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RESEARCH ARTICLE

DEVELOPMENT OF A PRODUCTION ALGORITHM FOR A LIQUID HUMIC FERTILIZER

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ABSTRACT

An algorithm for production of a liquid humic fertilizer (from the waste of the biotechnological manufacturing of a biological-origin liquid-phase biological product) has been presented. The algorithm includes factors affecting end-product qualities such as extraction agent concentration and volume, extraction temperature, etc. The work has several stages. The quality of the experimental samples of the liquid humic fertilizer has been assessed at each stage. Having taken account of all the results, the following optimum parameters for the liquid humic fertilizer have been evaluated: a thrice-repeated alkali extraction with a 1.5% potassium hydroxide solution at the waste:KOH ratio = 1:10 at the temperature of 60 ^{0}C for six hours. The mother liquor of the liquid humic fertilizer was dark brown and had no bad odor. Potassium humate, humic acid and spore-formers concentrations in the fertilizer were at least 17 g/l, at least 2.7 g/l and at least n*10^2 colony-forming units/ml, respectively. The liquid humic fertilizer was recommended for a root or out-of-root feeding of plants, as well as to activate soil microorganism effects.

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INTRODUCTION

Production of most humic preparations and fertilizers is based on the conventional method of treating humus-containing materials with an alkali reagent (Roganov *et al*., 2014). The only differences in these popular technologies deal with reaction components' proportions and processing parameters. Here, peat or peat-water suspensions (Khokhlov, 2011; Trofimov, 1994; Novitsky *et al*., 2000), sapropel (Novitsky *et al*., 2000), brown coal (Novitsky *et al*., 2000; Olshansky, 2003), etc. are usually used as the humus-containing material, and potassium or sodium hydroxide (Bulganina *et al*., 1987; Mitrofanova *et al*., 2001) as well as potassium pyrophosphate (Novitsky *et al*., 2000) are most often used as the alkali reagent. Each author proposes his/her own technological parameters and modes to produce a particular humic fertilizer. In particular, the proportion of the reaction components (humus-containing material : alkaline reagent) varies from 1:5 to 1:25 (Novitsky *et al*., 2000; Mitrofanova *et al*., 2002, Lyasin, 1993), the processing temperature varies from ambient values (Novitsky *et al*., 2000; Mitrofanova *et al*., 2002) to high ones (Morard *et al*., 2007; Titov *et al*., 1998). It should be noted that all the technologies are targeted, that is they are aimed at production of a particular liquid humic product from the very beginning and, in this connection, provide a necessary stage of raw materials preparation. Our institute developed a technology for the production of a liquid-phase biological product of biogenic nature used as a plant growth and development stimulator and a soil fertility activator. The basic raw material of this technology is a peat-and-manure mixture. Its technological procedures, parameters and modes are aimed to keep the biogenic nature of the product. One of the key stages of the biotechnological production gives a solid-phase sediment which is basically a waste product. At the same time, this waste product can be considered as a humus-containing raw material for production of a liquid humic fertilizer, because the inner potential of the peat is not used completely. This work trend is topical and promising because it can solve the problem of

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liquid-phase biological product manufacturing waste recycling and simultaneously produce a new type of liquid humic fertilizer that does not need the preliminary stage of its raw material preparation. The latter, obviously, will lower the product's cost.

MATERIALS AND METHODS

On the basis of analysis of scientific articles and patents pertaining to methods for production of humic fertilizers and products, an algorithm for production of a liquid humic fertilizer from wastes of biotechnological manufacturing of a liquid-phase biological product of biogenic nature has been worked out. In working out the algorithm, we took into account the factors affecting the quality of the end product and specified their boundary values (Figure 1). It should be noted that it is practically impossible to take account of all the factors and choose their optimum combination. That is why this work was done step by step. Experimental samples of the liquid humic fertilizer were obtained and analyzed for the content of humic acids (according to Russian State Standard GOST 9517-94 adapted to the method for peat's humic acids evaluation) and potassium humates (the solid residue left after evaporation). The total number of microorganisms (by limit dilutions on a nutrient medium of a meat infusion agar) and the number of bacilli (by limit dilutions on a nutrient medium of a meat-and-wort infusion agar) have been evaluated. A most widely used in such works and relatively inexpensive potassium hydroxide was chosen as an extragent. It can both activate the peat's humic complex and be the source of potassium as a mineral nutrient for plants. It is reasonable to choose a triple extraction since it is known (Kukharenko *et al.*, 1957) that the most complete and commercially adequate humic acids extraction requires three successive extractions with an alkaline reagent. Here, the first extraction gives the greatest amount of the humic acids, whereas the further extractions yield additional ones.

