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Assessment of heavy metals content in podzolic soil for various granulometric composition when applying activated sludge as the basis for nanofertilizer (the pulp-and-paper industry waste)

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ABSTRACT: Introduction. Activated sludge is one of thepulp-and-paper industry waste types. Within the framework of the rational natural resources' utilizationand the waste recycling, due to its composition, activated sludge could be subjected to certain technological solutions for the production of nanofertilizers, since it has been previously the basis for making of various soils and biological products. However,occasionally the composition of activated sludge may contain different toxic compounds, heavy metals, and their impact on soil fertility and plants vital state is profound. Thus, the purpose of our research is to study the effect of the activated sludge introductionas a basis for nanofertilizers on the heavy metals contentin podzolic soils of various granulometric composition in agricultural exploitation. **Methods and materials.** Research was conducted under the conditions of dummy experimentin vegetation vessels. We have used podzolic soils of various granulometric composition (clayey, loamy, sandy) and pulp-and-paper industry waste – activated sludge in concentrations of 1; 2.5; 5 and 10% of the dried soil weight. Determination of the heavy metals gross content has been carried out by the atomic absorption method with measurement on the AA-7000 spectrophotometer (Shimadzu, Japan); mobile fraction of heavy metalsdetermination (подвижныеформы) – utilizing acetate-ammonium buffer solution by inductively coupled plasma mass spectrometry methodology. **Results and discussion.** The paper presents the results of the research on mobileand gross forms of heavy metals in podzolic soil of various granulometric composition when applying activated sludge as the basis of organic nanofertilizer under the dummyexperiment. **Conclusion.** It was shown that the content of mobile and gross forms of the studied metals (Fe, Mn, Cu, Zn, Ti, Al, Ni, Co, Cr, Cd and Mo) were within the health-based exposure limits, with the exception of Cd gross form, where the maximum excess was 2.5 MAC (maximum allowable concentration).

KEYWORDS: podzolic soil, activated sludge, nanofertilizer, heavy metals, pulp-and-paper industry waste.

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INTRODUCTION

To date, the pulp-and-paper industry occupies leading position as a part of the timber processing complex of Russia, while using for production needs 18% of harvested rawwood materials, and its share in commercialoutput of the timber complex is 42.0% [1]. At the same time, the production process of the industry is accompanied by the generation of various wastes, as well as pollutant emissions and discharge into the environment. Environment pollution caused by pulp-and-paper mills affect production sector, agriculture, forestry, fisheries and housing and utility sector, as well as public health.

The waste management state can be defined as critical. Annually up to 7 bln metrictons of waste are generated in Russia, and only two of them are used as recyclable materials, while pulp-and-paper industry waste can account for as much as15% [2]. One of the waste typesis activated sludge, the generation of which is originated from biological treatment of industrial effluents at the pulp-and-paper mills, which, as a rule, is stored in mud settling pits. The activated sludge consists of microorganisms, mineral particles, organic residues, proteins, nitrogen and phosphorus [3, 4].

Meanwhile,there are a certain number of issues that have accumulated in the world practice of agriculturedue to population growth, climate change, soil covering degradation, a decrease in crop yields, the use of synthetic fertilizers that complicate the situation. To deal withthe above-mentioned issues, it is proposed to use nanotechnology aimed at maintaining sustainable agriculture through the production of fertilizers on an individual basis withprearranged properties. The nanotechnology utilization in the form of nanofertilizers is an innovative, effective and environmentally friendly alternative to synthetic fertilizers. That sort of fertilizers are applicable for improve plant nutrition, increase nutrient utilization, and improve the microbiome and soil fertility [5–7].

Nanofertilizers mode of action is based upon the fact that nutrients used separately or in combination bind to nanoscale adsorbents, which release nutrients rather slowly compared to conventional fertilizers [8, 9]. The main components of such fertilizers can be algae [10, 11], biochar [12, 13], plant biomass residues [14, 15], biopolymers [16], wastewater residues, but the usage must be with caution as they may contain high concentrations of heavy metals [17–19].

Within the framework of the rational natural resources' utilizationand the waste recycling, due to its composition, activated sludge could be subjected to certain technological solutions for the production of nanofertilizers, since it has beenpreviously the basis for making of various soils and biological products [20–22]. However, sometimes the composition of activated sludge may contain toxic compounds, heavy metals, pathogens that can have a negative effect on soil fertility and the vital state of plants [23, 24]. In this regard, **the purpose of our research was to** study the effect of applying activated sludge as a fertilizer on the content of heavy metals in podzolic soil of various granulometric composition located in agricultural use. However, occasionally the composition of activated sludge may contain different toxic compounds, heavy metals,and their impact on soil fertility and plants vital state could be profound. Thus, the purpose of our research is to study the effect of the activated sludge introduction as a basis for nanofertilizers on the heavy metals content in podzolic soils of various granulometric composition in agricultural exploitation.

