

## AN ATTEMPT OF CONTAMINATED DRILLING WASTE REMEDIATION BY BIOCOMPOSTING

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The evaluation of gas drilling sludges utilization using bioconversion technology in the condition of the model experiment was done in the article. The experimental chart included the mixtures consisting of the dry residue of the drilling pulp after the filtering pool of LLC "Eko TecOIL" technology, shredded wheat straw, cattle bedding manure, and high peat in different proportions based on the optimal N:P ratio in composting mass. The microbial active composition "EXTRACON"® was added to a few variants to try accelerating and shorting transformation duration. Composting process was controlled by temperature and humidity regulation. The composition of the studied mixtures and possible variants of the composting process were analyzed. The composition of the prepared composts was assessed by the main nutrient content measuring (N, P, K), the concentration of a few heavy metals (Pb, Cd), and testing their phytotoxicity. It was established that an additive of 20 wt. % of drilling wastes to compost demonstrated moderate phytotoxicity for wheat seeds germinating. In addition, a slight excess of Cadmium permitted level was observed in the three variants of compost composition. But potentially it could be led to its accumulation in root tissues, causing their damage and inhibiting the germinating.

In general, it can be concluded that the presence of unidentified chemical additives in the composition of dry drilling waste had a negative effect on the microbiological transformation of compost mixtures. This effect increased with an increase in the mass of dried drilling sludge in the initial compost composition. Apparently, as a result of this, it was not possible to detect a significant effect of introducing a microbial preparation "EXTRACON"®, since these chemical additives inhibited the activity of its microorganism's community. So, is that it is necessary to continue experimenting by changing the ratio of compost components. It is possible that reducing the mass of introduced waste or adding microbial preparations capable of degrading specific additives to drilling waste will reduce the phytotoxicity of composts. *Key words:* ecologically hazardous waste, drilling sludge wastes, bioconversion, phytotoxicity, composts, biotesting.

**Спроба утилізації забруднених відходів буріння шляхом біокomпостування. Заленська Є.А., Войтенко Л.В., Копілевич В.А., Оне О.-В.З.**

У статті проведено оцінювання можливості утилізації газових бурових шламів за технологією біоконверсії в умовах модельного експерименту. Схема дослідження включала суміші, що складалися з сухого залишку бурової пульпи після фільтраційної ванни за технологією ТОВ «Еко ТекОІЛ», подрібненої пшеничної соломи, підстилкового гною великої рогатої худоби та сапрінового торфу в різних пропорціях з урахуванням оптимального співвідношення N:P при компостній масі. До кількох варіантів додали мікробну активну композицію «ЕКСТРАКОН»®, щоб спробувати прискорити та скоротити тривалість трансформації. Процес компостування контролювався шляхом регулювання температури та вологості. Виконано аналіз досліджуваних сумішей та можливі варіанти процесу компостування. Склад підготовлених компостів оцінювали шляхом вимірювання вмісту основних поживних речовин (N, P, K), концентрації деяких важких металів (Pb, Cd) і тестували їхню фітотоксичність. Встановлено, що добавка 20 мас. % відходів буріння до компосту продемонструвала помірну фітотоксичність при проростанні зерна пшениці. Крім того, у трьох варіантах складу компосту спостерігалось незначне перевищення допустимої норми кадмію. Проте потенційно це може призвести до його накопичення в тканинах коренів, викликаючи їх пошкодження та перешкоджаючи проростанню.

Загалом можна зробити висновок, що наявність неідентифікованих хімічних добавок у складі сухих відходів буріння негативно вплинула на мікробіологічну трансформацію компостних сумішей. Цей ефект посилювався зі збільшенням маси висушеного бурового шламу у вихідній композиції для компостування. Очевидно, внаслідок цього не вдалося виявити істотного ефекту від введення мікробного препарату «ЕКСТРАКОН»®, оскільки ці хімічні добавки пригнічували життєдіяльність консорціуму його мікроорганізмів. Отже, необхідно продовжувати експерименти, змінюючи співвідношення компонентів компосту. Можливо, зменшення маси внесених відходів або додавання до відходів буріння мікробних препаратів, здатних розкладати специфічні добавки, знизить фітотоксичність компостів. *Ключові слова:* екологічно небезпечні відходи, шламові відходи буріння, біоконверсія, фітотоксичність, компости, біотестування.

