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DETERMINATION OF CADMIUM IN A MUNICIPAL SEWAGE SLUDGE BASED COMPOST BY SPECTROPHOTOMETRIC METHOD

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Abstract. An experimental investigation on cadmium detection in the municipal sewage sludge (MSS) based compost by spectrophotometric method, has been carried out in a scientific laboratory of the Department of Environmental Protection of Vilnius Gediminas Technical University. For this purpose a reagent of 5, 7 dibromo – 8 – hydroxyquinoline (DBHQ) has been used. In reaction of the reagent with cadmium a leach of greenish-yellow has been produced and absorption wavelength of spectrophotometer has been chosen at visible UV length, about 396 nm. The method is simple to use and sensitive enough for the detection of cadmium amounts in solid samples. The investigation has been carried out in two ways of samples preparation: mineralization, for the total cadmium amounts investigation in the compost and investigation of 24 h batch leach of compost for soluble cadmium values detection.

Keywords: Compost, sewage sludge, spectrophotometry, cadmium, municipal wastewater.

1. Introduction

About 183.2 mln/m^3 of municipal and industrial wastewater was taken back to the water sources in the year 2006 in Lithuania (Aplinkos … 2007). A residual product of wastewater treatment is sewage sludge whose annual production rate is about 66 thousand tonnes of dry matter (DM) (Environment … 2006). About 20 thousand tones of sewage sludge are composted and applied on arable lands for the soil fertilisation. Unfortunately, the mass consumption increase and it directly influences the natural resources' use, therefore there is a tendency for waste amount to increase as well (Vasarevičius *et al.* 2005).

Cadmium is a trace, extremely toxic metal. In nature it occurs in trace amounts and presents 0.00005% of the crust of the earth (Jankiewicz *et al.* 2000). In comparison with other heavy metals, it is an environmental pollutant, but easier than the others accumulated in the food chain. Cadmium can get into the bloodstream by absorption from stomach or intestines after food or water ingestion or by absorption from lungs after inhalation. It mainly accumulates in the kidneys and at high levels it can lead to serious kidney failure. The major route for cadmium intake is its ingestion. This is largely due to the presence of trace levels of cadmium in foodstuffs of natural origin or use of phosphate fertilisers and sludge on agricultural soils. The metal in trace amount is very important industrially, it is widely used in special alloys, pigments, coatings stabilisers and in rechargeable nickel cadmium batteries (Maria S. Di Nezio *et al.* 2005). General sewage sludge composition is shown in Table 1.

Municipal sewage sludge based compost with the intention to use in agriculture has to match the legal limits related with the policy restrictions for the negative environmental impact by associated compounds (Stehouwer 1999). According to the agricultural policy, every use of compost in farming must be tested and monitored. The main attention should be paid to the pathogens and heavy metals that have the highest direct and indirect impact on human health (Environmental … 2001).

The aim of this paper was to investigate the cadmium amounts in the compost from municipal sewage sludge and accumulation of it in the soil. The amount of the metal expected to be trace and the method chosen for the investigation should be simple to use and essentially sensitive for the determination. However, the determination of trace metal ions in natural waters and soil is difficult owing to different factors, particularly their low concentrations and matrix effects (Maria S. Di Nezio *et al.* 2005). The investigation is related with the complex procedures of solvent extraction, ion exchange or precipitation to eliminate the matrix or the source of the interfering ions (Kauneliene, Gelažiene 2002; Paliulis 2006).