METHODS AND MATERIALS

Research was conducted under the conditions of dummy experiment in vegetation vessels. Pre-dried to an air-dried basis, the humus-accumulative horizon of agricultural podzolic soil of various granulometric composition (clayey, loamy, sandy) was passed through a sieve with a diameter of 1 mm; then pulp-and-paper industry waste - activated sludge in concentrations of 1; 2.5; 5 and 10% of the dried soil weight (1 kg) was introduced and thoroughly mixed. Further the soil was moistened up to 70% of its total moisture capacity and the sample had been incubating at a constant temperature (23°C) and periodic stirring for 90 days. Samples collection for the determination of gross and mobile fraction of heavy metals (Fe, Mn, Cu, Zn, Ti, Al, Ni, Co, Cr, Pb, Cd, Mo) was carried out upon completion of incubation.

Determination of the heavy metals gross content has been carried out by the atomic absorption method with measurement on the AA-7000 spectrophotometer (Shimadzu, Japan); mobile fraction of heavy metals determination - utilizing acetate-ammonium buffer solution by inductively coupled plasma mass spectrometry methodology [25].

The maximum allowable concentration (MAC) and approximate permissible concentration (APC) of chemicals in the soil are given in accordance with the Decree ... (2021).

RESULTS AND DISCUSSION

The Republic of Karelia features of the natural and climatic conditions contributed to the formation of a wide variety of soils, among which primitive immature soil, podzolic, soddy, marshy and alluvial soils prevail. Podzolic soils are the most common, and, in turn, they are divided into two groups depending on the granulometric composition:developedon sands and loams; developed on clay loams and clays. In general, podzolic soils are characterized by an acidic reaction of the medium and lownatural fertility, however, they are actively used for arable farming. Arable and cultivation of podzolic soils primarily leads to a sharp decrease in the content of organic matter.

There has been a removal with the harvesting and leaching into the lower horizons of the mineral elements for plants (nitrogen, mobile forms of phosphorus and potassium), which leads to the need for their regular introduction into the soil in the form of fertilizers. Cattle manure is used as an alternative source of organic and mineral fertilizer in the region, and recently we consider options for timber manufacturing industry and pulp-and-paper industry waste to use.One of which is activated sludge[23].

Content analysis for the of gross forms of heavy metals (Fe, Mn, Cu, Zn, Ti, Al, Ni, Co, Cr, Pb, Mo) for possible experimental variantsin podzolic soil of various granulometric composition (clayey, loamy, sandy) and various rate of applicationfor activated sludge showed that there was no significant increase, as well as exceedin maximum or approximate permissible concentrations.

The only exception was the contentof Cd, which belongsto the first class of hazard, as the final concentration for possible experimental variants exceeded the MAC with a maximum value of 2.5 times (Table 1). Increase in the content of Cd in possible experimental variants depended on the dose of application of activated sludge, while the correlation coefficient was 0.6 for podzolic-claysoil; for loamy – 0.9 and for sandy – 0.6.

However, it should be noted that the Cdcontentin thebackground soils was initially close to the MAC (from 0.4 to 0.8), and at the same time, the presence of a certain amount forCd in the activated sludge has led to the manifestation of the "cumulative effect".

Themobile fraction of studiedheavy metalsanalysis has showed that for possible experimental variants their concentration was significantly lower than the MAC and APC.Nevertheless, there is a variety of trends for increase or decreasein their content (Table 2). Thus, there was a significant decrease in the content of mobile forms Fe, Mn, Ti, Pb and Cdin podzolic-sandy soil; Ti and Cd – in podzolic-loamy soil; Ti, Cd and Mo-in podzolic-clay soil.But there was an increase in Ni and Cr,meanwhileit should be noted that there were no correlations between the concentration of the studied metals and activated sludge various rate of applicationin some variants.

That sort ofmultidirectionality is most likely explained by the fact that the mobility of heavy metals in the soil is influenced by a host of factors, such as the content of organic matter, environmental acidity, the granulometric composition and chemical properties of each element individually and its ability to enter into various compounds [26, 27]. For this reason, a more detailed analysis of the heavy metalsdynamicsmobilityshould be carried out with this consideration in mind.

Table 1

Gross forms of heavy metals content changein podzolic soil with different granulometric composition

