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Research paper

Assessment of the possibility of using the method of sludge-lignin dehydration of the Baikal Pulp and Paper Mill by the freeze-thaw method

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Abstract

An estimation of the possibility of dehydration of sludge-lignin of the Baikal Pulp and Paper Mill by the freeze-thaw method was carried out. Change of sludge-lignin structure, physicochemical properties and chemical composition after freeze-thaw are shown. Toxicity and chemical composition of the water solution separated from the sludge-lignin during its freeze-thaw is estimated. An increase in the rate of germination of seeds of the cress in the soil with the addition of the frozen-out sludge-lignin in the ratio "sludge-lignin-soil" 1:3-1:4 is shown. Possibility of application and recommendations for the use of freezed out sludge-lignin as an organic fertilizer is considered. Principal possibility of the worm transformation of sludge-lignin, previously dehydrated by the freeze-thaw method, by red Californian worms Eisenia foetida Andrei Bouche (1972) is shown. The issue of prospects for processing this waste in a fertile bio-soil is discussed. The latter can later be used for recultivation of the territory of the sludge accumulator plots of the Baikal Pulp and Paper Mill.

Keywords: *sludge-lignin, Baikal pulp and paper mill, freeze-thawing, sludge dewatering, worm transformation of sludge-lignin, organic fertilizer, bio-soil.*

1. Introduction

The amount of sludge-lignin accumulated in the maps of the sludge accumulators of the Baikal Pulp and Paper Mill is more than 6 million cubic meters. It is a gel-like product with a water content of up to 90 %. The basis of sludge-lignin is slightly soluble substances. They were formed at the stages of chemical and biological treatment of wastewater from the Baikal Pulp and Paper Mill. The danger of this waste, in particular, is the formation of toxic, foul-smelling gases: hydrogen sulphide, methyl mercaptan, and explosive methane in the process of storing it. There are also organochlorine compounds and phenols [30, 32]. One of the stages of neutralization of sludge-lignin waste is freezing technology with subsequent thawing. It allows to relieve sludge-lignin from a significant volume of water [2]. The composition of sludge-lignin waste, depending on the different technologies for pulp processing, is significantly different. Therefore, it was of interest to verify the possibility of using this technology on the example of sludge-lignin waste of the Baikal Pulp and Paper Mill.

2. Materials and methods of research

Sampling for laboratory studies was conducted in February 2017 from plot No. 2 of the Baikal Pulp and Paper Mill. In the modeling of sludge-lignin freezing processes, a sample weighing 2500 g was placed in a plastic container at 18 °C for 24 hours. After that, the sample was thawed at a temperature of +19 °C. Drying after preliminary freezing-thawing of lignin slurry was carried out in air at room temperature.

The toxicological parameters of the sludge-lignin samples and the water released from them were evaluated by methods of biotesting. The bioassays recommended for state ecological control were used: according to the survival rate of Daphnia magna [28], inhibition of luminescence of the bacterial test system "Ecolum" [27]. Toxicometric studies were conducted in the Accredited Laboratory of Water Toxicology of the ISU (Accreditation Certificate No. ROSS RU.0001.21AG02 issued on August 1, 2014).

Determination of the chemical and physicochemical parameters of the investigated samples of sludge-lignin and water over the sludge was carried out in the Accredited Test Laboratory of the Center for Agrochemical Service "Irkutsk". Samples were analyzed for the content of heavy metals in them according to the methods described in [12, 17]. The arsenic content in sludge-lignin was estimated by the photometric method [16]. The amount of bound water was determined by [4], and the hydrogen index by [9]. The mass fraction of total nitrogen was analyzed according to [6], phosphorus – according to [7], potassium – [8], total content in samples of organic matter – according to [5], ash – [10].