**Problem Statement.** Natural gas extraction is accompanied by the formation of massive volumes of polluted solid and pulp wastes. Drilling fluids and cuttings are classified as high-hazard wastes for the environment [1]. The main pollutants are the drilling fluid chemicals, heavy metals and radionuclides [2, 3].

Methods of such wastes unitization could be classified basing on the action of toxic compound neutralization or separation: physical, physic-chemical, chemical,

or biochemical, or their combinations [4-6]. It seems that nowadays the most popular method of gas drilling waste utilization is solidification. This technology involves mixing the sludge with cement and other fillers. The final solid mass is used for obtaining building materials [1].

**Actuality of research.** Currently, in Ukraine, the sludge wastes from the gas production industry are implemented in the cheapest, but the most dangerous way for the environment – by the burial on sludge map-

sor under the guise of reclamation of soil areas, i.e., introduction into the topsoil layer [7]. Obviously, such a waste self-purification process will be so slow that it will take decades or even more. The construction project for the processing and utilization of drilling waste for construction materials was developed in 2004-2005 during the active development of the Yabluniv gas field (Poltava region). But it remained at the level of the pilot plant. So, the attempt to accelerate gas drill cuttings neutralization by using environmental-friendly technology of bioconversion is a potentially effective and relatively cheap solution [1].

**The goal of the research is** (i) to assess the efficiency of gas drilling wastes bioconversion; (ii) the possibility to obtain ecologically safe mineral-organic substrates as a final product of bio-neutralization, and (iii) evaluation of their phytotoxicity. We proceeded from the fact that the project for the utilization of the studied drill sludge involved the use of the biological decomposition of organic compounds to accelerate its neutralization. To do this, it was supposed to introduce phosphogypsum (PG) (in the amount of 2...3 % by weight), straw (1...2 % by weight), and organic substrate (3...5 % by weight) into the sludge pits [8]. Based on the proposed composting composition, we have compiled a matrix for studying of bioconversion process.

Biotesting is the most useful approach for the assessment of natural or artificial substrates [9]. Lettuce, mustard, barley, oat, and wheat plants are considered as the most sensitive objects for phytotoxicity assessment of contaminated soils [10].

## 2 Materials and methods

### 2.1 Initial samples and their pre-treatment

Initial samples of drilling wastes were collected from the well near the village Antonovka, Pyryatinsky dis-

trict, Chernigiv Oblast. The research was initiated by the owner of this drill gas well – PoltavaGasVydobuvannya Gas Production Division, the subdivision of Naftogaz Corporation.

According to ecological passport of the object, the estimated mass of drilling waste from one well is about 3,000 tons. The composition of drilling waste, in addition to ecologically safe silicates for the soil (bentonite clay, polygorskite clay), may contain ingredients mainly with III-IV hazard classes according to oriented permissible concentrations (OPC) in soil (Table 1).

In addition, excepting the declared identified inorganic and organic chemicals in the composition, the drilling wasters contain chemically unidentified components of special purpose. They have been presented as Trade mark labels (i.e., PAC-LV, PAC-HV, CMC-LV, CMC-HV, PAG-KM, etc.) or as descriptions of their role in the technological process (for example, lubricant additive for drilling solution). Their safety is guaranteed by the conclusion of the state sanitary and hygienic examination only.

Since we will attempt to utilize the dry residue of the drilling pulp after the filtering pool of LLC “Eko TecOIL” (Fig. 1), its composition according to the following indicators is also important (Table 2). According to TU U 08.9-40371287-002:2018 Mixture of minerals for arranging ECOTEC-GEO+ territories, drilling waste was treated by undefined pH regulating agents, flocculants, and coagulants, but environmentally-friendly and safe. It is known the relative volume ratios of these chemicals and treating wastes.