Variants of experiment	Fe	Mn	Cu	Zn	Ti	Al	Ni	Co	Cr	Pb	Cd	Mo
	mg/kg of soil											
Podzolic-claysoil (PCS)												
PCS, background	837	23,3	0,9	25,1	17,9	655	3,17	0,87	2,06	11,44	0,59	0,22
PCS + 1% AS*	832	18,5	2,4	22,4	14,7	616	4,44	0,86	3,49	11,70	1,09	0,45
PCS + 2,5% AS	820	22,6	0,7	27,5	18,8	638	4,43	0,83	2,87	13,27	1,17	0,21
PCS + 5% AS	870	20,5	0,6	25,9	14,3	680	3,31	0,87	2,13	10,18	1,11	0,23
PCS + 10% AS	860	23,1	2,3	29,4	16,3	641	3,23	0,91	2,13	13,45	1,18	0,23
Podzolic-loamysoil (PLS)												
PLS, background	3799	42,9	3,9	6,6	404,0	2823	2,39	1,56	6,11	0,61	0,42	0,06
PLS + 1% AS	3691	40,6	3,6	6,0	539,1	2629	3,26	5,52	5,67	0,19	1,49	0,06
PLS + 2,5% AS	3609	38,1	3,1	3,4	387,9	2446	3,58	3,01	5,60	0,83	1,57	0,02
PLS + 5% AS	3913	40,6	3,4	6,2	512,7	2586	3,13	2,77	6,15	0,94	1,68	0,09
PLS + 10% AS	4018	46,1	3,4	6,6	619,3	3164	3,22	3,05	6,19	0,87	2,57	0,07
Podzolic-sandysoil (PSS)												
PSS, background	19903	416,5	6,9	33,3	687,4	7533	8,22	4,25	15,13	9,77	0,80	0,36
PSS + 1% AS	23838	433,7	6,7	33,1	835,0	8754	8,36	4,45	15,33	11,06	1,01	0,36
PSS + 2,5% AS	21308	462,1	7,6	37,8	994,3	8814	7,54	4,87	17,04	11,61	1,83	0,20
PSS + 5% AS	21845	476,0	6,9	34,4	710,8	8942	7,89	3,83	19,53	9,78	1,93	0,16
PSS + 10% AS	20606	442,1	6,9	34,2	836,2	8747	7,69	4,03	16,54	10,24	1,62	0,29
MAC*/APC*	–	1000	66,0	110,0	–	–	40,0	–	–	65,0	1,0	–

Notes: AS – activated sludge, MAC – maximum allowable concentration, APC – approximate permissible concentration

Table 2

Mobile fraction of heavy metals content change in podzolic soil with different granulometric composition

Variants of experiment	Fe	Mn	Cu	Zn	Ti	Al	Ni	Co	Cr	Pb	Cd	Mo
	mg/kg of soil											
Podzolic-claysoil (PCS)												
PCS, background	20	11,4	0,3	24,0	0,4	63	0,99	0,55	0,16	5,07	0,40	0,004
PCS + 1% AS	19	7,7	0,2	20,4	0,2	61	1,35	0,45	0,26	6,90	0,09	0,001
PCS + 2,5% AS	17	8,9	0,1	23,1	0,1	61	1,39	0,68	0,40	7,23	0,11	0,001
PCS + 5% AS	23	8,2	0,3	23,4	0,1	56	1,17	0,65	0,21	5,04	0,10	0,001
PCS + 10% AS	24	16,0	0,1	23,4	0,1	67	2,26	0,73	0,33	6,05	0,11	0,002
Podzolic-loamysoil (PLS)												
PLS, background	3	1,1	0,1	0,3	1,80	10	0,04	0,03	0,03	0,04	0,01	0,001
PLS + 1% AS	3	1,1	0,1	0,3	0,02	10	0,04	0,03	0,03	0,03	0,001	0,001
PLS + 2,5% AS	3	1,3	0,1	0,4	0,01	12	0,05	0,04	0,03	0,04	0,005	0,001
PLS + 5% AS	3	1,9	0,1	0,4	0,02	11	0,04	0,05	0,03	0,04	0,004	0,002
PLS + 10% AS	4	1,5	0,1	0,6	0,10	14	0,07	0,03	0,04	0,09	0,001	0,001
Podzolic-sandysoil (PSS)												
PSS, background	103	38,2	0,1	2,2	0,001	22	0,13	0,18	0,12	0,38	0,01	0,002
PSS + 1% AS	41	16,8	0,05	0,7	< 0,0001	18	0,10	0,10	0,09	0,08	0,002	0,001
PSS + 2,5% AS	68	22,3	0,1	3,3	< 0,0001	20	0,14	0,12	0,08	0,10	0,003	0,002
PSS + 5% AS	82	21,6	0,03	2,1	< 0,0001	24	0,17	0,12	0,10	0,12	0,002	0,001
PSS + 10% AS	39	16,2	0,03	1,1	< 0,0001	18	0,11	0,08	0,08	0,06	0,002	0,002
MAC*/APC*	–	80,0	3,0	23,0	–	–	4,0	5,0	6,0	6,0	–	–

Notes: AS – activated sludge, MAC – maximum allowable concentration, APC – approximate permissible concentration

CONCLUSION

Thus, the studies carried out in the dummy experiment on the study of the content of gross and mobile forms of heavy metals whenapplying activated sludge as a waste of the pulp-and-paper industry into podzolic soil of various granulometric composition (clayey, loamy, sandy) has showed that in among the gross forms, the content of Fe, Mn, Cu, Zn, Ti, Al, Ni, Co, Cr, and Mo was lower than MAC; and the content of Cd has exceeded health-based exposure limits. The values of mobile forms remained within normal limitsin the studied metals with the multidirectional dynamics of their contents.

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Maria G. Yurkevich – general leadershipof the research and the conduct of the experiment.

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