Optimisation of Thermal and Biochemical Methods for Recovery of Wastes from Extraction Processing of Birch Bark*

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Abstract

Various methods for recovery of birch bark processing wastes to produce energy, carbon sorbents, biological products, and humin substances have been investigated. Optimum ways for combustion of fixed residues from extraction processing of a birch bark, which differ in moisture and ash content, have been determined by means of calculations. Clearing of a part of bast pores in the birch bark during recovery of extractives is favourable to form the developed cellular structure after carbonization and subsequent activation of bast. The produced sorbents match commercial powder sorbents, which are made from qualitative wood, with respect to their performance. Selection of cultivation conditions is performed for fungi of *Trichoderma* species on bast of a birch bark, these conditions providing the production of biological preparations with nutrition from ammonium nitrogen and the production of humic substances with nitric nitrogen used.

INTRODUCTION

Wood biomass wastes that are formed in great quantities in wood harvesting and reprocessing are traditionally considered as recycled energy feedstock [1, 2]. Along with creating effective devices for direct combustion of wood residue (for example, the fluidised bed furnaces), technologies for its pyrolysis and gasification to form liquid and gaseous combustibles [3–5] are developed.

Carbon materials, which can be then converted to carbon sorbents, are also produced by means of the pyrolysis processes for the wood residue [6–8].

The alternative direction to recover lignocellulose waste is its biotechnological

processing to yield biogas, humic substances, and biological products [9-11].

The combined technologies for recovery of the lignocellulose waste that are based on the integration of thermal, chemical, and extraction processes [12, 13] are developed in recent years. Adoption of these technologies allows a more complete tapping of chemical and power potentials of the vegetative biomass through expansion of the assortment of manufactured production [14].

Large-tonnage waste product from wood reprocessing is the bark, which can amount from 10 to 15 % of the wood trunk volume [15]. The bark with high content of tanning matter (an oak, a willow, a larch, *etc.*) is exposed to extraction processing to yield tanning agents. The basic part of bark waste products

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with a low content of tanning agents is dispatched to dumps or is burnt.

The bark of a birch, the significant wastes of which are formed upon reprocessing of the birch wood raw at the woodwork manufacturers, in production of plywood and wood charcoal, is classed with low utility kinds of bark. The birch belongs to the basic forest forming species of Russia [16]. At present, an interest in the employment of biologically active triterpenic compounds that are inherent in the birch bark has increased drastically. The external part of the birch bark (outer birch bark) contains up to 25-30 mass % of betulin [17], and the interior part (bast), up to 10-11 mass % of tanning matter [18]. The extraction of these substances yields tanning agents and anthocyanidin dyes.

The majority of developed extraction processes to yield valuable materials from a birch bark includes a stage of birch bark separation from the bast. Significant resources of bast of the birch bark are formed also in birch bark harvesting when manufacturing birch-bark products and tar.

The purpose of the present work is to study the viable ways for utilization of the birch bark bast and fixed residue from its extraction processing with the aim to produce energy, carbon sorbents, biological products, and humic substances.

EXPERIMENTAL

Bast from a bark of drooping birch (fraction less than 1.5 mm) and fixed residue from its extraction processing was used as raw material. The studied specimens differed in the recovery degree of extractives. Chemical composition of initial bast (% from the mass of absolutely dry material): polysaccharides total 49.1 (including readily hydrolysable -26.3, hardly hydrolysable - 22.8), the lignin materials 34.8, water-extractable - 13.7, mineral substances - 3.4. The fixed residue from aqueous-alcohol-alkaline extraction of bast was applied for extraction of tanning agents. It had the following chemical composition (% from a. d. s. mass): polysaccharides total 37.9 (including readily hydrolysable – 17.9, hardly hydrolysable – 20.0), the lignin materials 20.2, waterextractable – 1.5, mineral substances – 2.4.

