Study of Resistance of Phosphorus Biocomposite Fertilizers Based on Birch Bark to Washing-out of the Active Components

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Abstract

The resistance of biocomposite fertilizers (BF) obtained *via* impregnation of porous substrates of bast and birch bark by an aqueous solution of K_2HPO_4 to washing-out of potassium was studied. The effect of drying temperature, acid treatment conditions, and initial potassium contents on its aqueous washing-out from the resulting fertilizers was found. Washing-out of potassium and phosphates from BF obtained under different conditions was compared. It was demonstrated that fertilizers based on a substrate of bast were superior to that of birch bark on average in 1.2 times in their resistance to washing-out. The salt amount applied to substrates should not exceed 29.5 mass %, as established for obtaining fertilizers with maximum resistance to washing-out of potassium and phosphates. The resulting BF are characterized by the ability to slow evolving of the active components. After aqueous washing-out for 30 days, no less than 19.5 mass % of phosphates and potassium remains therein, which ensures the long-acting effect. The introduction of BF into peat soil in the amount of 5.0–15 mass % has little effect on its acidity. It was shown that BF based on substrates of bast and birch bark were not inferior to industrial granular fertilizer in their resistance to washing-out of phosphates, and in resistance to washing-out of potassium, even surpassed it. .

Keywords: biocomposite fertilizer, birch bark, impregnation, potassium, phosphates, washing-out

INTRODUCTION

Birch refers to the main hardwood species, the wood of which is prepared on an industrial scale. Large-tonnage wastes of birch bark are mainly burned or disposed of, thus polluting the environment.

A promising area of biomass waste disposal, including the bark, is the production of complex organomineral fertilizers based therein [1–4]. Their use improves soil structure and aeration. The soil is enriched with a series of valuable organic compounds resulting from biodegradation of wood components of fertilizers, which is important to preserve and increase soil fertility that is the basis of high crop yields and food security of the country.

Biomass wastes are of great interest as components of prolonged action fertilizers that are able to release the active component (urea) during a long time [5, 6]. In some cases, additives of organic compounds, particularly, esters are used to give elevated water resistance [7]. Products of chemical processing of vegetable wastes may be used to obtain polymer coatings of traditional fertilizes, which ensures controlled, slow release of nutrients into the soil [8]. It is worth noting that the use of prolonged action fertilizers allows dealing comprehensively with a number of problems: to improve the efficiency

of the use of traditional water-soluble fertilizers due to reducing their washing-out with ground water; to decrease pollution level of ground and surface water in active agricultural areas; reduce the cost for the introduction of fertilizer, *etc.* [9, 10].

The use of vegetable absorbents to fix liquid wastes of the vital functions of animals in the development of complex fertilizers is known [11]. Wood wastes as reproducible and relatively inexpensive raw materials are quite promising as such carriers. Papers [12−14] demonstrated an opportunity of production and described properties of BF based on porous substrates of bast and birch bark containing a variety of nutrients and characterized by the slow release of the active components. To remove polyphenol compounds that exert negative effects on the growth and development of plants processing of initial raw material with a 1.5 % alkaline solution was proposed. The development of substrate porous structure happens simultaneously. An increase in drying temperature of substrates of bast and birch bark impregnated by potassium chloride solution has a significant effect on the texture formation of the resulting fertilizer and contributes to an increase in the resistance of BF to washing-out of potassium with water, as demonstrated by the studies in [13]. Conditions for the production of fertilizers based on birch bark *via* the impregnation method of substrates with dibasic potassium phosphate solutions that show elevated resistance to washing-out of phosphates were determined considering these results [14]. The authors developed the sequence of operations (isothermal ageing, drying, and acid treatment) and their conditions that allowed substantially decreasing washing-out of phosphates with water from the resulting biocomposites. However, a phosphorus-containing salt (K_2HPO_4) used for substrate impregnation is also a source of potassium that is an important biogenic element for plant nutrition. In this regard, to search for the optimum conditions of production of fertilizers that ensure maximum resistance of fertilizers based on birch bark to washing-out of not only phosphates but also potassium, it is required to study washing-out of potassium of these fertilizers.