Table 1. Chemical composition of dry matter (DM) of municipal sewage sludge (European Commission 2001)

Dry matter	30 g/L	Ca	10%
pH	7%	Fe	2%
C	49%	Mg	0.6%
H	7.7%	Cd	0.4–3.8 mg/kg
O	35%	Zn	142–2000 mg/kg
N	6.2%	Cr	16–275 mg/kg
S	2.1%	Cu	39–641 mg/kg
P	2%	Hg	0.3–3 mg/kg
Cl	0.8%	Ni	9.0–90 mg/kg
K	0.3%	Pb	13–221 mg/kg
Al	0.2%		

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Several analytical techniques, such as flame atomic absorption spectrometry (FAAS) inductively coupled plasma atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS), absorption molecular spectroscopy (UV-V) (Maria S. Di Nezio *et al.* 2005) can be used for the determination of traces of cadmium amounts using different reagents with enough sensitivity for the related applications.

The chosen method used for the investigation is spectrophotometric cadmium detection using 5, 7 dibromo – 8 – hydroxyquinoline (DBHQ) reagent, proposed by J. M. Jamaluddin (2004). Spectrophotometry is a chemical analysis tool, simple and sensitive to use for the essential trace analyses. The reagent used for the experiment is reported to be a reagent for the investigation of vanadium and molybdenum as well as cadmium (Jamaluddin Ahmed, Tauhidul Chowdhury 2004). The method is based on the reaction of non-absorbent DBHQ with cadmium in slightly acidic solution producing greenish-yellow solution product.

2. Experimental and simulation background

The experimental research on the cadmium determination in the samples of compost soils was carried out in the scientific laboratory of the Department of Environmental Protection of Vilnius Gediminas Technical University.

The municipal sewage sludge and "green waste" mass based compost were investigated. The samples were taken from the municipal waste water treatment and composting plant. The origin of the municipal wastewater was the city of around 700 thousand inhabitants with a low rate of manufacturing industry and industrial wastewater reserves and with non-separate rain water collection system.

The composting process lasted from 30 to 60 days in a windrow composting site. The sludge in a phase of cake came to the field of composting. For the compost the municipal sewage sludge and green waste were mixed in proportion of 1:3. After the regular (7 days period) screening of piles for the oxygen ingestion the drying process was to follow. The temperature in the piles during the process reached 40–70 °C. Carrying on the experiment it is important not to exceed the temperature range. If the temperature is too high, the compost micro-organisms, necessary for decomposition, may be inhibited (Wilson *et al.* 1980), when the temperature is too low there is a risk of survey of pathogenic organisms. The moisture in a pile must to be about 40% that gives sufficient air spaces for an aerobic environment. The pH of the product ranges from 6.9–8.5 and it increases because of the wood chips' burning capacity. In the end of the period the piles were screened again for the particles reduction till the substances of 0.2–2 cm and final product – compost was ready.

Basic reagents and instruments used in the experiment were as follows:

- Distilled deionised water;
- Ethanol;
- DBHQ;
- crystallized cadmium sulphate ($3CdSO_4 \times 8H_2O$);
- Hydrochloric acid HCl;
- Nitric acid HNO_3;
- Compost samples;
- Spectrophotometer KFK-3;
- pH meter
- Mixer;
- Centrifuge;
- Glass vessels, flasks;
- Retorts 25 ml, 50 ml, 100 ml, 1 000 ml;
- Pipettes of 1, 2, 5, 10 ml;
- Paper filters „blue film".

Two types of laboratory tests were carried out by the research team. Samples for the investigation were prepared by the method of mineralization in case of total Cd determination. The second method was the batch and rinse of prepared solution for 24 h before the spectrophotometry. The leaching method was also used for the compost elemental composition determination. The advantage of the method is a leaching environment, which represents conditions similar to natural world (pH similar to soil ph). Time period can be the key for the elemental flow simulation in time scale of material flow analysis. The results would show additionally the properties of the absorption process of elements. Time range is an important condition which influences the leaching (dissolving) of cadmium. That is why the research group chose 24 hours duration of the experiment. A prepared solution had to be within pH-value of 6.