In parallel, the physicochemical characteristics of an aqueous solution that separated from lignin sludge after its freezing-thawing were investigated. Thus, the pH was determined [22], the amount of suspended matter [20], COD [11], electrical conductivity [15], the concentration of phenols [24], petroleum products [23],

heavy metals (copper, cadmium, nickel, zinc, chromium, lead, mercury) and arsenic [26, 25, 21].
The total microbial number of water samples was determined by the Koch method. The number of cells of Escherichia coli bacteria was found by inoculation on Endo's medium [14, 31].
The effect of the product-lignin sludge formed during dehydration on the germination of the cress-salad seeds was evaluated. For this, the frozen lignin slurry was mixed with gray podzolic soil in the ratios 1:1, 1:3, 1:4. In order to control, soil without the addition of sludge-lignin was used. In addition, the germination capacity of seeds in the fumigated and original sludge-lignin without adding soil was investigated.
The processes of the worm transformation of sludge-lignin, previously dehydrated by freezing, were studied. After thawing, the precipitate released from the water was transferred to clear plastic containers. There were also placed 15 specimens of the red Californian worm *Eisenia foetida* Andrei Bouche (1972). Capacities were incubated for a month in a dark room with a constant humidity and a temperature of 20±2 °C. Periodically, counted formed coprolits, cocoons and young individuals. All experiments were carried out in at least 5 independent experiments with 3-6 parallel measurements in each. For statistical processing of the received data, the Microsoft Excel software package was used. The conclusions are made with the probability of an error-free forecast P ≥ 0.95. The reliability of the differences in the results was determined using the Student's test.

3. Results of the study

The data of the conducted studies of the chemical composition of the sludge-lignin samples are presented in Table 1. Comparison of the results of the analysis with the standards required in accordance with GOST 54651-2011 [13] did not reveal an excess of the MPC of the components under consideration. Thus, the content of copper in sludge-lignin was 30% of the established standard, lead – 10%, zinc – 25%, cadmium – 23%, nickel – 13%, chromium – 27%, mercury – 87%, arsenic – 61%. At the same time, lignin sludge had a strong specific unpleasant odor due to the presence of phenolic compounds, methyl mercaptan, hydrogen sulphide and other sulfur-containing compounds.

Table 1: The results of chemical analysis of samples of sludge lignin selected from plot No. 2 of the Baikal Pulp and Paper Mill

Test parameter, unit of measure	Indicator value	Standards of indicators in accordance with [13]
Mass fraction of moisture,% ± Δ	72.00±0.90	-
pH, ± Δ	5.90±0.30	-
Mass fraction of total nitrogen,% ± Δ	2.50±0.20	-
Mass fraction of total phosphorus,% ± Δ	2.10±0.20	-
Mass fraction of total potassium,% ± Δ	0.05±0.03	-
Mass fraction of ash,% ± Δ	23.69±0.80	-
Mass fraction of organic matter,% ± Δ	38.20±0.50	-
Copper (acid-soluble form), mg / kg, ± Δ	43.10±9.00	132.0
Lead (acid-soluble form), mg / kg, ± Δ	9.70±3.40	130.0
Zinc (acid-soluble form), mg / kg, ± Δ	54.70±11.50	220.0
Cadmium (acid-soluble form), mg / kg, ± Δ	0.45±0.16	2.0
Nickel (acid-soluble form), mg / kg, ± Δ	10.30±3.60	80.0
Chromium (acid-soluble form), mg / kg, ± Δ	24.11±8.40	90.0
Mercury, mg / kg, ± Δ	1.83±0.33	2.1
Arsenic, mg / kg, ± Δ	1.22±0.31	2.0

Note: the data are submitted by the testing laboratory of the Center for Agrochemical Service "Irkutsk", test report No. 515 of December 23, 2016. Responsible performers E.P. Tatarinova, E.N. Khoroshikh, V.V. Yakimova, N.V. Velyako, T.S. Moskvitina.

The mass fraction of moisture in the investigated sample of sludge-lignin was 72.0±0.9%. It had a homogeneous, gel-like consistency (Figure 1). After thawing the sludge, a considerable amount of the free solution (1200 ml) was easily separated. As a result, the mass of the test sample decreased 2.1 times from the initial value. A considerable change in the structure of sludge-lignin was observed. From a gel-like mass, it was converted into a structured product in a certain way. Under the microscope, individual fibers were clearly visible (Fig. 2).