The work purpose is the study of resistance to washing-out of potassium with water from BF obtained by impregnation of porous substrates of bast and birch bark with K_2HPO_4 solution and comparison of processes of washing-out of phosphates and potassium therefrom.

EXPERIMENTAL

Air-dry (moisture content of (7.5±0.5) %) Silver Birch bark and bast separated of birch-bark were used as raw materials to produce porous substrates of BF. After grinding of raw materials, mixtures of the following fractional composition were prepared, mass %: 0.25−0.50 mm 23.7, 0.50−1.00 mm 76.3.

Porous substrates (PS) of birch bark and bast were obtained by treatment with 1.5 % NaOH solution under the following conditions: temperature of (80±5) °C; hydromodulus 5; processing time of 1 h; the mixing intensity of (130±5) rpm. The preparation technique of PS is given in detail in [15]. The resulting substrates were dried at (50±5) °C to the air-dry condition.

Biocomposite fertilizers were obtained by substrate impregnation with an aqueous solution of K_2HPO_4 of various concentrations according to the scheme proposed in [14]. Figure 1 gives the scheme.

Salt solution amount for impregnation corresponded to the moisture capacity of substrates that was determined by GOST 24160−80 technique. Phosphorus content in BF varied from 3.5 to 5.0 mass %. The amount of potassium introduced into biocomposite fertilizer was calculated based on the formula of $K_2HPO_4 \cdot 3H_2O$. Table 1 gives phosphorus, potassium and salt amounts corresponding to the stoichiometric ratio.
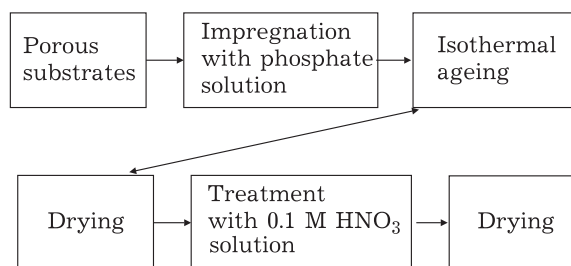

Fig. 1. Scheme for obtaining biocomposite fertilizers based on substrates of bast and birch bark.

TABLE 1

Amounts of phosphorous and potassium corresponding to the amount of the salt $K_2HPO_4 \cdot 3H_2O$ deposited onto the substrate, mass %

$K_2HPO_4 \cdot 3H_2O$	Phosphorous	Potassium
25.8	3.5	8.9
29.5	4.0	10.1
33.2	4.5	11.4
36.9	5.0	12.7

Impregnated samples were aged in closed weighing bottles at room temperature for no less than 4−6 h. Afterwards, impregnated samples underwent isothermal aging at 100 °C for 2 h. BF samples were then dried to the air-dry condition varying temperature from 60 to 250 °C.

Their acid treatment was carried out by impregnation with HNO_3 solutions of various concentrations (0.05−1.0 mol/L). Acid solution amount for impregnation corresponded to substrate moisture capacity. Impregnated samples were aged in closed weighing bottles at room temperature for no less than 2 h and then dried at 80−100 °C.

Biocomposite fertilizers quality was assessed by their resistance to washing-out of phosphates and potassium with distilled water at (20±2) °C under stationary conditions. The ratio of BF samples and water in experiments was 0.4 g to 100 mL. The duration of washing-out varied from 10 min to 96 h. The replacement of an aqueous solution during 30 days was carried out each 24 h when studying washing-out of phosphates and potassium from BF samples.

Phosphorus concentration in water was determined by the mass-spectrometry method using ICP-MS 7500 Agilent device. Additionally, the determination by the photometric method was carried in accordance with GOST 200851.2−75.

The resistance of BF to washing out of the active components was assessed by their residual content (in the percentage, in terms of P_2O_5 and K_2O). During calculations, the initial amount of phosphorus and potassium in BF samples also recalculated for P_2O_5 and K_2O was taken as 100 %.