Compost for the experiment had to be as homogeneous as possible and also dry. For the processes of mineralization, 0.5 g of compost was weighted and mixed in appropriate amounts (37.5 mg and 12.5 mg) of respectively HCl and HNO_3 solution and kept for 24 hours. After the mineralization, solution samples were supplemented with water and bases to reach and sustain a constant level of pH 6.0. For the second method, homogeneous compost was weighted and rinsed with 200 ml of water solution within the 24 hours period. This method is more similar to natural conditions like rainfall and slow dilution. The particles of sewage sludge have properties to be very small in size of 1–3 μm. Before the spectrophotometer, the solutions of compost samples were centrifuged to separate solid particles and filtered by blue label filters.

Cadmium standard solution (divalent cadmium stock solution) of 2.97×10^{-3} M was prepared by dissolving crystallized cadmium sulphate ($3CdSO_4 \times 8H_2O$) in distilled water. 5, 7-dibromo-8-hydroxyquinoline (DBHQ) was used as spectrophotometric reagent for cadmium and powder of DBHQ reagent which was dissolved in a standard volume of ethanol.

Sulphuric acid was selected as the best acid for the system in comparison to nitric, phosphoric, hydrochloric (Jamaluddin Ahmed, Tauhidul Chowdhury 2004). The acid is prepared to be 0.0005 M.

For the spectrophotometric investigation, solution of neutral aqueous solution of compost or cadmium standard (in case of standard solution) in volumes of 0.2–3 ml,

1 ml prepared DBHQ and 0.5 ml H_2SO_4 solutions were mixed and fulfilled with water till 10 ml.

The spectrophotometer KFK–3 was used for the cadmium investigation. According to the researches, the best absorbance (Fig. 1) is at wavelength of 396 nm (Jamaluddin Ahmed, Tauhidul Chowdhury 2004). The absorbance was done at the wavelength of 396 nm. The UV/visible absorption spectrum of cadmium is shown in Fig. 1. Since the maximum absorbance is at 396 nm, here should be the greatest sensitivity. Therefore, this wavelength was used for the quantitative analysis of Cd in compost samples.

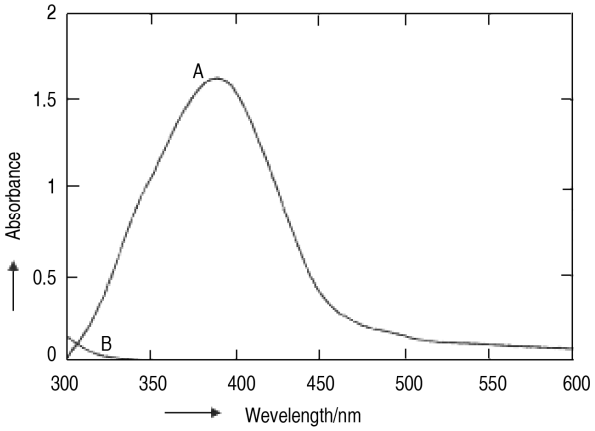

Fig. 1. Absorption spectrum of cadmium with DBHQ reagent (A) and the reagent blank (B) in aqueous solutions

The concentration of Cd in the solutions was measured by molarity M, which is expressed by number of moles of solute per volume (usually litre) of solution; molarity (m) that is the number of moles of solute per kilogram of solvent.

Movement of water and chemical compounds in soils is a dynamic process, dependant on time and space (Butkus, Konstantinova 2005). Soil, chemical properties, water and chemical application rates interact in complex ways within the soil system (Butkus *et al.* 2008). Soils are relevant to the estimation of the density of contamination of soil layers, exposure forecast, determining the penetration depth (Butkus, Konstantinova 2008). Metals behaviour in the soils is strongly affected by the processes of metal release from the solid phase into soil solution, that is interaction of metals chemical forms in the soil solution (Vasarevičius *et al.* 2005; Paliulis 2006).

The "Maximum Export" (ME) model was simulated. It expresses the initial conditions, shown in Table 2. The ME model presents the possible maximum export of cadmium from the soil or conditions for the minimum cadmium accumulation in the soil.