RESULTS AND DISCUSSION

At the first stage, a temperature mode and an extraction time were adjusted. According to the algorithm, the following starting parameters were chosen: extragent – KOH, triple extraction, extragent concentration = 2.0 %, extragent volume = 1:10. With extracted mass temperature rise, concentration of humic acids and potassium humates increased, and their maximum concentrations could be observed at maximum temperatures. However, temperatures over 60 ^{0}C caused hydrolysis of the humic substances in the alkaline medium (Titov *et al.*, 1998). Moreover, inactivation of some biologically active substances and lysis of microorganism cells occur even at 40 ^{0}C. We determined the top temperature limit, 60 ^{0}C. At this temperature, three experimental samples of the humic fertilizer were extracted: within three, six and 12 hours of extraction. The best quality indices were obtained in the longest time interval (Table 1). Though, it is clear that extraction at 60 ^{0}C for 12 hours consumes much energy, which is unreasonable in this case. When temperatures were below 60 ^{0}C, the humic substances were extracted considerably slower. The quality of such liquid humic fertilizer was lower, its production time increased. In this case, even 36-hours-long extraction at 40 ^{0}C failed to give the values of the parameters evaluated that were reached in six-hours-long extraction at 60 ^{0}C.

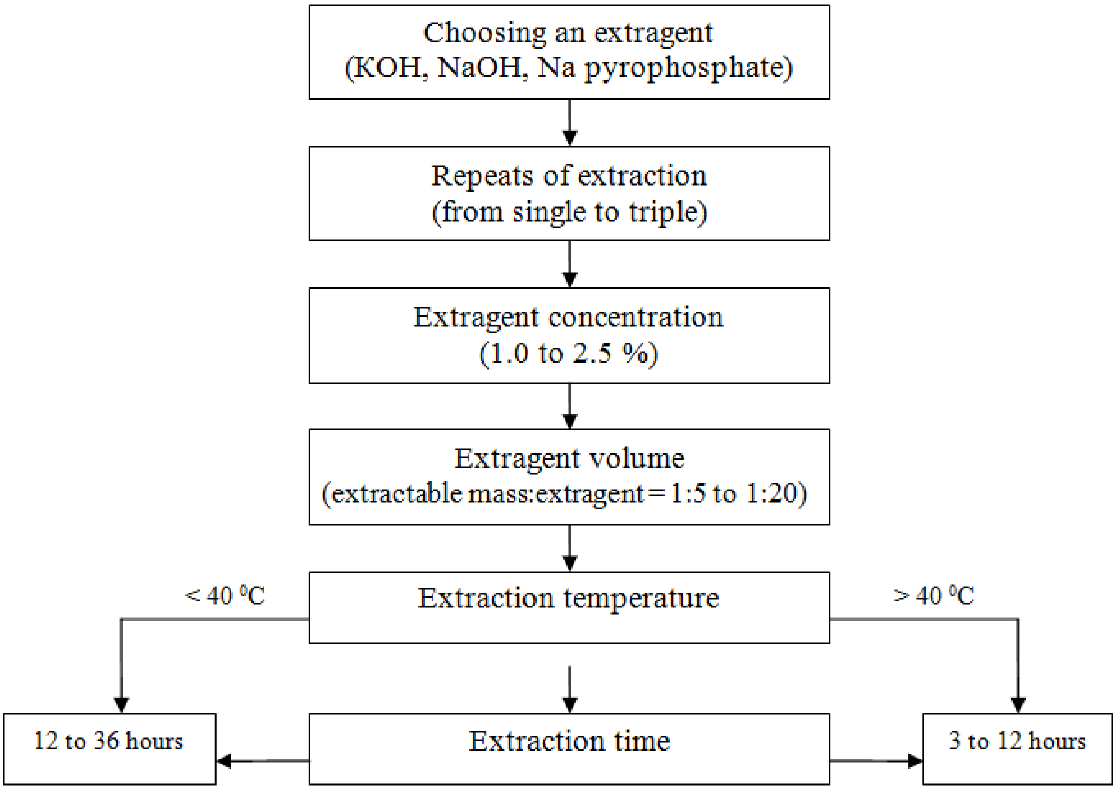