Granulated fertilizer with the trade name of Kemira Universal 2 (ZAO Fertica, Moscow, Russia) was used as a reference sample. It contained 7.6 % P_2O_5 and 13.5 % K_2O.

The effect of additions of BF on soil acidity was studied on an example of universal peat soil (Express Kemilax OOO, Novosibirsk, Russia). It had the following characteristics: moisture content of 9.5 %, humus content of 15.8 %, ash content of 74.7 %, the content of total nitrogen of 162 mg/L, phosphorus of 155 mg/L, potassium of 206 mg/L. Soil analysis was performed according to standard techniques [16]. BF samples contained 4.0 mass % phosphorus and 10.1 mass % of potassium and were obtained under the following conditions: isothermal aging of impregnated substrates at 100 °C for 2 h; drying at 200 °C; treatment with a 0.1 M HNO_3 solution. The amount of BF introduced into soil varied from 5 to 25 mass %. Air-dry soil in an amount of 500 g was mixed with biocomposite fertilizer, then, the resulting mixture was selected by 20 cm^3. The samples were treated with 100 cm^3 of a 0.1 M KCl solution. After 10 min, the value pH KCl in the filtrate was determined by the potentiometric method.

Thermal properties of porous substrate samples were studied by TG and DTG methods using Netzsch STA 449F1 Jupiter device in the 30−800 °C temperature range under argon. The heating rate for samples of substrates of birch bark was 5 °C/min, of bark bast 10 °C/min.

An electron-microscopic study of samples of BF based on a substrate of birch bark bast was carried out using TM-1000 and TM-3000 scanning electron microscopes (Hitachi, Japan).

RESULTS AND DISCUSSION

An increase in drying temperature of a substrate of birch bark bast impregnated with K_2HPO4 solution to 200 °C leads not only to a change in salt distribution character in a substrate, as established in earlier studies. The resulting sample is of a darker colour compared to samples dried at 60−100 °C temperatures [14]. Alkali-soluble bast substances formed resulting from substrate impregnation with an alkaline salt solution followed by drying give this colour to the sample. Caramelization of polysaccharides contained in bast may also lead to a colour change.

These compounds are likely to form cross-linked structures. Their presence in the

substrate surface has been determined *via* scanning electron microscopy (Fig. 2, *a–c*). Such structures have not been detected in the surface of the blank substrate of bast (see Fig. 2, *d*).

It can be seen that the size of holes in these structures is substantially lower than that of pores and substrate cavities, on the walls of which they are located (Fig. 2, *a, b*). It may be suggested that these formations will contribute to a decrease in washing-out of potassium and phosphates from the resulting biocomposite fertilizer. Salt distribution in the substrate is more even, as established earlier [14]. In combination with the detected formations, this ensures elevated water resistance of fertilizers.

The darkening of samples of BF based on substrates of bast and birch bark happens with increasing their drying temperature of over 170 °C, as visually detected. Substrates not impregnated with salt solution did not change their colour with increasing drying temperature to 220 °C. Application of K_2HPO_4 is likely to affect thermal conversion processes of substrates.

By an example of a substrate of bast, a change in its decomposition process due to the impregnaion of an aqueous solution of K_2HPO_4 is demonstrated (Fig. 3). Three main temperature ranges in thermal decomposition profiles of the samples under study may be distinguished.

Heating of the blank substrate to 230 °C is accompanied by the removal of adsorbed water. The mass loss at this temperature is 7.3 mass %. Intense degradation of substrate organic matter proceeds in the 230–400 °C range and gaseous and liquid products are formed [17]. Mass loss in this temperature range is 49.1 mass %. Cyclization and aromatization processes determine the third temperature range (400–800 °C) that corresponds to coke residue formation [17]. The residual mass of a substrate of bast at room temperature is 30.0 mass %, as established.