The heavy metal load was assumed to be the same with ML model with cadmium values in the compost about 2.28 mg/kg and the soil density of 1.4 g/cm^3 that is assumed to be sandy soil. According to the soil, the rates of runoff and leaching were set to be by 10%, wheat moisture – 15% (European Commission 2001), the annual yield assumed to be 10 t/ha.

Table 2. General information and assumptions for the "Maximum Load" model

No.	Indicator	Unit
1	Soil density	Sandy soil, D = 1.4 g/cm^3
2	Compost amount, applied on the soil	43.86 t/ha
3	Runoff level	10%
4	Leaching level	10%
5	Historic cadmium accumulation in the soil	0
6	Agricultural yield	10 000 kg/ha
7	Plants humidity	15%

3. Results and discussion

The non-absorbent DBHQ in a slightly acidic solution reacts with cadmium into greenish yellow colour. The absorbance values of a series of solutions, containing fixed concentrations of Cd (standard cadmium solution with the crystallized cadmium sulphate ($3CdSO_4 \times 8H_2O$)) and solutions prepared using the compost were all measured in triplicate at 396 nm relative to a blank solution prepared without Cd standard solution. The individual measured absorbance, averages and relative ranges are listed in Table 2.

The analysis of the data was started with the construction of a calibration curve (Fig. 2). The average absorbance of each known solution was plotted as a function of cadmium concentration. The relationship between absorbance and cadmium concentration appears in linear with $R^2 = 0.999$ 1. The highest concentration data point was excluded from the linear regression calculations. As the lower concentrations data showed a better linearity, rejection of the highest concentration data point prevented from the wrench of cadmium concentrations in the compost samples.

The linear regression gave a slope for the calibration curve of 11×10^4 abs./conc. and interception of 0.011 abs. The interception is around zero and is thus in agreement with Beer's law, which predicts a linear relationship between absorbance and concentration. Fig. 3 shows the results of mineralized samples of compost solution. The minimum total cadmium concentration is 1.32± 0.07 mg/kg, maximum total cadmium concentration is 2.81±0.14 mg/kg of compost, the average total cadmium concentration is 2.28±0.11 mg/kg of compost. The results show the waving concentrations of total Cd compounds. This might have occurred due to the measuring error or compounds homogeneity. As well, it could have been influenced by pH value. Since the solution was kept at pH 6 level, the volumes of samples have got maximum value range of 30 ml.

For the soluble cadmium detection the method of 24 h lechate solution investigation was applied. After 24 hours time samples of compost solution of rinse were filtered and tested by the same spectrophotomeric apparatus. The results are shown in the Fig. 4.

The range of results confirms the wave of results in comparison to mineralized samples. As the samples had been rinsed for 24 hours, the homogeneity was much

Table 3. The data of absorbance, averages and relative range, used for the calibration curve

Flask number	Cd concentration g Cd/l	Absorb 1	Absorb 2	Absorb 3	Average absorbance	Relative range %
ST 1	1.9962E-07	0.014	0.013	0.013	0.013 3	7.50
ST 2	3.327E-07	0.029	0.026	0.028	0.027 7	10.84
ST 3	6.654E-07	0.069	0.071	0.071	0.070 3	2.84
ST 4	9.981E-07	0.105	0.108	0.11	0.107 7	4.64