Based on the previously experience [13], the composition of mixture for the bioconversion of drilling sludge includes shredded wheat straw, cattle bedding manure; sapric peat. The mass ratio of the components was cal-

Table 1  
Chemicals in the composition of drilling wastes, according to ecological passport of drill well

Reagent	Hazard class according to DSTU-N B A.3.2-1:2007 [11]	OPC in soil, mg/kg according to GSTU 41-00 032 626-00-007-97 [12]
Graphite C	IV	5000
Sodium carboxymethyl cellulose $[C_6H_7O_2(OH)_{3-x}(OCH_2COOH)_x]_n$	III	3000
Polyacrylamide flocculant $(C_3H_5NO)_n$	IV	400
Oil	III	4000
Potassium chloride KCl	III	560
Sodium chloride NaCl	III	2500
KSSB-2M – condensed sulphite – alcohol the bard	-	2000
Calcined soda of technical grade B (powdery) $Na_2CO_3$	-	200
Chromium Cr	III	6,0
Slaked lime (calcium hydroxide) $Ca(OH)_2$	IV	8000
Caustic soda (sodium hydroxide) NaOH	II	2000
Barite (Barium sulphate) $BaSO_4$	IV	5000

culated based on the optimal proportion of Nitrogen and Phosphorus (in dry state), which is equal to 1 : 20...25 for the bio-fermentation of the substrate in aerobic conditions [13, 14]. Initialization of the biochemical transformation of compost mass was accelerated by microbial active composition “EXTRACON”®. This activator is a natural soil microbial consortium [15].

## 2.2 Composition and treatment of the composting mixtures

The composition of the examined mixtures containing drill wastes and additives are presented in Table 3.

Compost mixes prepared by thorough homogenization mechanical mixing of 1,5 kg of total weight were placed in polyethylene containers with a lid with a volume of 10 L. Mixing the components of the composts; their hydration was brought up to 60% by adding the calculated amount of water [13]. The substrates were

mixed once every three days throughout the experiment, maintaining a given level of moisture.

## 2.3 Control of the composting process parameters

The progress of the composting process was monitored by temperature (measured remotely with a pyrometer) and pH (in a 10:1 aqueous suspension using Ecotest 2000).

## 2.4 Composition and properties of the final products – composts containing drilling wastes

The composition of the prepared composts was assessed by the main nutrient content measuring (N, P, K), the concentration of a few heavy metals (Pb, Cd), and testing their phytotoxicity. The content of total nitrogen was determined by the Kjeldahl method, Phosphorus – photometric determination as phosphomolybdenum complex, Potassium – using method of flame photometry method [16]. Lead Pb and Cadmium Cd

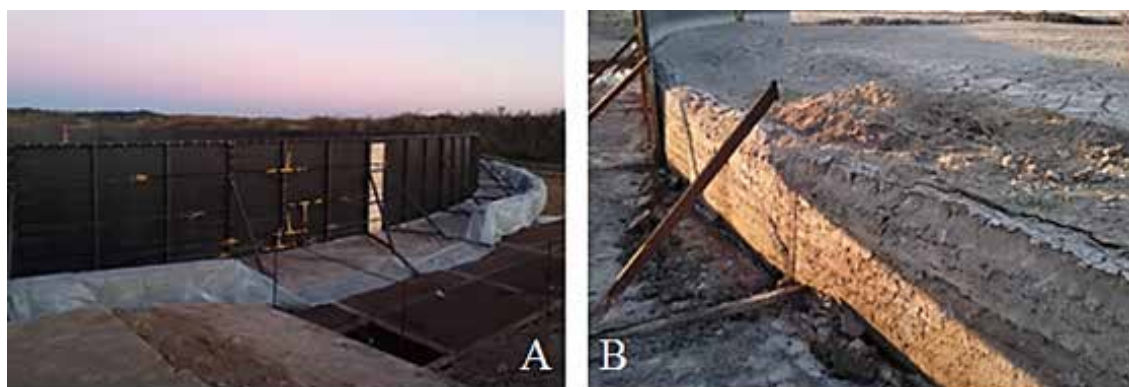


Figure 1. The general view of open air filter pool for drilling wastes filtrating (A) and the dry residue after liquid phase removal (B)