Impregnation of a substrate of bast with an aqueous solution of K_2HPO_4 leads to changes in the thermal decomposition process, especially, in the range of medium and high temperatures (see Fig. 3).
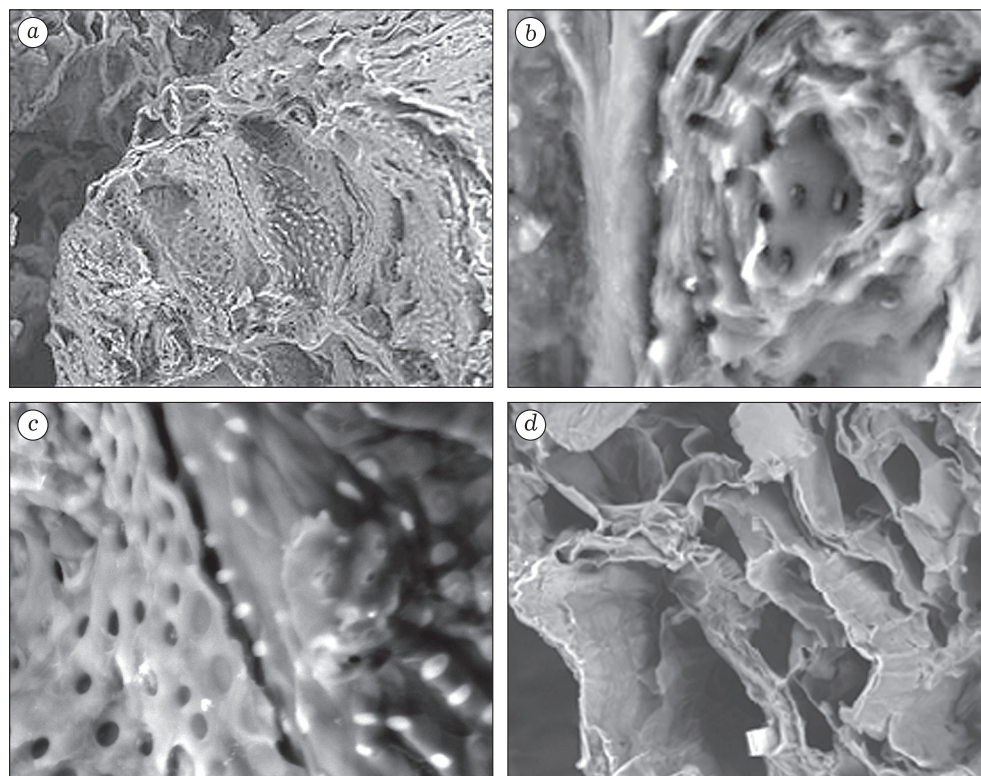

Fig. 2. SEM-images of the surface of biocomposite fertilizer (*a–c*) based on a substrate of birch bast obtained at drying temperature of 200 °C, and the blank substrate (*d*). Magnification of 1000 (*a, d*) and 5000 (*b, c*).
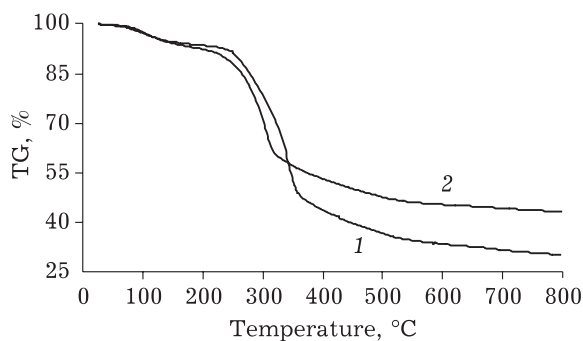
Fig. 3. TGA data of the blank substrate of birch bark bast (*1*) and a substrate of bast containing K_2HPO_4 (*2*). The initial phosphorus content in the substrate is 3.5 mass %.

The temperature of maximum decomposition and associated mass loss rate are increasing in the low-temperature range (Table 2).