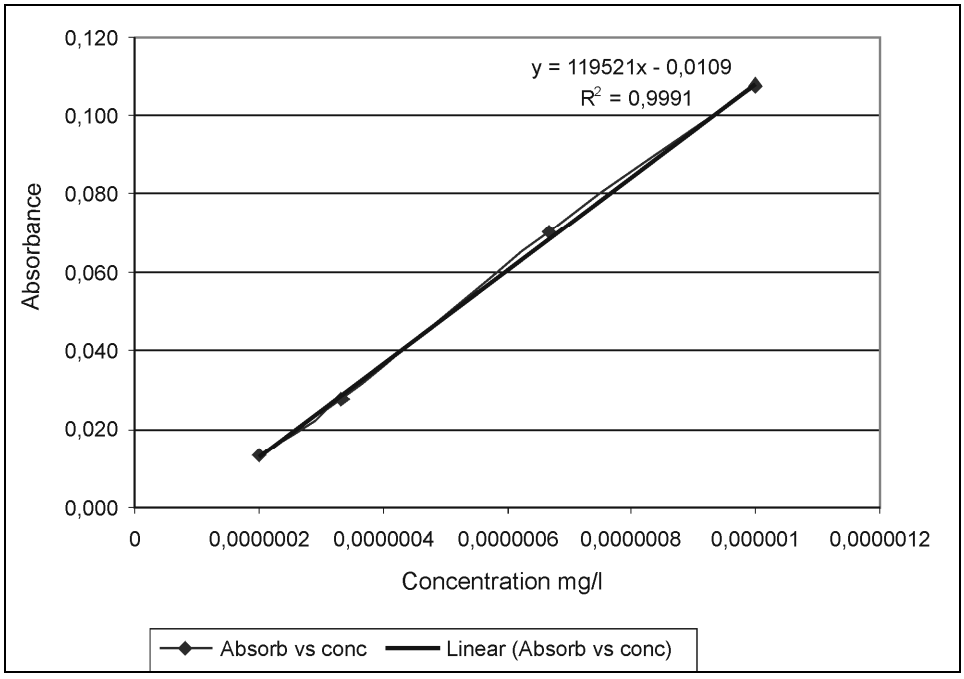

Fig. 2. Calibration graph of standard solution with divalent cadmium stock solution

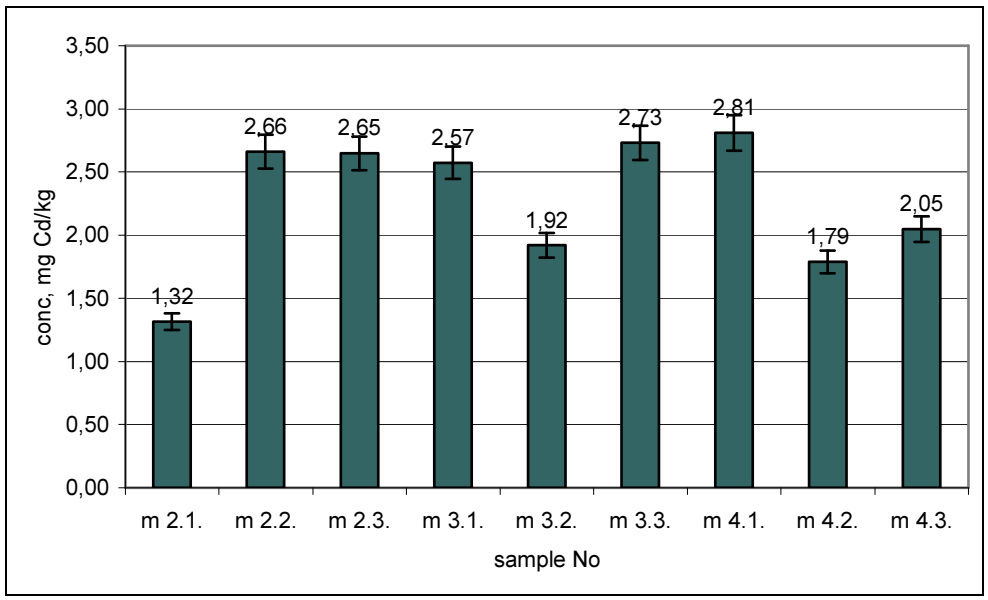

Fig. 3. Total Cd concentration in mineralized samples of compost, mg/kg

reasonable factor. Results, shown in Fig. 4, express the equality of soluble cadmium compounds production in the time period of 24 hours that depends on soluble compounds and pH conditions. The pH level in the samples was naturally about 6, but it wasn't equalized after 24 h to keep the condition natural as much as possible. The minimum concentration of soluble cadmium was 0.21±0.01 mg/kg, maximum concentration of soluble cadmium was 0.52±0.03 mg/kg of compost, average concentration of soluble cadmium was 0.34±0.02 mg/kg of compost. The trend of pH level was to decrease from around pH 6.1±0.4 to pH 5.2±0.3.