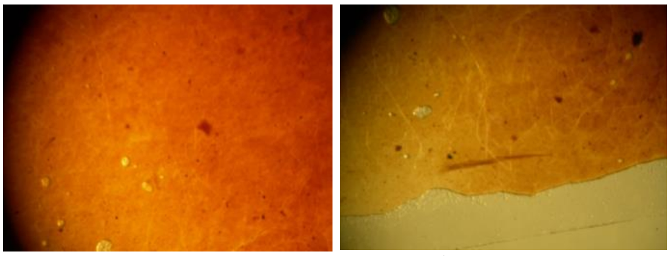

Fig. 1: Sludge-lignin under a microscope (sample from map No. 2, microscope Micromed 3 var. 3-20, magnification 200 times)

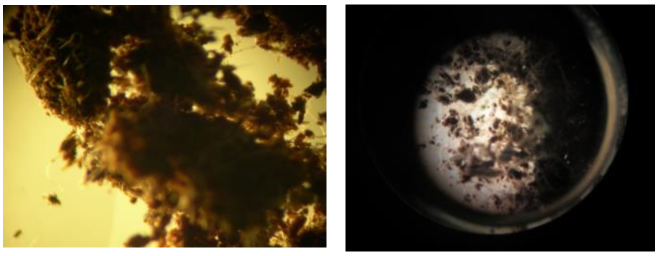

Fig. 2: Sludge-lignin after freezing and thawing under a microscope (sample from plot No. 2, microscope Micromed 3 var. 3-20, magnification 200 times)

Drying after preliminary freezing-thawing of sludge-lignin resulted in the formation of a dry fibrous product. Its mass was 10 times less than the original sludge-lignin (Table 2).

Table 2: Physico-chemical parameters of the sludge-lignin of the Baikal Pulp-and-Paper Mill (plot No. 2) after its dehydration by the freeze-thaw method

Weight of sludge-lignin (initial), g.	2500
Weight of sludge-lignin after removal of water during thawing (wet), g.	1190
Weight of sludge-lignin after removal of water during thawing (dry), g.	230
The amount of aqueous solution released, ml	1200
Weight of suspended solids in water, g	4,9
Total microbial number in the separated water, CFU/ml	< 100
The number of sanitary-indicative microorganisms in the separated water, CFU/ml	Not detected

The results of the chemical, microbiological and toxicological analysis of the aqueous solution separated from the sludge-lignin during its dehydration of the freeze-thaw methods are given in Table 3.
The total microbial number in the samples of the aqueous solution that separated after freezing-thawing of the lignin sludge was not

more than 100 cfu/ml. Sanitation-indicative microorganisms (bacteria of *Escherichia coli* group) were practically absent in the water samples under study. The toximetry of the tested samples by 2 methods of biotesting showed their harmlessness for the analyzed test objects. So, harmless dilution multiplicity HDM_{10-96} for Daphnia magna and $HDM_{20-0.5}$ for luminous bacteria "Ecolum" were equal to 1.

The excess of heavy metals in samples of water released from sludge-lignin during its freeze-thawing was not revealed. But the samples showed a large number of phenols – 1.31±0.29 mg/dm^3. This is 1000 times higher than the standards (GOST R 54651-2011). In addition, in the test samples, an increased (0.693±0.243 mg/dm^3) content of oil products was detected. This exceeds the normative index by 2.3 times (Table 3).

Table 3: The results of the chemical and physico-chemical analysis of the aqueous solution released from the sludge-lignin of plot No. 2 of the Baikal Pulp and Paper Mill during its freezing-thawing