Perhaps the removal of crystallization water that is a part of potassium hydrophosphate determines this. The values of temperatures of the beginning (T_{in} = 233.7 °C) and end (T_{fin} = 327.4 °C) of primary decomposition are shifted to a low-temperature region compared to the blank substrate, for which T_{in} = 250.3 °C and T_{fin} = 382.1 °C. In this temperature range, the maximum decomposition rate is a little different from the value obtained for the blank substrate, however, the maximum temperature substantially shifts to the low-temperature range. Herewith, mass loss of a sample containing K_2HPO_4 decreases (see Table 2). The residue mass after reaching the final temperature of thermolysis is by 13 % higher than that of the blank substrate. Application of K_2HPO_4 onto a substrate of bast is likely to contribute to an increase in its resistance to thermal decomposition.

There are two peaks of thermal decomposition in a substrate of birch bark, unlike that of bast, as demonstrated by Table 2 data. Perhaps this is driven by differences in removal processes of water of birch-bark and bast. Additionally, they substantially (in two times) differ by thermal degradation rates with a small difference in temperatures of maximum decomposition (7.9 °C) of substrates made of bast of birch bark. It is worth noting that changes in thermal degradation processes, to which impregnation of a substrate of birch bark with hydrophosphate solution leads to, are similar by their nature to those for a substrate of bast and differ by temperature values of decomposition maxima (see Table 2).

The absence of any additional decomposition processes, apart from water removal, during heating of impregnated substrates attests to the thermal stability of substances formed during interaction with K_2HPO_4 and, as a consequence, netlike neoformations in substrates (see Fig. 3, Table 2).

Thermal analysis results demonstrate that the maximum temperature of drying of BF with various salt contents is advisable to limit by a value of 230 °C.

As established earlier, an increase in drying temperature of BF based on substrates of birch bark bast leads to a decrease in washing-out of phosphates with water [14], however, this affects in different ways in the

TABLE 2

Data on differential thermal analysis (DTG) of samples of birch bark bast and birch bark

Substrate material	Temperature of peaks of thermal decomposition, T_{max}, °C	Rate of decomposition at T_{max}, %/min	Mass loss at T_{max}, %
Birch bark	73.7	0.38	0.6
	83.7	0.41	1.4
	353.7	3.14	43.6
Birch bark bast	98.2	0.79	2.6
	345.8	6.55	43.5
Bast with K_2HPO_4*	106.2	0.90	2.9
	306.2	6.61	32.3
Birch bark bast	87.6	0.85	2.6
with K_2HPO_4*	312.4	4.58	32.1

* Initial phosphorous content 3.5 mass %.

resistance of BF to washing-out of phosphates and potassium therefrom (Fig. 4). Thus, the resistance of BF to washing-out of potassium is higher and increases almost linearly in the 60–170 °C temperature range. Perhaps at these temperatures, the interaction of potassium with functional groups of bast, in particular, ion exchange with COOH functionalities, contributes to the more durable fastening of this metal in the substrate.

Biocomposite fertilizers obtained as a result of their drying at 180–230 °C show maximum resistance in respect to washing-out of phosphates and potassium. Herewith, the resistance of phosphates, on average, by 8.8 % exceeds this indicator for potassium (see Fig. 4). The resistance of BF to washing-out of active substances decreases, which agrees with thermal analysis results.

It is worth noting that using the stage of isothermal ageing of substrates of bast and birch bark impregnated with salt solution leads to a minor increase in their water resistance against potassium: the achieved increase does not exceed 3.2 %. This technique allows increasing resistance to washing-out of phosphates from BF on average by 13 % in a temperature range of drying of 180–220 °C [14].

Treatment with a 0.1 M HNO3 solution (see Fig. 1) leads to an increase in water resistance of BF samples dried at a temperature of 170 °C and higher, as established (see Fig. 4 and 5). Acid treatment has a great positive impact on resistance of BF to washing-out of potassium, as demonstrated by comparative data analysis of Fig. 4 and 5. Thus, sample resistance of fertilizer dried at 200 °C with respect to potassium and phosphates increases after acidification in 1.4 and 1.2 times, respectively. Note that this sample is characterized by somewhat better (by 3.5 %) ability to retain potassium compared to phosphates (Fig. 5).