Investigation of chemical composition of bast before and after extraction, as well as after bioprocessing, was conducted according to practical standards [19]. To determine readily hydrolysable polysaccharides, treatment of bast with 2 % muriatic acid at 100 °C was applied. Hardly hydrolysable polysaccharides were determined using treatment of bast, which was free from readily hydrolysable polysaccharides (they were removed by 80 % sulphuric acid at 20-25 °C), with the subsequent dilution of oligosaccharide solution and then with boiling for 3 h. For quantitative determination of lignin, the carbohydrate part of bast was removed via hydrolysis with 72 % sulphuric acid. Humin substances were determined by means of their extraction with 2 % NaOH solution for 1 h at ambient temperature. The quantity of mineral substances (ash content) was determined by the combustion method.

Calculations of energy potential of waste recovery through extraction processing of the birch bark were conducted taking into account the data of technical and elemental analyses of raw material. The lowest combustion heat of bark waste was determined according to practical standard [20].

Various versions for liquefaction of bark wastes are compared: following the technology of "Tekhenergokhimprom" (Berdsk city); version with cyclone furnace on the basis of "Sibtekhenergo" (Novosibirsk) and "Biyskenergomash" technologies (Barnaul); version with a multi-step furnace following the technology of NESA Company (Belgium); version with a fluidised bed furnace following Segher technology (Belgium); version with a cyclone furnace following technology of Steinmuller Company (Germany); joint combustion with pulverous coal in thermal power station.

Experiments with obtaining carbon sorbents from the initial bark and from the bark residue upon the extraction were conducted in the pilot laboratory setup that simulated thermal regimes of the commercial device for the combined pyrolysis-activation process. The main reactor of the set-up was made from stainless steel; inner diameter of the reactor was 43 mm and height was 500 mm. The temperature in the reactor was controlled by means of RIF gauge. The flow of gases delivered to the reactor was set by means of rotameters, and that of superheated steam, by a metering pump. The reactor pressure was about 1 atm.

Specimens of absorbent carbon were generated as follows. A reactor with the loaded specimen was heated in a current of nitrogen with a rate $0.5 \,^{\circ}C/s$ up to the temperature of 600 °C and then maintained at this temperature for 30 min. Then the temperature in the reactor was raised up to an activation temperature (700 °C) and the delivery of activating gas started. Gas mixture from steam, nitrogen, and oxygen that simulated the composition of flue gases was applied as an activating agent. Upon the termination of experiment, absorbent carbon was discharged and weighed. Key parameter of activation, the burning index, was calculated with the assumption that the weight of coal-raw is 100 %. Technical features of coals were determined following the standard procedures [21].

For bioconversion of bast and its afterextraction residue, the native *Trichoderma asperellum* strain MT-97 (VKPM F-765) sect. Trichoderma has been chosen that showed antagonistic properties as to various originators of fusarial wilts of coniferous trees in the forest nurseries of Siberia [22].

Substrate was inoculated with micellar suspension of fungi at the rate of $1 \ 10^6$ spores/g of a.d.s. Mycelium was grown on the Capek medium, whose qualitative composition of inorganic salts NaNO₃, MgSO₄, KCl, FeSO₄, KH₂PO₄ and H₂O is standard for micellar fungi. Nitric nitrogen was additionally introduced in the form of NaNO₃, or ammonium nitrogen was introduced in the form of (NH₄)₂SO₄; its amount was 3 % from the substrate mass. The cultivation was held in a thermostat at a temperature of (27±1) °C.

The fungi cultivation rate was judged from the quantity of formed fungi conidiospores, which were counted up under a microscope. In parallel with this, the decrease in substrate mass with an increase in duration of cultivation was registered.