Table 2

## Content of heavy metals and generals parameters of drilling waste dried drilling sludge

Parameters	Content				
	Pb	Ni	Cr	Cd	Zn
Trace metals, mg/kg	1,27	0,16	1,59	0,58	12,99
Technological properties/ composition	a pH	Humidity, wt. %	Organic part, wt. %	Mineral part, wt. %	SiO <sub>2</sub> content, g/kg
	8,83	7,9	8,0	92,0	370

Table 3

## he composition of examining compost series containing drill wastes

# of variant	Component contribution, wt. % (at natural humidity)					C:N ratio
	Dried drill waste	Shredded wheat straw	Cattle bedding manure	Sapric peat	“EXTRACON”®	
1 (control)	-	25	50	20	-	1:18
2	10	30	60	-	-	1:17
3	10		60	30	-	1:18
4	20	30	50	-	-	1:18
5	10	30	60	-	+	1:17
6	10		60	30	+	1:17

concentrations were measured by the inversion chronopotentiometry method [17].

The phytotoxicity of the obtained substrates was determined in a laboratory experiment according to the method of contaminated soil phytotoxicity determination [18] using wheat (*Triticum spp.*) as a test object. Ten visually healthy wheat seeds were planted directly on substrate in Petri dishes. The recommended optimal amount of air-dry, and moistened to 33,3% substrate brought to a homogeneous consistency was 20 g. For grain germination, it was not pre-soaked and cultivated in closed Petri dishes in the absence of light in a thermostat at +24 °C. After 14 days, the number of germinated seeds was counted, the length of roots and shoots was measured, and the phytotoxic effect was calculated. The experiment was set in 3 replications.

The phytotoxic effect (FE, %) as the inhibition level of growing processes was expressed as Eq. 1 [19]:

$$FE = \frac{L_0 - L_x}{L_0} \times 100, \% \quad (1)$$

where  $L_0$  – average root length of a plant grown on control medium;

$L_x$  – average root length of a plant grown on examining medium at the action of predicting toxic factor.

### Results and Discussion

The process of bioconversion of organic substances at composting can occur both in anaerobic and aerobic conditions. They are accompanied by changes in the temperature and the pH of the reaction mixture. In artificial conditions, aerobic decomposition of the organic substrate by saprophytic aerobic microflora demonstrates the highest efficiency compared with anaerobic ones. At the aerobic decomposition of organic components, the energy output is at least 20 times higher than the exothermic effect of the anaerobic process at the same substrate. The overall thermochemical equations of the specified reactions are as follows [13]:

– In aerobic condition:  $C_6H_{12}O_6 + O_2 + H_2O \rightarrow 6CO_2 + 12H_2O + 38ATP$ : Energetic output: 1 mol ATP corresponds to 40...60 kJ: so, 38 mol  $\times$  50 KJ/mol (average) = 1900 kJ or 456 kcal;

– In anaerobic condition:  $C_6H_{12}O_6 \rightarrow 2C_2H_5OH + 2CO_2 + 2ATP$ : Energetic output: 1 mol ATP corresponds to 40...60 kJ. So, 2 mol ATP  $\times$  50 KJ/mol (average) = 100 kJ or 25 kcal.

The first step of composting starts at environmental temperature (20...25 °C), when wild microbiota presenting in the initial compost mixture (or with added microbial active consortium "EXTRACON"® in two variants) has begun to reproduce actively. After 12-20 days, it was recorded that the temperature increased up +40 °C. This means that the mesophilic stage has been passed. This stage was accompanied by a change in pH value. As a result of the acidification of reacting mixture, the pH has dropped by 0.6...1.5 units in variants the 2<sup>d</sup>, 3<sup>d</sup>, 4<sup>th</sup>, and 5<sup>th</sup>; by 0.3-0.4 pH units – in variants 1<sup>st</sup> and 6<sup>th</sup>.

A few days later, the temperature continued to rise to +65 °C. This means the beginning of the thermophilic stage when the mesophilic microorganisms died. After reaching the temperature maximum, a decrease in the number of fungi (cellulose and lignin destructors) was observed.