As already noted, acid treatment leads to a decrease in the colour of aqueous extracts of BF samples due to a decrease in the solubility of substances that formed as a result of the interaction of substrates of bast and birch bark with solutions of an alkaline salt (K_2HPO_4) used for impregnation followed by drying at 200 °C [14]. Obviously, this treatment leads to an increase in resistance of netlike structures detected in substrate surface by the SEM method (see Fig. 2) and able to slow down washing-out of active components from the resulting fertilizers.

Acid treatment of BF based on a substrate of birch bark affects in different ways resistance to washing-out of phosphates and potassium, as established (Table 3). The studies were carried out on samples containing 3.5 mass % of phosphorus and 8.9 mass % of potassium, respectively. Biocomposite fertilizers are characterized by a lower resistance to washing-out of potassium. Note that washing-out of potassium from BF samples with water for 24 h is only slightly dependent on the concentration
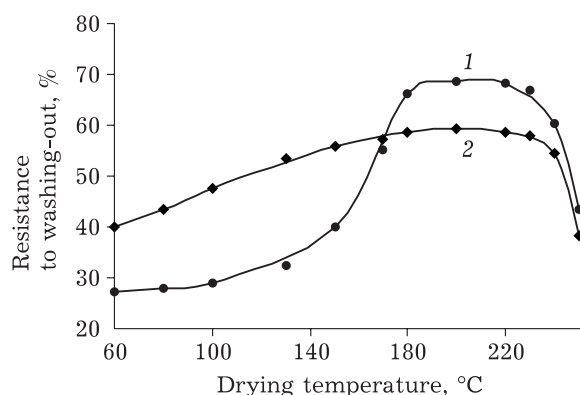

Fig. 4. Effect of drying temperature of biocomposite fertilizers based on a substrate of birch bast on the resistance to washing-out of phosphates (*1*) and potassium (*2*). Initial content of phosphorus of 3.5 mass %, potassium – 8.9 mass %.
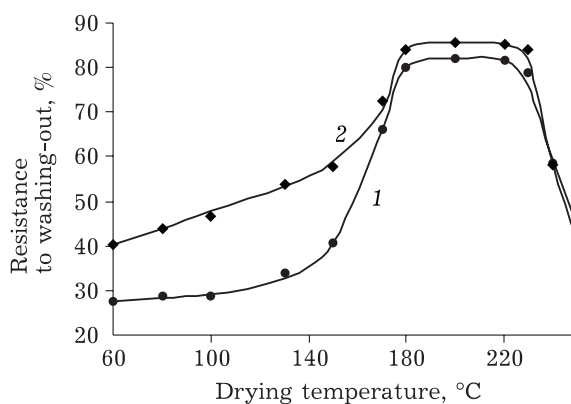

Fig. 5. Effect of acid treatment of biocomposite fertilizers based on birch bark bast dried at different temperatures on resistance to washing-out of phosphates (*1*) and potassium (*2*). Initial content of phosphorus of 3.5 mass %, potassium – 8.9 mass %.

of acid used for processing, and the positive effect of this treatment is minor.

There is the same trend for the effect of acid solution concentration to resistance to washing-out of both phosphates and potassium (see Table 3). The maximum increase in resistance of these BF to washing-out of potassium is reached due to their treatment with 0.10–0.15 M HNO_3 solutions on average in 1.5 times.

In their resistance to washing-out of phosphates, BF based on a substrate of birch bark are inferior to BF based on substrates of bast only in the first 10 min. Further, the properties of these samples are a little different from each other. The maximum increase in resistance of BF samples based on substrates of birch bark is achieved as a result of treatment with 0.10–0.15 M HNO_3 solutions (an average of 1.2 times). The use of more concentrated acid solutions is impractical due to a decrease in sample resistance.