RESULTS AND DISCUSSION

Thermal recovery of the birch bark wastes

The studied wastes from extraction processing of a birch bark are comparable in combustion heat with brown Kansk-Achinsk coals. According to calculated data, the energy potential of fixed residue from extraction processing of the birch bark bast is about $(8\pm1.5)\cdot10^9$ J/t (per dry mass). However, physical properties of a bark differ significantly from the properties of fossil coal. This is the cause for technical problems in its combustion with traditional furnaces. Bark wastes are characterized by small density and bad looseness that complicates their transportation and dosage. Owing to low-density of bark particles, their premature removal from a furnace during combustion increases, which causes an efficiency of a boiler to reduce and the dust content of flue gases to grow.

When the conditions of stable transportation and dosing of a bark are complied with, moisture and ash content represent the major crucial factors for selection of the way of energetic employment of the bark waste materials.

To compare various methods for recovery of bark waste products, the dependences of the basic heat-engineering characteristics of these materials on their composition have been obtained by means of calculations. When determining the suitability for a fuel to be used in various devices, the major characteristics is adiabatic combustion temperature, *i.e.* theoretical temperature, which can be reached at combustion of a given kind of combustible with stoichiometric air quantity in the absence of thermal and mass-transfer processes with an environment.

Presented in Fig. 1 is one of the examples of the obtained dependences. Based on the completed computations, recommendations can be given on the selecting the optimum ways for combustion of fixed residues from extraction processing of a birch bark that vary in their composition. When an ash content of the bark residue is less than 20 % and moisture content is not more than 35 % (per dry mass), this residue can be used as a combustible that is



Fig. 1. Dependence of adiabatic combustion temperature of fixed residues from extraction of the birch bark bast on their water and ash content (per dry mass): 1 - content of ash is 10 % ($R_2 = 0.9994$), 2 - content of ash is 20 % ($R_2 = 0.9995$).

alternative to coal in the majority of serially made furnace arrangements.

When moisture is more than 35 %, the bark residue can be burnt in the fluidised-bed boilers or when it is mixed with combustible of higher quality. However, to reach an acceptable level of fuel supply for one package boiler with capacity of just 5 MW, one will need at least 10 thousand ton of bark a year. When an output of the extraction production is less 10 thousand ton of bark waste a year, the most economically feasible way of their energetic employment is a joint combustion with coal in thermal power stations under operation.

Economic feasibility of the bark waste recovery to yield carbon sorbents (active coals) is caused by an increasing scale of their use and by rather high cost. With current prices for quality carbon sorbents, economic efficiency of their production calculated per ton of the manufactured bark is much higher than with the use of the bark as combustible. However, the instances of commercial development of like technologies are rather few in number. The problem consists in the necessity of creating special set-ups for thermal activation of bark wastes, whereas the investment cost with their energetic employment either not required at all or is rather insignificant.

Traditional technologies for obtaining active coals include a stage of carbonisation of the wood raw material to produce coal-raw and a stage of activation of the last-mentioned by steam at elevated temperature [23]. In the course of activation, the pore space and specific surface of sorbent increases, the ratio between the micro, meso, and macro pore space changes. Gasification rate of superficial carbon during activation depends on the degree of structural regularity of carbon material. Carbon is gasified most easily and quickly in the disordered regions of carbon surface.

Competitiveness of technologies for reprocessing of bark waste products to active coals can be raised with new engineering solutions applied. We have used technique that is more efficient than the traditional one. This is a steam way of bark thermal activation with a mixture of flue gases and steam; this way was applied with profit in reprocessing of alternative kinds of wood raw material [24, 25].

Experimental study of the carbonisation and activation processes has been performed with specimens of initial bast (IB) of a birch bark and with specimens of fixed residue from bast extraction reprocessing: the bark extracted with hexane (H), as well as the bark consecutively extracted with hexane and ethyl acetate (HE), with hexane, ethyl acetate, and isopropanol (HEI), and with hexane, ethyl acetate, isopropanol, and water (HEIW).

It has been found that all five specimens are close to each other as to engineering data (water content, percentage of ash, particle size distribution). Some increase in the percentage of ash (from 3.3 to 4.0 mass %) is evidenced in a series of specimens: IB < H < HE < HEI ~ HEIW.