At this stage of composting, bacillary forms of microorganisms dominate and the fastest decomposition occurs for sugars, starches, fats, proteins, and then more complex compounds. The decomposition process is accompanied by gaseous ammonia, methane and carbon dioxide releases. The substrate medium stabilized at a slightly alkaline level for variants the 2<sup>d</sup>, 3<sup>d</sup>, 4<sup>th</sup>, 5<sup>th</sup>, and 6<sup>th</sup> and in the control, variant 1<sup>st</sup> had a pH below 7. This stage lasted about 10 days. In the next stage (decay stage), the temperature is reduced to the ambient level. The pH of the compost mass is also slightly reduced. Fungi and actinomycetes actively decomposed polysaccharides, hemicellulose, and cellulose to monosaccharides. At the final stage (maturation phase), the processes of transformation of lignin and proteins of dead microorganisms took place, the end products of which are humic acids. This stage is the longest. In the described laboratory experiment, it was about 30 days.

It was visually established that the most complete bioconversion of the reaction mixture was observed in the 3<sup>d</sup> and 6<sup>th</sup> variants, which in their composition contain 60 wt. % cattle manure, 30 % wt. peat and 10 % wt. drilling waste. In addition, "EXTRACON"® accelerated composting process in 6<sup>th</sup> variant. Since these options didn't contain straw, which is a natural complex polymer, decomposition and composting processes were faster and more intensive. Fig. 2 shows the view of the reaction mixtures 60 days after the start of the experiment.



Figure 2. The view of composted mixture series after 60-day bioconversion (numbers are corresponded to variants in Table 3)

The temperature in the reactors stabilized and kept at 25 °C. In some cases, mold fungi began to actively develop on the compost surface due to the presence of nutritive organic substances and sufficient moisture.

Data on the content of nutrients in composts are presented in Fig. 3. It was found that the compost mixtures

of the 2<sup>d</sup>, 3<sup>d</sup>, 5<sup>th</sup>, and 6<sup>th</sup> variants contained Potassium and Phosphorus 1.5...2 times more than the original manure. Apparently, this is due to Potassium influx with drilling waste, and the increase in Phosphorus content is the result of microbiological bioconversion of organic additives.

In the 4<sup>th</sup> variant, where the content of drilling waste increased to 20 %, a relative decrease in Nitrogen and Potassium content was observed. It could be due to the toxic effect of drilling waste on the microbiota.

Analysis of Lead and Cadmium content in composts (Table 4) shows that the slight excess of Cadmium permitted level was observed in the 4<sup>th</sup>, 5<sup>th</sup>, and 6<sup>th</sup> variants. But potentially it could be led to its accumulation in root tissues, causing their damage and inhibiting the germinating. Lead content in all variants didn't exceed permitted concentrations.

For the evaluation of potential possible toxicity of composts contained drilling wastes, the next stage of this research was the aim to assess their phytotoxicity to seedlings of common wheat *Triticum aestivum* L. Results are presented in Fig. 4 and Table 5.

According to data presented in Table 5, a positive dynamics of germination was observed in the 2d version of the compost mixture only, compared to the control

one. The rest of the compost variants, containing drilling waste, tend to suppress seed germination, which may be evidence of the presence of undefined wheat germination inhibitors in the substrates.

For a more complete understanding of the phytotoxic effect, the length of the root and shoot was measured for each germinated seed. After measuring, the phytotoxic effect was determined according to Eq. 1. The results of the FE calculation are shown in Table 6.

Assessment of phytotoxicity of composts shows the negative impact of drilling waste presented in compost composition. In particular, growth processes of root formation of wheat are inhibited, although in general, the level of toxicity is relatively weak. The second version of the compost substrate, which included manure, straw, and drilling waste, despite low phytotoxicity, may be quite promising for use in agriculture, for example, for fertilization of low-sensitive technical crops.