Less firm retention of potassium by a substrate of birch bark is probably driven by the presence in its surface of Fe^{3+} and Mn^{2+} ions detected using EPR. Herewith, a substrate of birch bark contains considerably more Fe^{3+} ions than a substrate of bast [12]. The
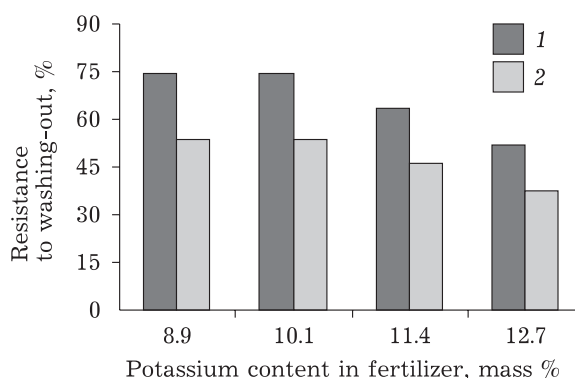

Fig. 6. Effect of the initial potassium content in biocomposite fertilizers based on a substrate of bast (*1*) and birch bark (*2*) on its resistance to washing-out with water. Duration of washing-out of 24 h.

electrostatic repulsion between potassium and positively charged active sites of the substrate is likely to limit its fixation strength in the studied surface.

The initial content of potassium in BF (Fig. 6) affects fertilizer resistance to its washing-out with water. It can be seen that the application of over 10.1 mass % of potassium onto the substrate leads to a decrease in resistance of the resulting BF to its washing-out.

TABLE 3

Effect of nitric acid concentration on resistance of biocomposite fertilizers to washing-out of phosphates and potassium with waterof nitric acid concentration on resistance of biocomposite fertilizers to washing-out of phosphates and potassium with water

Substrate material	Concentration of HNO_3, mol/L	Resistance to washing out*, %			
		of phospates		of potassium	
		Time			
		10 min	24 h	10 min	24 h
Birch bark bast	0	68.7±1.2	56.7±0.9	59.3±1.2	49.5±1.1
	0.05	72.7±1.3	63.8±1.1	67.4±1.3	59.6±1.2
	0.10	81.7±1.3	70.5±1.2	85.2±1.3	74.7±1.3
	0.15	81.3±1.2	69.7±1.2	84.9±1.2	74.1±1.2
	0.25	77.6±1.1	64.2±1.1	79.6±1.2	66.3±1.1
	0.50	74.8±1.2	56.1±1.0	74.5±1.1	60.5±0.9
Birch bark	0	64.1±1.2	54.6±1.0	60.1±1.2	50.4±1.2
	0.05	68.5±1.3	59.6±1.1	65.4±1.3	53.3±1.2
	0.10	76.3±1.2	68.6±1.3	71.5±1.2	53.8±1.3
	0.15	75.9±1.3	68.2±1.2	70.9±1.3	54.2±1.1
	0.25	70.7±1.2	62.1±1.2	66.3±1.1	52.3±1.0
	0.50	67.2±1.1	55.4±1.1	63.7±1.0	51.4±1.1

* From the initial content.

TABLE 4

Washing-out biocomposite fertilizer (BF) based on birch bast and birch bark (initial content of phosphorous and potassium in BF is 4.0 and 10.1 mass %

Substrate material	Resistance to washing-out*, %							
	of phosphates				of potassium*, %			
	Time, h							
	24	48	72	96	24	48	72	96
Birch bark bast	70.3	68.1	65.9	63.6	74.2	71.8	69.9	67.6
Birch bark	68.2	66.7	65.5	63.4	63.3	59.4	55.1	52.6
"Kemira-Universal 2"**	72.1	61.5	47.4	36.6	52.3	24.3	2.8	0

* Of the initial component content.