Upon carbonisation in the inert medium at 600 °C within a stationary layer, the yield of coal-raw for all specimens is virtually identical and comprises (34 ± 0.5) %. Water content of the produced coals does not exceed 2 mass % and the percentage of ash comprises 6–7 mass %.

The higher reactivity regarding a steam has been detected for the specimens of bark bast that were first extracted and then carbonised. High yield of active coal (50-80% from the mass of coal-raw) is attained with duration of activation of just ~5 min at 850 °C, while to activate coal from birch wood under the similar conditions, 30-60 min are required. By virtue of the fact that so short activation regime for coal-raw from bast is difficult to comply with in practice, it is suggested that its activation is effectuated at a temperature of 700 °C, which TABLE 1

Parameter	Sorbent specification					
	BAU-MF	UAF	AUP	Sorbent from bark*		
	(GOST 6217-74)	(TU 6-16-2409-80)				
Visual appearance	Not standardized	Not standardized	Close-grained product, free from mechanical impurities	Fine-grained powder, black in colour, free from mechanical impurities		
The main size						
of particles, mm Activity with respect to	0.5-1.5	» »	0.1-1.0	0.1-1.0		
iodine (%)	≥70	≥70	≥70	≥72		
Content of moisture (%) Grinding size,	≤10	≤10	≤10	≤7.2		
the remainder on mesh						
with cell size 100 $\mu m,\%$	Not standardized	≤ 5	Not standardized	Not available		
Content of ash (mass %)	≤10	Not standardized	≤10	≤0.8		

Comparison of parameters for commercial carbon sorbents and those produced from a birch bark

*It has been obtained through carbonization and activation of HEIW specimen.

makes it possible to reach a high porosity and the required burning index for 15-30 min.

The elevated reactivity of coal from extracted bark bast is caused by clearing of a part of its pores from extractives during extraction treatment. The total pore space of active coals from the birch bark bast comprised 0.44-0.55 cm³/g, the iodine adsorption capacity was 50-70 %, and that with respect to the methylene dark blue was 40-85 mg/g.

Performance specifications for the powder carbon sorbents that are produced in Russia are presented in Table 1. Presented in the same table are the characteristics of sorbent, which has been obtained through activation up to the burning factor of 50 % from carbonised HEIW specimen (a consecutive extraction of bast with hexane, ethyl acetate, isopropanol, and water has been applied). It is evident that the carbon sorbent produced from the birch bark bast is comparable in standardized indexes to the commercial sorbents of UAF, AUP, BAU-MF specifications.

Bioutilization of birch bark waste by Trichoderma fungi species

One of the ways to recover lignocarbohydrate waste is a biological degradation using various fungi. Fungi of *Trichoderma* species are most widely applied for these purposes [26]. These microorganisms quickly assimilate ligno-carbohydrate substrate, take active part in destruction of organic compounds, and they are easily grown.

Capability of these fungi to depress development of phytopathogenes has been used for creating of a series of biological preparations "Trikhodermin", which are applied for protection against plant diseases transmitted *via* soil. Solid ligno-carbohydrate raw material can be used as substrates for their production [22, 27].

The big content of polysaccharides (49.1 mass %) and of the materials of the lignin nature (34.8 mass %) available within the composition of the bark bast of drooping birch facilitates application of bast as a substrate to yield biological products.

The process of bast bioconversion by fungi of *Trichoderma* species with the use of an active purified clone of Maganskiy strain has been investigated. When carrying out the research, we used a standard composition of inorganic salts with additionally introduced nitrate nitrogen or ammonium nitrogen in the form of NaNO₃ or $(NH_4)_2SO_4$ (content of 3 mass %).

