.75
62	13.50	63.75	169.88	112.63	40.50	400.26
83	8.85	69.50	162.75	120.50	59.40	421.00
111	9.30	67.00	157.00	141.00	54.70	429.00
133	10.80	71.63	148.25	145.50	77.33	453.51
Sewage sludge	28.95	146.63	253.25	91.13	31.80	551.76

Table 4. Average Cu content in fractions

Composting time day	Average Cu content in fractions. [mg/kg d.m]					
	FRI	FRII	FRIII	FRIV	FRV	sum
Windrow no. 1 – 4:1:1 (sewage sludge/straw/wood chips and innoculum)						
3	14.25	5.25	4.13	120.75	44.63	189.01
10	7.73	3.38	4.38	131.25	57.53	204.27
16	7.65	3.00	3.75	138.00	56.35	208.75
24	6.75	2.50	3.25	143.13	56.63	212.26
29	5.33	2.25	3.50	133.88	74.13	219.09
48	4.35	1.75	3.38	145.88	80.15	235.51
62	3.83	1.50	3.25	155.63	74.75	238.96
83	4.20	1.50	3.25	171.25	65.55	245.75
111	3.75	1.25	3.00	169.13	78.13	255.26
133	4.50	1.50	2.75	166.00	78.75	253.50
Windrow no. 2 – 8:1:2 (sewage sludge/straw/wood chips and innoculum)						
3	18.00	6.75	5.25	117.63	34.88	182.51
10	6.38	2.63	3.63	145.50	44.38	202.52
16	7.13	3.00	3.75	132.00	46.88	192.76
24	6.08	2.25	3.63	120.25	37.80	170.01
29	6.08	2.13	4.50	133.00	48.05	193.76
48	5.48	1.75	2.88	120.50	44.90	175.51
62	4.13	1.38	3.00	129.38	41.63	179.52
83	3.15	1.63	2.88	122.00	54.60	184.26
111	3.45	2.13	3.00	122.63	59.05	190.26
133	3.90	2.13	2.63	125.88	66.73	201.27
Sewage sludge	27.75	8.88	9.00	131.38	48.25	225.26

Observed increases of total contents of tested metals were associated with successive ullage of organic matter content during composting causing decrease of the dry matter content. In the reaction close to neutral (pH ≈ 7), heavy metals practically do not form any soluble compounds. Therefore, the total content of heavy metals does not change significantly but their concentration, translated into diminishing dry matter, increases. The phenomenon of increase of heavy metals concentration during composting is confirmed by values of calculated Pearson correlation coefficients defining the linear interdependence level. Data put in Table 5 show strong negative correlation between the tested metals and organic matter (OM) although in Cu case, obtaining of the result that would confirm the increasing concentration phenomenon required intervention in the data structure and removal of the so-called outlier not falling within the predefined 95% level of confidence.

Table 5. Pearson correlation coefficients values

Series	Correlation		
	Ni /*om*	Cu /*om*	Zn /*om*
No 1	-0.69	0.14/-0.38	-0.44
No 2	-0.28	-0.58/-0.65	-0.55

Following removal of one case from the available database, value of the correlation coefficient describing the force of relation between Cu and OM (organic matter) decreased from 0.14 down to -0.38 in series 1 and from -0.58 down to -0.65 in series 2. Action consisting in elimination of outliers impact, or possibly their substitution with average values, may change the force of the observed relation fundamentally and is an element of proprocessing used in statistical analysis. Figures 1 and 2 show course of the regression lines using all the data respectively and after removal of one case describing copper content in sewage sludge (Table 4).