The analysis of the two methods showed that the amount of soluble cadmium is lower in comparison to mineralized samples, which results show the total cadmium rate in compost. During the 24 hour period about 15% of soluble cadmium dissolved on average in experimental conditions. Maximum dissolved amount was more than 24% in comparison to the average amount of the total detected cadmium. This shows that cadmium solubility in the compost is about 0.236 mg/kg s^{-1}.

Simulation of cadmium accumulation has been done by Excel programme. Cd accumulation in and movement through the soil is very slow. The slow movement regards to Cd concentration in long-term scale. Dissolved in soil solution Cd is transported and can be reachable and taken up by transpiration of plants or may accumulate into the soil (Vasarevičius *et al.* 2005). Cadmium ions reach plants roofs by mass flow, diffusion and interception. In this case the uptake by roots is bigger than can be supplied by mass flow, then ion concentration would be lowered at the root surface and diffusion will become increasingly important (Ingwersen 2001).

The soil and plant properties have a significant impact for the cadmium accumulation. Fig. 5 represents "Maximum Export" model.

The ME model results show cadmium transport according to the conditions with active cadmium movement due to the soil and plant properties. The annual accumulation rate is about 50% of Cd load value. The plant uptake is about 37% of Cd load. The plant uptake value is higher than cadmium solubility which, according to the experimental