Dynamics of cultivation for the quantity of fungi spores on the birch bark bast upon the introduction of nitric nitrogen is illustrated by the data given in Table 2. Formation of puffed mycelium, white-grey in colour, is evidenced on the fourth day of cultivation. With an TABLE 2

Cultivation	Vield of spores quantity	Gain in yield of spores as compared			
Cultivation field of spores, quantity		duin in yield of spores us compared			
duration, days	of spores/g of a. d. bast	to the sown quantity, times			
0	$1.00\cdot 10^6$	1.0			
4	$1.16\cdot 10^7$	11.6			
10	$1.60\cdot 10^7$	16.0			
14	$1.90\cdot 10^7$	19.0			
19	$2.10 \cdot 10^7$	21.0			

Dynamics of cultivation for conidiospores of *Trichoderma* fungi species (the MG 97/6 strain) on the birch bark bast in the presence of $NaNO_3$ at 27 °C

increase in duration of cultivation, mycelium gets gradually the colour of a green pea. The yield of conidiospores as compared to their sown quantity increases by a factor of 12 after 4 days of cultivation and by a factor of 21 after 19 days.

Nearly linear character of the mass loss of the birch bark bast is evidenced with an increase in duration of fungi cultivation up to 19 days (Fig. 2). As is obvious, the substrate mass loss on the 19th day amounts to 19 %.

During biological degradation of bast by *Trichoderma* fungi species (Maganskiy strain), change in the content of all its basic components occurs (Table 3). Various components of a birch bark bast differ in their stability against



Fig. 2. Dynamics of the mass loss for the birch bark bast when *Trichoderma* fungi species is cultivated in the presence of $NaNO_3$ (*a*) and $(NH_4)_2SO_4$ (*b*) at 27 °C.

biological degradation. After 4 days of biotreatment of substrate, the most appreciable decrease is observed for the content of substances extractable with hot water (from 13.7 to 8.1 mass %) and of readily hydrolysable polysaccharides (from 26.3 to 17.3 mass %). The content of hardly hydrolysable polysaccharides and substances of the lignin nature drops less notably (by 5.5 and 5.2 mass %, respectively).

Thus, primarily recovered substances in the course of biological degradation with nitrate nutrition are water-soluble matter and readily hydrolysable polysaccharides. Biological degradation of hardly hydrolysable polysaccharides proceeds supposedly through the stages of transformation into readily hydrolysable polysaccharides and then into oligo- and simple sugars. Decrease in the proportion of substances of the lignin nature is attributable to the reactions of biological

TABLE 3

Chemical composition of the birch bark bast before and after biological degradation by *Trichoderma* fungi species in the presence of NaNO₃ during 4 days at 27 $^{\rm o}{\rm C}$

Substances	Content in the bast, mass %				
	Initial	After biological			
		degradation			
Mineral ones	3.42	3.98			
Water-extractable	13.71	8.04			
Readily hydrolysable					
polysaccharides	26.26	16.15			
Hardly hydrolysable					
polysaccharides	22.83	17.32			
Polysaccharides total	49.09	39.47			
Lignin substances	34.82	29.57			
Humic substances	-	17.62			

Cultivation duration,	Yield of spores, the quantity	Gain in the spore yield as compared
days	of spores/g of a. d. bast	to sown quantity, times
0	$1.00\cdot 10^{16}$	1
5	$0.42\cdot 10^{18}$	42
8	$0.55\cdot 10^{18}$	55
13	$0.63\cdot 10^{18}$	63

Dynamics of cultivation for conidiospores of Trichoderma fungi species (the MG 97/6 strain) on the birch bark bast in the presence of $(\rm NH_4)_2SO_4$ at 27 $^{\rm o}C$

degradation of phenolic acids and carbohydrates related to lignin. The process of biological degradation of bast is followed by its huminification, the content of humin substances being as great as 17.6 mass % even on the 4th day of cultivation.