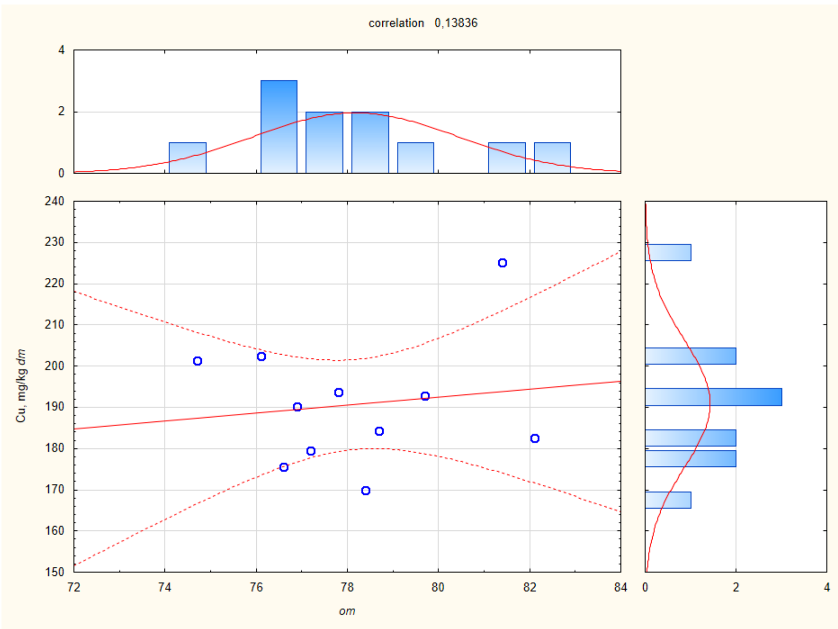

Fig. 1. Regression line and force of correlation describing Cu-*OM* association before data correction

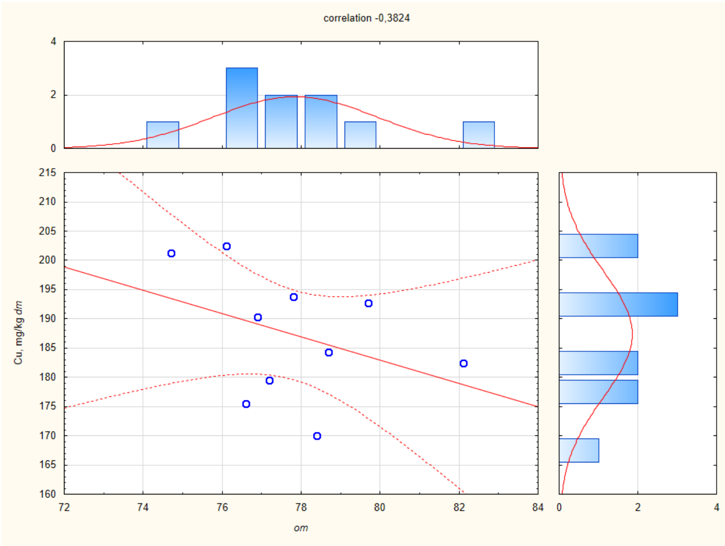

Fig. 2. Regression line and force of correlation describing Cu-*OM* association after data correction

Despite a failure to determine the admissible Cu and Zn contents in soil improver, high concentration of both elements, likewise Ni, was the main cause of fractioning operation for all three heavy metals. Metal fractioning allows for settlement of chemical forms of given element in the same compost sample. Contents of heavy metals bound in form of various compounds results from settlement of a natural equilibrium depending on the environmental conditions. A partial objective for this stage of research was the assessment of a risk originating from presence of mobile forms of nickel, copper and zinc posing a real hazard for the environment. It is considered that binding of heavy metals in the solid phase during composting, e.g. in the structure of humic compounds and argillaceous minerals, compared to other chemical forms, is exceptionally strong. Thus, the risk of heavy metals liberation to the ground and their bioaccumulation in cultivated plants is reduced.

Compost samples fractioning used to distinguish metal compounds groups featuring specific properties has been performed using the Tessier's method. According to the said method, five groups have been identified as: rechangeable metals (fraction I), carbonates bound metals (fraction II), metals bound with hydrated oxides of iron and manganese (fraction III), metals bound with organic matter (fraction IV) and metals bound with aluminosilicates (fraction V). Fractions I and II are considered unstable, liable to liberate metals to the environment. However. metals in fractions IV and V are bound in a permanent way (Szymański et al. 2005).