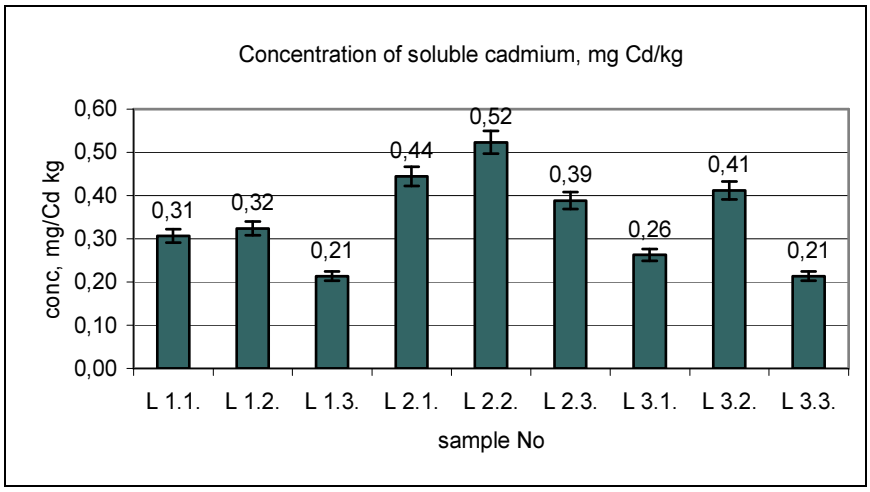

Fig. 4. Concentration of soluble Cd in lechate of compost solution, mg/kg

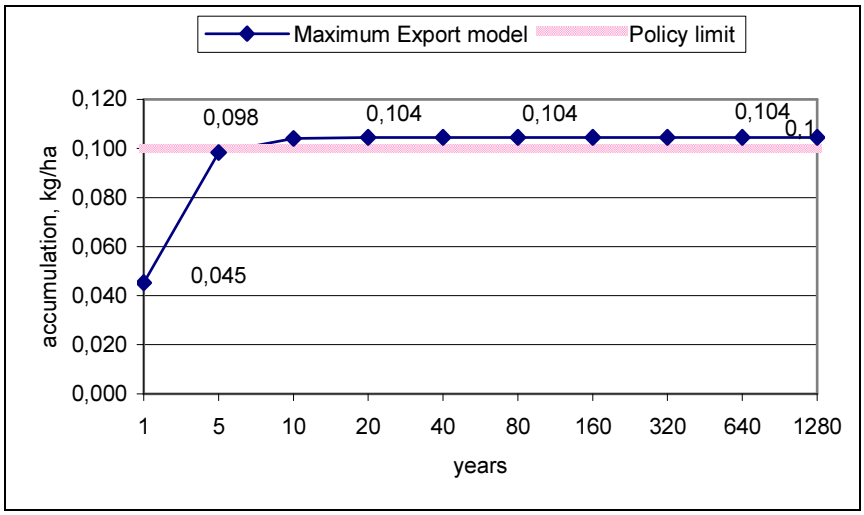

Fig. 5. Total accumulation rate of cadmium in year scale of 1–1280 in "Maximum Export" model

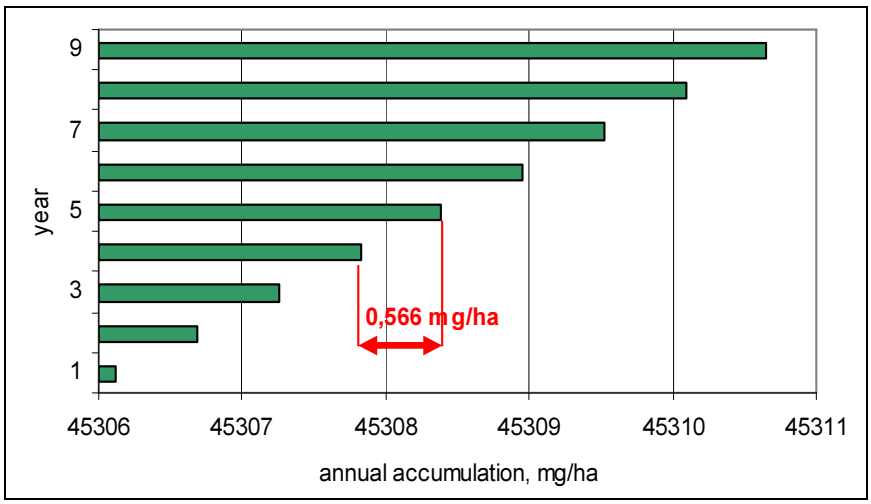

Fig. 6. Annual accumulation increase of cadmium in ME model

research, was on average 15%. Thus cadmium uptake capacity in natural condition could be lower. Cadmium accumulation values for a period of 1280 years are on limit values according to the policy requirements (according to LAND 20-2005; Nuotekų dumblas … 2005), annual accumulation increase by 0.566 mg/ha (Fig. 6). The accumulation reaches the limit values in the 6th year and appears to be constant from year 11.

4. Conclusions

An experimental investigation on cadmium detection in the municipal sewage sludge (MSS) based compost by spectrophotometric method using reagent of 5, 7 dibromo – 8 – hydroxyquinoline (DBHQ) has been carried out. The results of total and soluble cadmium in the samples of compost have been analysed.

The values of cadmium compounds in the compost samples are compatible with the legal requirements. Total cadmium values are 2.28 mg /kg. The limits for cadmium compounds in sewage sludge and sewage sludge based compost are 3 mg/kg; the compost, which exceeds the limits, can not be applied on the arable lands. Therefore, the investigated compost can not be used for land applications.

In the 24 hours batch test the measured compost solutions contained 0.34±0.02 mg/kg amount of soluble cadmium. This presents cadmium accumulation into the soils and transport delays in farming sector, and attention has to be paid to these attributes.