At the same time, the use of obtained biocomposts as bioinorganic fertilizers is regulated by the content of toxic components, such as heavy metals Pb and Cd. The regulation of the content of heavy metals in fertilizer is based on the maximum allowable concentrations of metals in the soil. Calculation of the theoretically permissible norms of the obtained biocomposts accord-

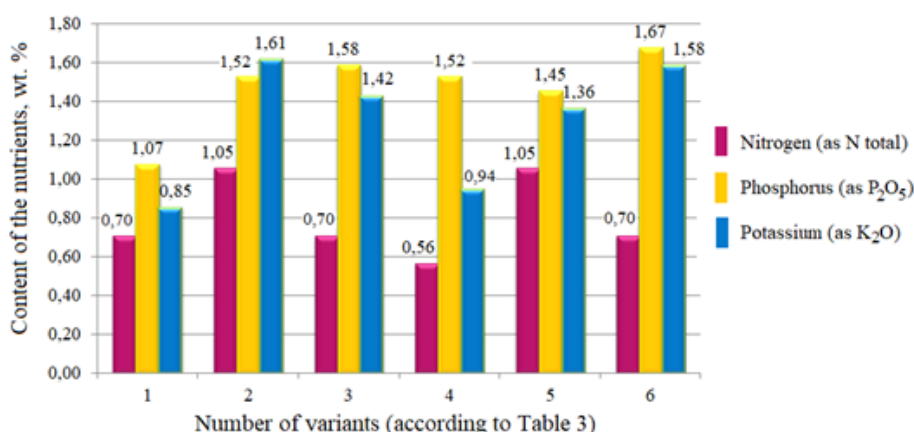


Figure 3. Content of nutrients in composts

Table 4

Content of heavy metals in examined composts

# of variant	Concentration, mg/kg	
	Lead Pb	Cadmium Cd
1 (control)	0,34 ± 0,07	0,24 ± 0,05
2	1,44 ± 0,21	0,24 ± 0,04
3	1,76 ± 0,30	0,51 ± 0,10
4	2,26 ± 0,34	0,75 ± 0,11
5	1,76 ± 0,30	0,90 ± 0,14
6	2,67 ± 0,40	0,83 ± 0,13
The maximum permitted level (MPL) in soils, mg/kg	32 mg/kg (total) 6 mg/kg (exchangeable or moving form) [20] 100 mg/kg [21]	6 mg/kg (total) 0,7 mg/kg (exchangeable or moving form) [20] 34 mg/kg [21, residential area]



ing to the content of Pb and Cd in them is carried out according to Eq. 2 [13]:

$$D_{HM} = \frac{(0,8 \times MPL - B) \times 3000}{C_{HM}} \quad (2)$$

where  $D_{BM}$  – theoretically permissible amount of compost, t dry wt. /ha;

MPL – maximum permitted level of heavy metal content in soil, mg/kg of soil (See Table 4);

B – background heavy metal content in soil, mg/kg;

$C_{HM}$  – content of a heavy metal in compost, mg/kg of soil dry wt.;

3000 – the dry weight of the arable soil layer, t/ha.

The results of such calculations for conditional soil with the content of Pb 20 mg/kg and Cd 0.4 mg/kg, where the obtained biocomposts would be applied, are shown in Table 7.

It is known that in order to maintain a deficit-free humus balance in soils, the need for organic fertilizers is 10-18 t/ha [13]. When processing the estimated mass of 3000 tons of drilling waste from one well into biocomposts according to the recipe of the 2<sup>d</sup> and 3<sup>d</sup> variants, 30,000 tons of compost mass can be obtained. Another factor that may limit the nutrient content of

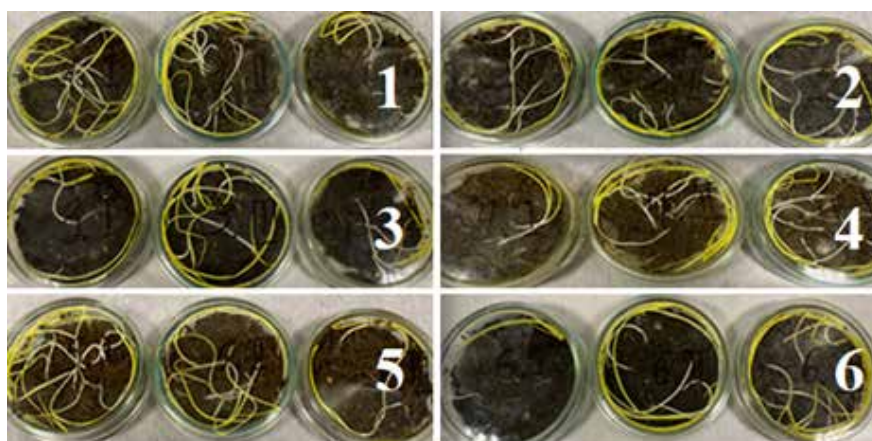


Figure 4. The view of test plants grown on examined composts (the 1<sup>st</sup> variant – control).  
Numbers of variants – as in Table 3