Test parameter, unit of measure	Indicator value	Standards of indicators in accordance with [13]
pH, ± Δ	5.5±0.2	6-9
Suspended substances, mg / dm^3, ± Δ	14.0±3.0	0.75
Total stiffness, ± Δ	2.0±0.3	7
Dry residue, mg/dm^3, ± Δ	433.0±39.0	1000
COD, mgO$_2$/dm^3, ± Δ	515.0±72.0	30
Phenols, mg/dm^3, ± Δ	1.31±0.29	0.001
Conductivity, mS/cm	0.033	1.0
Copper, mg/dm^3, ± Δ	0.004±0.002	1.0
Cadmium, mg/dm^3, ± Δ	<0.001	0.001
Nickel, mg/dm^3, ± Δ	0.012±0.004	0.02
Zinc, mg/dm^3, ± Δ	0.015±0.005	1.0
Chromium, mg/dm^3, ± Δ	<0.005	0.05
Lead, mg/dm^3, ± Δ	0.005±0.002	0.01
Arsenic, mg/dm^3, ± Δ	0.002±0.001	0.01
Mercury, mg/dm^3, ± Δ	0.00011±0.00005	0.0005
Petroleum products, mg/dm^3, ± Δ	0.693±0.243	0.3

Note: the data are submitted by the testing laboratory of the Center for Agrochemical Service "Irkutsk", test report No. 79 dated March 17, 2017. Responsible executors E.P. Tatarinova, V.V. Yakimova, N.V. Velyako, G.P. Skornyakova.

The chemical analysis of the product, which was formed as a result of sludge lignin dehydration, showed that it corresponds to the norms for groups of fertilizers of group I in practically all the investigated parameters (Table 4). This group includes fertilizers based on sewage sludge, used for the cultivation of technical, fodder, grain and sideral crops. Accordingly, the normative documents they can be applied and in the personal subsidiary farm when growing seedlings of vegetable and flower crops [13].

Table 4: The results of chemical and physicochemical analysis of samples of lignin, selected from plot No. 2 of the Baikal Pulp and Paper Mill, after its dehydration by freeze-thawing and drying

Test parameter, unit of measure	Indicator value	Standards of indicators in accordance with [13]	
		Norm for fertilizer groups I *	Norm for fertilizer groups II**
Mass fraction of moisture, % ±Δ	14.2±0.3	Not more than 70	
pH, ±Δ	5.6±0.3	6.0 – 8.0	
Mass fraction of total nitrogen, % ±Δ	3.05±0.3	Not less than 0.6	
Mass fraction of total phosphorus, % ±Δ	1.73±0.1	Not less than 0.7	
Mass fraction of total potassium, % ±Δ	<0.03	Not less than 0.1	
Mass fraction of ash, % ±Δ	17.9±0.4	-	
Mass fraction of organic matter, % ±Δ	41.1±0.8	Not less than 30.0	
Copper (acid-soluble form), mg/kg, ±Δ	28.8±6.0	132.0	750.0
Lead (acid-soluble form), mg/kg, ±Δ	8.0±2.8	130.0	250.0
Zinc (acid-soluble form), mg / kg, ± Δ	56.2±11.8	220.0	1750.0
Cadmium (acid-soluble form), mg/kg, ±Δ	0.51±0.18	2.0	15.0
Nickel (acid-soluble form), mg/kg, ±Δ	8.6±3.0	80.0	200.0
Chromium (acid-soluble form), mg/kg, ±Δ	9.8±3.4	90.0	500.0
Mercury, mg/kg, ±Δ	0.9594±0.2878	2.1	7.5
Arsenic, mg/kg, ±Δ	0.89±0.22	2.0	10.0

Note: the data are submitted by the testing laboratory of the Center for Agrochemical Service "Irkutsk", test report No. 77 dated March 13, 2017. Responsible executors E.P. Tatarinova, E.N. Khoroshikh, V.V. Yakimova, N.V. Veelyko

* Group I fertilizers: fertilizers based on sewage sludge, used for cultivation of technical, fodder, grain and sideral crops, in personal plots for growing seedlings of vegetable and flower crops.

** Group II fertilizers: fertilizers based on sewage sludge, used for planting forestry crops along roads, in nurseries for forest and ornamental crops, floriculture, for cultivating depleted soils, for reclamation of disturbed lands and for slopes of highways, for reclamation of landfills for solid household waste.