Spectrophotometric cadmium detection method using 5, 7 dibromo–8–hydroxyquinoline (DBHQ) reagent was simple and sensitive enough to use for the low level metals investigation in the solid samples as compost or soil. The results are logical and can be accepted in relation to the results of other researches.

The accumulation of cadmium in the soil is linear and depends on soil and plant properties. The model results show a constant accumulation from the year 11 and it does not exceed the policy limits of 0.1 mg/ha of cadmium concentration in the soils.

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SPEKTROMETRINIS KADMIO KIEKIO NUSTATYMAS KOMPOSTE, PAGAMINTAME IŠ NUOTEKŲ DUMBLO

V. Podgaiskytė, P. Vaitiekūnas

S a n t r a u k a

Tyrimo tikslas buvo nustatyti kadmio kiekius komposte, pagamintame iš komunalinių nuotekų dumblo ir „žaliųjų“ atliekų. Tyrimas atliktas Vilniaus Gedimino technikos universiteto Aplinkos apsaugos katedros mokslinėje laboratorijoje. Sunkiojo metalo kiekiai komposte nustatyti spektrofotometriniu būdu, naudojant 5, 7 dibromo-8-hidroksikinolino (*DBHQ*) (*5, 7 dibromo-8-hydroxyquinoline*) reagentą. Reagentui reaguojant su kadmio komponentais, tirpalas įgauna žaliai gelsvą spalvą. Spektrofotometru matuojant absorbcijos tankį pasirinkta 396 nm ultravioletinės (UV) bangos ilgis. Iš tyrimo rezultatų paaiškėjo, kad reagentas yra jautrus ir tinkamas naudoti mažoms kadmio metalo koncentracijoms aptikti vandens ir dirvožemio ar kitų sausųjų medžiagų terpėje. Bandymas atliktas taikant du tiriamųjų bandinių ruošimo būdus. Pirmuoju būdu komposto tirpalas buvo mineralizuojamas suminiam kadmio kiekiui medžiagoje sužinoti. Antruoju – tiriama komposto tirpalo, maišyto maišytuve 24 valandas, ištrauka. Nustatyta tirpaus kadmio (kadmio jonų) kiekis bandinyje.

Reikšminiai žodžiai: kompostas, nuotekų dumblas, spektrofotometrija, kadmis, komunalinės nuotekos.

ОПРЕДЕЛЕНИЕ КАДМИЯ В КОМПОСТЕ ИЗ ИЛА СТОЧНЫХ ВОД СПЕКТРОФОТОМЕТРИЧЕСКИМ МЕТОДОМ

В. Подгайските, П. Вайтекунас

Р е з ю м е

Целью исследования было определить количество кадмия (Ca) в компосте, полученном из ила коммунальных сточных вод (МИС) и «зеленых» отходов. Экспериментальные исследования проводились в научной лаборатории кафедры охраны окружающей среды Вильнюсского технического университета им. Гедиминаса. Количество тяжелого металла Ca в компосте определено спектрофотометрическим методом с использованием реагента 5-, 7-дибром-8- гидроксинолин (hydroxyquinoline) (DBHQ). Во время реакции реагента с компонентами кадмия раствор приобрел зеленовато-желтую окраску. Для спектрофотометра была выбрана длина ультрафиолетовой волны, приблизительно равная 96 нм. Метод прост в использовании и чувствителен к обнаружению следов кадмия в твердых образцах. Исследования проводились с применением двух способов подготовки образцов: минерализации компостного раствора для определения суммарного количества кадмия в нем и исследовании компостного раствора, выдержанного в смесителе в течение 24 часов.

Ключевые слова: компост, ил сточных вод, спектрофотометрия, кадмий, коммунальные стоки.

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Research interests: hydrodynamics, convective heat and mass transfer and thermo physics, computational fluid dynamics, mathematical modelling of transfer processes in the environment.