Table 5

The number of germinated grains on the variants of compost mixtures

# of variant	Replication			Average
	The 1 <sup>st</sup>	The 2 <sup>d</sup>	The 3 <sup>d</sup>	
1 (control)	8	10	3	7,0
2	7	10	7	8,0
3	3	3	5	3,7
4	2	6	8	5,3
5	9	6	3	6,0
6	1	5	9	5,0

Table 6

Variability in seed germination and phytotoxicity assessment of the substrates

# of variant	The average quantity of germinated seeds	The level of the growth process inhibition or phytotoxicity (FE, %)	Phytotoxicity relative assessment (verbal description)
1	7,0	0	Control
2	8,0	12	Weak
3	3,7	32	Moderate
4	5,3	Minus 19	No symptoms; results were better than control – stimulation effect
5	6,0	Minus 11	No symptoms; results were better than control – stimulation effect
6	5,0	6	Weak

Table 7

**Calculation of the theoretically permissible quantity of compost (tons/ha)  
based on the content of Pb and Cd**

# of variant	Calculation based on Lead Pb content in compost		Calculation based in Cadmium Cd content in compost	
	Pb, mg/kg of compost	D <sub>Pb</sub> , tons/ha	Cd, mg/kg of compost	D <sub>Cd</sub> , tons/ha
Soil (background)	20,0		0,40	
1	0,34	49411	0,24	25000
2	1,44	11666	0,24	25000
3	1,76	9545	0,51	11754
4	2,26	7433	0,75	8000
5	1,76	9545	0,90	6666
6	2,67	6296	0,83	7229

composts is their total nitrogen content. It is not allowed to apply total nitrogen with organic fertilizers over 300 kg/ha, including mineral nitrogen, which exceeds the annual yield of the crop under which the fertilizer is applied [22].

According to this regulation, the biocompost application prepared as in the 2<sup>d</sup> and 3<sup>d</sup> variants, is limited to 30 and 40 tons per hectare, respectively. From this, it can be calculated that the application of 30000 tons of compost mass on an area of 1000 ha will practically not change the natural background of the soil in terms of the content of Pb and Cd.

### Conclusion

Finally, it was shown the principle possibility to convert drilling wastes to environmentally friendly bioinorganic substrates using their microbiological treatment in the bioconversion process in the mixture containing dried drill waste, wheat straw, cattle bedding manure, and sapric peat in different proportions. We tried to accelerate the organic matter transformation in composting mixture by addition of microbiological community "EXTRACON"<sup>®</sup>.

It has been established that the most effective formulations for bioconversion processing contain 10 % drill cuttings, and the remaining organic components

(manure, straw, peat as sources of nitrogen and phosphorus). The limiting factors for the duration of the composting process are temperature and humidity. An increase in the content of drill cuttings up to 20 % in the mixture slows down the bioconversion process.

The negative impact of drilling waste in the composition of the tested organo-mineral compositions of biocomposts was revealed by laboratory studies of phytotoxicity for wheat seedlings. The inhibition of growth processes of root formation of wheat was revealed, although in general the level of phytotoxicity is relatively weak. The variant of the compost substrate, including manure, straw, and drilling waste, despite its weak phytotoxicity, can be quite promising for use in agriculture, for example, for fertilizing industrial crops with low demands. The limiting factors for the use of the resulting biocomposts as bioinorganic fertilizers were calculated in terms of the content of toxic components of heavy metals Pb and Cd and the norms of total nitrogen. It can be assumed that the processing of 3,000 tons of drilling waste from one well for 30,000 tons of compost mass and its application as a bioinorganic fertilizer at the rate of 30 tons/ha is not only environmentally friendly, but can also provide a fairly high level of plant nutrition.

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