Test results have shown that sewage sludge composting process runs correctly even at initial proportion of C/N ≈ 10, i.e. much lower than the recommended value 15-25. When we compared test results for performed composting trials (series), it turned out that the lower the volume of sewage sludge with relation to straw per batch, the higher the temperature inside compost windrows and longer the period of the thermophilic phase (STEP 2018). Consequently, more favourable changes as to allocation of heavy metals chemical forms had been occurring. Having analysed structure of the data in terms of change of determined fractions contents with flow of composting time, it can be stated that metal contents bound in fraction I and II decreased and increased in fractions IV and V (Table 2-4).

For nickel, in both series (Table 2), total concentration values in mature compost attained the following levels respectively: 40 and 34% of the admissible value i.e., 50 mg/kg DM (Regulation EU 2019). Average nickel content in fractions IV and V in mature compost produced in each performed series constituted more than 97% of the total content. Ni contents (in mature compost) in ion-exchange fraction, carbonate bound and Fe/Mn oxides fractions did not exceed 1% of the total contents. This means that any toxic hazard originating from nickel impact, in the case of compost usage in agriculture, practically does not exist. Changes of contents of nickel bound in particular fractions in series 1 and 2 is illustrated by linear regression graphs shown in Fig. 3 and 4 respectively. Similar trend has been noted for copper and zinc.