Figure 1. An algorithm for production of a liquid Humic fertilizer from biotechnological production wastes

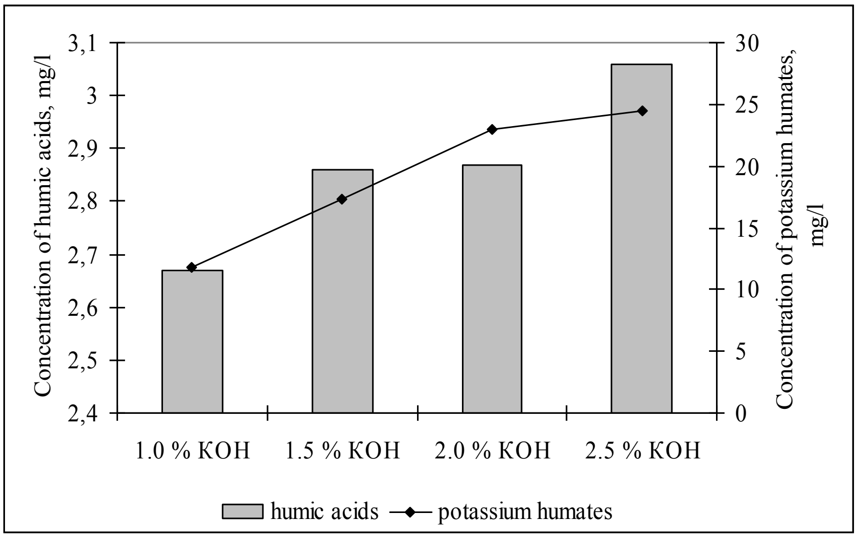

Figure 2. An extragent concentration effect on the quality of the liquid Humic fertilizer

Table 1. An extraction time effect on the quality of the liquid humic fertilizer

Extraction time, hours	Concentration of humic acids, g/l	Concentration of potassium humates, g/l
3	1.58	21.40
6	2.86	22.13
12	3.16	23.26

Table 2. An extragent proportion effect on the quality of the liquid humic fertilizer

Waste:KOH proportion	Concentration of humic acids, g/l	Concentration of potassium humates, g/l
1:5	4.41	23.54
1:10	2.86	22.13
1:15	1.64	21.28
1:20	0.91	20.12

Table 3. Microbiological indices of the liquid humic fertilizer

KOH concentra tion	Waste:KOH proportion = 1:5		Waste:KOH proportion = 1:10	
	Total number of microorganisms, C.F.U./ml	Non-pathogenic spore-forming microorganisms, C.F.U./ml	Total number of microorganisms, C.F.U./ml	Non-pathogenic spore-forming microorganisms, C.F.U./ml
1.5	10	40-100	20	400-600
2.0	Unobserved	20-60	Unobserved	40-100

Table 4. An effect of the extragent concentration and input components proportion on the final product output, (%)

KOH concentration	Waste:KOH proportion = 1:5		Waste:KOH proportion = 1:10	
	Final product output	Unusable sediment	Final product output	Unusable sediment
1.5	75.1	24.9	85.2	14.8
2.0	80.4	19.6	86.4	13.6