Experiments have shown that the most optimal for the germination of seeds cress-salad is the ratio of "sludge-lignin after freezing-thawing + soil" 1:3, 1:4. The mixture of fumigated lignin slurry with soil in a ratio of 1:1, as well as the fumed lignine itself, proved to be of little use for germination of seeds. In these substrates the seed germination was much lower than in the control variant (soil). The lowest samples of seed germination differed in the sample with the original sludge lignin (Fig. 3, 4, 5).

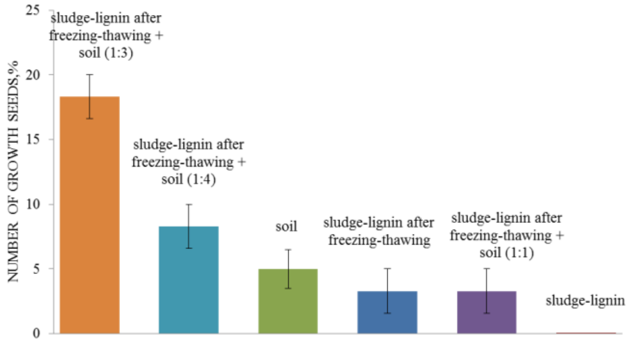

Fig. 3: Germination of seeds cress-salad in samples with sludge-lignin and soil in various ratios (after 1 day)

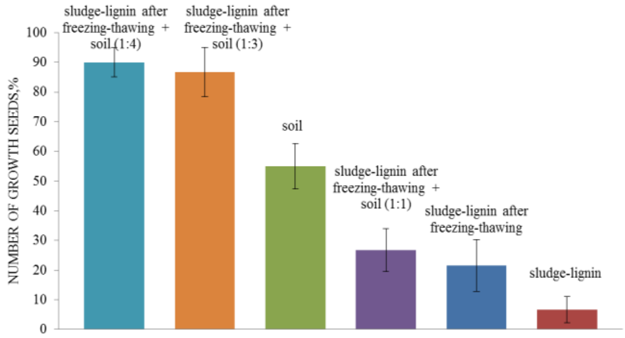

Fig. 4: Germination of seeds cress-salad in samples with sludge-lignin and soil in various ratios (after 2 day)

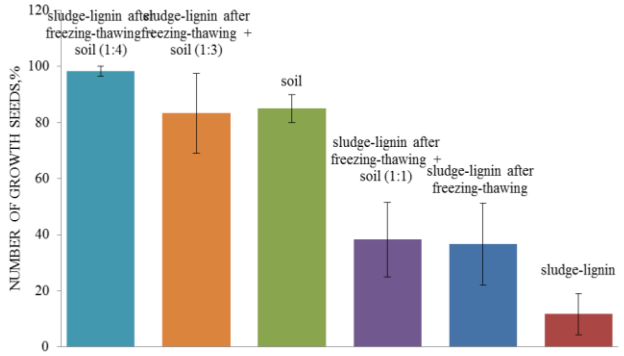

Fig. 5: Germination of seeds cress-salad in samples with sludge-lignin and soil in various ratios (after 3 day)

Worm transformation is one of the most environmentally friendly ways of processing various wastes [3, 18, 19]. As a result of vital activity of earthworms, many of the wastes processed by them turn into fertile soil. Experiments have shown the possibility of the worm transformation of sludge-lignin, previously dehydrated by the freeze-thaw method, by red California worms. Within 1 month of the experiment, worms placed in pre-dehydrated freeze-thawing sludge-lignin, multiply well enough. The total number of worms increased by 30% of the original. In addition, a large number of cocoons were found. In the transformed substrate, a considerable number of products of sludge-lignin processing were observed by worms-coprolites (Table 5). On the other hand, in non-frozen lignin sludge, worms died within 1-2 days after their introduction.

Table 5: Worm transformation of the sludge-lignin of the Baikal Pulp and Paper Plant, previously dehydrated by defrosting-thawing, with red Californian worms E. foetida Andrei Bouche (1972)

Incubation time	Number of co-coons	Coprolits	Survival of worms,%	Growth,% of the initial number of individuals
0 days	no	-	100	no
7 days	no	-	100	no
13 days	no	+	100	no
17 days	*	++	100	no
23 days	*	++++	100	7
30 days	***	++++	100	30

Note: * small cocoons; *** numerous cocoons; - coprolites are absent; + isolated coprolites; ++ small coprolites; ++++ abundant formation of coprolites.