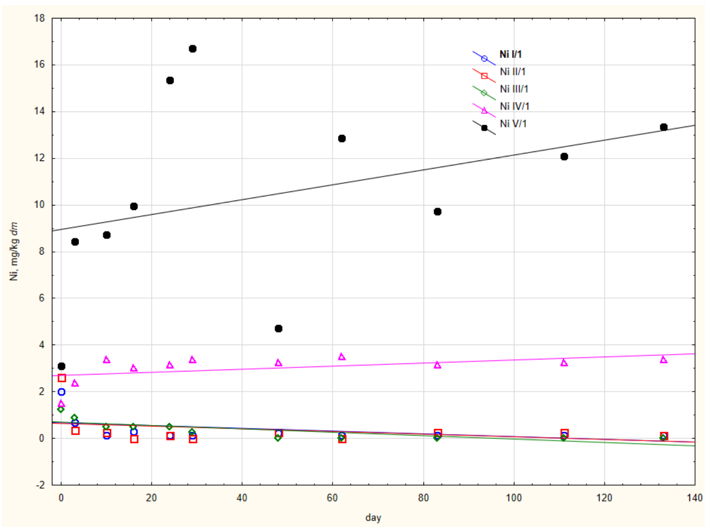

Fig. 3. Trend lines for contents of Ni bound in five tested fractions – series 1

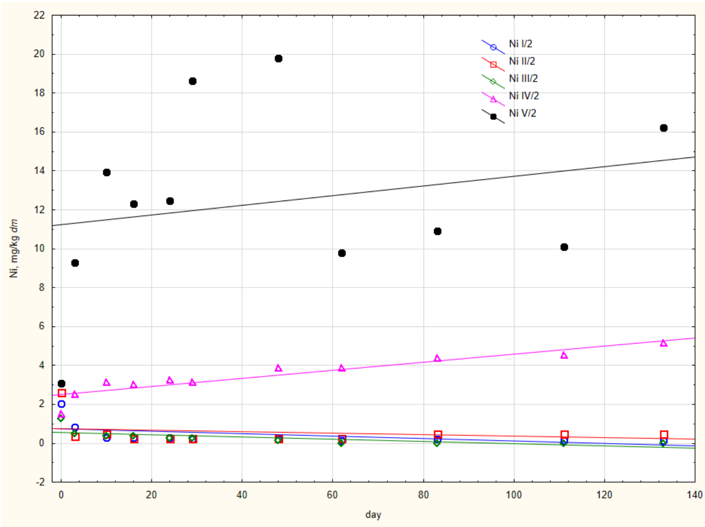

Fig. 4. Trend lines for contents of Ni bound in five tested fractions – series 2

Total zinc contents in mature compost samples in both series was 555.02 mg/kg DM and 453.51 mg/kg DM respectively and did not exceed the admissible value set in the Regulation of the European Parliament and of the Council (EU) 2019/1009 of 5 June 2019 (Regulation EU 2019) – i.e. 800 mg/kg DM (Tab. 3). Zinc occurred in tested samples mainly in the Fe/Mn oxide (FIII) and organic (FIV) fractions. Percentage of Zn in fraction FIII series 1 amounted from approximately 32% to approximately 44% whereas in series 2 from approximately 34% to 40% of total content. In both compost windrows (series 1 and 2), decrease of ion-exchange zinc compounds (FI) was observed (Table 3). Compost samples taken during the last day of series 1 process (lower sewage sludge content in the composted biomass), featured higher percentage of Zn in fraction I (2.38%) than the samples taken for series 2 (1.88%).

During the course of the process decrease of zinc compounds in carbonate fraction content was noted. Percentage of Zn, in samples taken on the 133rd day, amounted to, in this fraction, 15.79% (series 1) and 11.76% (series 2) of the total content. However, concentration in the fraction bound with organic matter (FIV) was increasing (Table 3).

Total content of Cu in the samples taken at the final composting stage amounted to 253.50 mg/kg DM (series 1) and 201.27 mg/kg DM (series 2) (Tab.4) and did not exceed the admissible value set in the Regulation of the European Parliament and of the Council (EU) 2019/1009 of 5 June 2019 (Regulation EU 2019). It has appeared from the fraction analysis that copper occurred mainly in the organic fraction (FIV). Percentage of Cu bound with organic fraction taken from windrow during composting was from 61% to 72% of total content of this element. Percentage of Cu in the residual fraction (FV) has been noted within the 19-34% interval. The lowest copper compounds contents percentage at both stages has been noted for the fraction bound with Fe/Mn oxides and with carbonates. It was 1.30% for series 1; 1.08% for series 2 and 1.06% for series 1; 0.59% for series 2 respectively. Percentage of ion-exchange copper compounds at the first composting stage (up to the third day) was approximately 9% of total content of this element whereas in mature compost samples the percentage of ion-exchange copper forms did not exceed 2% of the total content. Compost samples taken from a windrow containing higher share of sewage sludge in the composted mass (series 1) featured higher content of copper forms in fractions I, II and III than in series 2.

4. Summary

The speciation analysis results pertaining to determination of zinc, copper and nickel in sequentially separated chemical forms isolated from compost samples have shown that as composting time flows, beneficial changes as to allocation of the tested heavy metals take place. Systematic increase of the contents of tested elements in stable fractions at the expense of the fractions, which relatively easily liberate heavy metals bound with them, has been ascertained. Fractions III, IV and V, unlike fractions I and II, are treated as biologically stable. During sewage sludge composting with straw added in proportion 4/1, amount of zinc bound in three of the above-mentioned fractions increased from 271.9 to 371.1 mg/kg DM, which indicates 37% increase. For Cu and Ni, increase being estimated in similar way amounted to 24% and 43% respectively. Increase of sewage sludge volume in straw mixture up to the level having mass proportion of both components 8/1, results in higher allocation of the tested heavy metals in non-bioavailable fractions. Total contents of Zn, Cu and Ni in fractions III-V increased by 58%. 46% and 73% respectively.

Therefore, a conclusion can be drawn that decrease of supplementation consisting in lower amount of straw added to composted sewage sludge, thus reduction of C/N proportion, clearly improves the effect of heavy metals bonding in non-bioavailable chemical forms.

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