So, further increasing of temperature for this extraction time is commercially unreasonable. The first stage of the algorithm development yielded the optimum extraction time and temperature mode: six hours at 60 ^{0}C. The second stage of working out the production algorithm for the liquid humic fertilizer consisted in selection of the best extragent concentration. To do that, a series of liquid humic fertilizer samples were obtained by treating the raw material with potassium hydroxide in different concentrations (see Figure 1): 1.0 %; 1.5 %; 2.0 %; 2.5 %. Here, the following input parameters were left constant: extragent volume = 1:10, extraction temperature = 60 ^{0}C, and extraction time = six hours. The concentration of humic acids and potassium humates increased with increasing extragent concentration (Figure 2). Using either 1.5 % or 2.0 % potassium hydroxide gave practically the same amount of humic acids. So, commercial point of view favored the lower concentration. Using 1.0 % to 2.0 % KOH yielded a linear increase of the potassium humates concentration, whereas further raising KOH concentration up to 2.5 % gave no noticeable rise in them. It should be noted that using maximum concentrations of potassium hydroxide makes the liquid humic fertilizer production more expensive. The third stage of working out the production algorithm for the liquid humic fertilizer dealt with the extragent volume selection. For this purpose, liquid humic fertilizer samples produced when the proportion of the biotechnological production waste and extragent were 1:5; 1:10; 1:15, 1:20. In these experiments, other parameters were left constant: extragent concentration = 2.0 %, extraction time = six hours, extraction temperature = 60 ^{0}C. Increasing of the extragent volume led to a poorer quality of the final product. Moreover, it needed more expenditures on preparing a greater volume of the alkaline solution (Table 2). It should be noted that the higher waste:KOH proportion, first of all, affected the amount of humic acids in the fertilizer. At the same time, the concentration of potassium humates lowered not so noticeably (approximately by 5 % for each next proportion of components). From the analysis of the results of the second and third development stages, it was believed reasonable to additionally study two proportions between the biotechnological production waste and extragent, such as 1:5 and 1:10, as well as two extragent concentrations, such as 1.5 % and 2.0 %. For a deeper characterization of the experimental samples of the liquid humic fertilizer obtained, a non-pathogenic spore-forming microorganism concentration and a total number of microorganisms were evaluated (Table 3). Table 3 shows that these characteristics are the best in the liquid humic fertilizer sample produced using the proportion of components equal to 1:10 and 1.5 % potassium hydroxide. The concentration of non-pathogenic spore-forming microorganisms in the finished liquid humic fertilizer suggested that that fertilizer had a high agrochemical effect. When using the humic fertilizer as intended, the spore-forming microorganisms get into favorable conditions and go to their vegetative form, that is they become capable to grow and multiply again, activating microbiological processes in the soil, as well as processes of the growth and development of plants.

For the final choice of the optimum extragent concentration and proportion of the initial components, additional experiments were carried out. They included the liquid humic fertilizer production in conditions similar to industrial-scale ones. Table 4 suggests that waste:KOH proportion equal to 1:10 gives the final product, the liquid humic fertilizer, more by 7 % to 11 %, and, correspondingly, less unusable product than in case of raw materials proportion equal to 1:5. Besides, when the proportion was 1:10, extragent concentration changes had an insignificant effect on the product output.

Considering all the data obtained concerning the product's quality, amount and cost, we came to the conclusion that the waste:KOH proportion equal to 1:10 and potassium hydroxide concentration equal to 1.5 % are the optimum conditions for the liquid humic fertilizer production.

Conclusion

An algorithm for liquid humic fertilizer production from biotechnological production wastes has been worked out. The analysis of the results obtained allowed choosing optimum parameters for the waste processing, which became a basis for the liquid humic fertilizer production procedure development: the triple alkaline extraction by the 1.5 % potassium hydroxide solution with the waste:KOH proportion equal to 1:10, at 60 ^{0}C, for six hours. This works was awarded with a Russian Federation patent (Fomicheva *et al.*, 2014). The liquid humic fertilizer produced by this technique is a dark-brown mother solution without any bad odor, containing at least 17 g/l potassium humate, at least 2.7 g/l humic acids, and at least $n*10^2$ colony-forming units/ml. The liquid humic fertilizer is recommended for use as a root or out-of-root feeding of plants, as well as to activate soil microorganism effects.

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