The discussion of the results

The results of laboratory studies, presented in the paper, proved the possibility of successful and effective application of the freezing-thawing method for neutralizing waste from the Baikal Pulp and Paper Mill. The conclusion is confirmed by the pilot-industrial tests carried out in 2005-2006. [29]. This is evidenced not only by the similarity of the composition of the waste, but also by the similar climatic conditions. So, in the territory of the city of Baikalsk, the average daily temperature in November, December, January differs from similar temperatures in Bratsk at 4-5 °C. In February-March temperatures are similar (Figure 6). The conclusion, drawn by us, agrees with the data of experiments on freezing out the sediments of Baikal Pulp and Paper Mill carried out earlier by A.V. Bogdanov and employees [1].

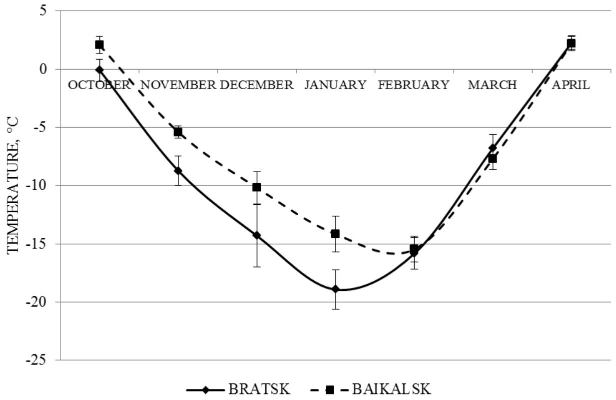

Fig. 6: Average daily temperatures in Baikalsk and Bratsk in the period from October to April (average temperatures for 2012 through 2017 are shown according to the data of the hydrometeorological services of Baikalsk and Bratsk, the data are presented by the Company "Reliable Prognosis" Ltd. (official site www.rp5.ru))

Freezing-thawing of lignin sludge from the Baikal Pulp and Paper Mill can significantly reduce the volume of this waste (the loss of volume is up to 50%). Such a procedure will reduce the cost of further processing of sludge lignin in any way. In addition, the change in the structure of sludge lignin, which occurs as a result of its freezing, makes it more accessible for biotransformation. The conclusion is confirmed by the results on the survival and reproduction of red Californian worms in the samples of sludge-lignin. So, in the untreated lignin sludge, worms died within 1-2 days after application. At the same time, in pre-fouled worms, not only survived, but also gave a significant increase in numbers and an increase in the number of coprolites. The sludge-lignin, formed in the fertile soil, can be subsequently used for recultivation of the territory of sludge accumulator plots of the Baikal Pulp and Paper Mill.

4. Conclusion

Thus, in this paper, the applicability and prospects of using the freeze-thaw method for dehydrating sludge-lignin from the Baikal Pulp and Paper Mill are demonstrated. This procedure leads to a significant reduction in the amount of waste due to the separation of water from them (up to 50% of the total volume). Its structure changes from gel-like to structured. The availability of sludge-lignin components for biotransformation processes is increasing.

The experiments showed the principal possibility of the worm transformation of sludge lignin, previously dehydrated by the freeze-thaw method, by the red Californian worms *E. foetida* Andrei Bouche (1972). This allows the waste to be recycled into a fertile soil. It can be later used for recultivation of the territory of maps of sludge accumulators of the Baikal Pulp and Paper Mill.
In addition, the conducted experimental studies prove that the product formed during the freezing and thawing of sludge-lignin without further processing can be used as an organic fertilizer for planting forestry crops, floriculture and recultivation of disturbed lands. According to the relevant chemical indices, it is also suitable for growing technical, fodder, grain and sideral crops. The resulting product can be used in a personal subsidiary farm for growing seedlings of vegetable and flower crops. The amount of

this fertilizer, recommended for use, in relation to the volume of soil is 1:3 – 1:4.

Acknowledgement

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