** Reference sample.

Importantly, BF samples with the maximum water resistance initially contain 8.9 and 10.1 mass % of potassium and 3.5 and 4.0 mass % of phosphorus, respectively (see Table 1). Biocomposite fertilizers with the minimum content of phosphates have been obtained with this initial content of phosphorus [14]. Obviously, BF obtained with the application of no more than 29.5 mass % of $K_2HPO_4 \cdot 3H_2O$ are most resistant to washing-out of potassium and phosphates with water (see Fig. 6, Table 1).

Washing-out of the active components with water for 96 h from BF samples based on substrates of bast and birch bark containing 4.0 mass % of phosphorus and 10.1 mass % of potassium has been studied. The maximum amount of phosphates and potassium is washed-out in 24 h from BF samples, as it follows from the data from Table 4. Washing-out of the active components is slowing down within the subsequent time, as evidenced by a change in resistance of BF. Thus, average daily decreases

in resistance to washing-out of phosphates and potassium for BF sample based on substrates of bast are 2.24 and 2.20 % a day. Considering these values, the period of complete removal of phosphates and potassium from BF are 31.3 and 33.7 days, respectively.

By resistance to washing-out of phosphates and potassium, BF based on substrates of bast and birch bark are superior to industrial granular fertilizer with a trade name of Kemira-Uneversal 2 (Fertica ZAO, Moscow, Russia, Table 4).

An experiment on washing-out of phosphates and potassium with water for 30 days from BF based on substrates of bast and birch bark demonstrated that no less than 19.5 % of the active components of their initial amount in fertilizers remained therein. The ability to slow washing-out of these active components for a long time determines the long-acting effect of the resulting BF.

Since to increase the resistance of BF to washing-out of the active components therefrom acid treatment stage was used, it was of interest to assess the effect of introducing these fertilizers on soil pH. Table 5 gives the results of the determination of the indicator pHKCl of peat soil containing different amounts of fertilizers, the properties of which are illustrated in Table 4.

It can be seen that the introduction of up to 15 mass % of the fertilizers under study into soil does not lead to a significant change in its acidity. These pH values are the boundary between slightly acidic and neutral soil classes. A soil mixture becomes slightly acidic when introducing of a large amount of BF [16].

TABLE 5

Effect of additions of biocomposite fertilizers based on substrates of bast and birch bark on the index pH_{KCl} of peat soil

Content of BF in soil, mass %	pH value of soil in the presence of BF	
	of substrate of birch bast	of birch bark
0	6.55±0.05	6.55±0.05
5	6.55±0.05	6.55±0.05
10	6.45±0.05	6.50±0.05
15	6.40±0.05	6.40±0.05
25	6.25±0.05	6.30±0.05

CONCLUSION

It has been demonstrated that the impregnation method of porous substrates of bast and birch bark with an aqueous solution of K_2HPO_4 allows obtaining biocomposite fertilizers with high resistance to aqueous washing-out of phosphates and potassium.

The resistance of biocomposite fertilizers based on a substrate of bast and birch bark to washing out of potassium is higher on average in 1.2 times compared to a substrate of birch bark, as established.

As a result of research, it has been established that maximum resistance of biocomposite fertilizers to washing-out of potassium and phosphates is reached under the same preparation conditions: isothermal aging of samples impregnated at 100 °C for 2 h, drying at 180–220 °C followed by treatment with a 0.1 M HNO_3 solution. Herewith, the amount of salt applied onto substrates should not exceed 29.5 mass %. The detected changes in the morphology of biocomposite fertilizers obtained under these conditions ensure an increase in their resistance to washing-out of the active components. It has been demonstrated that the resulting fertilizers have the ability to slow washing-out of phosphates and potassium with water. No less than 19.5 % of the active components remain therein after 30 days. This ensures the long-acting effect of the proposed fertilizers.

The introduction of these fertilizers into the soil in an amount up to 15 mass % has a slight effect on its acidity, as established.

It has been demonstrated that biocomposite fertilizers are not inferior to industrial granular fertilizer in resistance to washing-out of phosphates, and they are superior to it in resistance to washing-out of potassium.

Acknowledgements

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