It is known that not only availability of nitrogen in substrate, but also the nature of nitrogen compound applied may have an essential effect on the efficiency of cultivation of fungi spores. We have investigated the effect of nitric nitrogen replacement by ammonium nitrogen in the mineral nutrition of Trichoderma fungi species on biological degradation of the birch bark bast. It follows from evidence presented in Table 4 that the cultivation of fungi conidiospores is considerably intensified in the presence of ammonium nitrogen as compared to the case when nitric nitrogen is used. On just the 5th day, the yield of spores increased 42 times as compared to the sown quantity. On the further increase in duration of cultivation, retardation of fungi cultivation is observed; therefore, an increase in the cultivation time up to 13 days is inexpedient.

Comparison of spore yield with azotic and ammonium nutrition demonstrates that the yield of spores in the presence of $(NH_4)_2SO_4$ is 3–4 times higher than in the case of NaNO₃ during 4–10 days of cultivation (see Tables 2, 4).

However, the mass loss of the birch bark bast in the case of fungi cultivation with ammonium nutrition (see Fig. 2, b) is noticeably less than with the use of nitrate nutrition (see Fig. 2, a). With duration of cultivation for 4–14 days, the bast mass loss in the presence of $(NH_4)_2SO_4$ is 1.5–2.5 times less than is in the case of NaNO₃.

As suggested by the data presented in Table 5, biological degradation of substances from bast that are soluble in hot water proceeds most intensively in the presence of $(NH_4)_2SO_4$. Upon 5 days of cultivation, their content has decreased almost by a factor of two as compared to the content of initial bast. However, further increase of cultivation duration up to 8 and 13 days does not lead to significant decrease in the mass fraction of water-soluble matter.

The content of readily hydrolysable polysaccharides on the 5th day of cultivation

TABLE 5

TABLE 4

Chemical composition of the birch bark bast before and after biological degradation by Trichoderma fungi species in the presence of $(NH_4)_2SO_4$ at 27 °C

Substances	Content in the bast, mass %				
	Initial	With du	With duration of cultivation, days		
		5	8	13	
Mineral ones	3.42	3.13	3.67	3.59	
Water-extractable	13.71	7.00	6.05	5.19	
Readily hydrolysable polysaccharides	26.26	18.32	16.98	15.49	
Hardly hydrolysable polysaccharides	22.83	37.99	36.58	34.81	
Polysaccharides total	49.09	34.34	34.82	33.63	
Lignin substances	34.82	13.45	13.69	13.95	
Humic substances	-	2.89	4.74	8.32	

has decreased 1.3 times as compared to the initial bast. However, their recovery rate slowed down after 8-13 days of cultivation. Biological degradation of hardly hydrolysable polysaccharides proceeds less intensively in the presence of $(NH_4)_2SO_4$ than with the use of NaNO₃. The content of lignin matter practically does not change during biological degradation of bast in the presence of ammonium nitrogen. Hence, in this case, fungi use preferentially extractives and polysaccharides as their nutrition. The yield of humin substances in the presence of $(NH_4)_2SO_4$ is almost 6 times lower than in biological degradation with nitric nitrogen for 4-5 h (see Tables 3, 5).

On the strength of the obtained results, conclusion can be drawn that it is advisable to perform cultivation of *Trichoderma* fungi species on the birch bark bast with ammonium nutrition to yield the biological products with a high content of fungi spores. With the aim to produce humic substances and fertilizers from bast, nitric nitrogen, 3 % in quantity from the weight of substrate should be used when cultivating fungi.

CONCLUSIONS

On the basis of the completed research, the optimised ways of thermal and biochemical recovery of waste products from extraction processing of bast of a birch bark have been offered.

It has been demonstrated by means of calculations that when a content of ash in the bark residue is less than 20 % and moisture content is not more than 35 % (per dry mass), the residue can be employed as a fuel that is alternative to coal with the majority of serial furnace arrangements. At higher moisture content, it is advisable that the residue be burnt in fluidised bed boilers or, when mixed with coal, in thermal power stations under operation.

Clearing of some part of pores during recovery of extractives is favourable for the developed cellular structure to be formed after carbonisation and activation of the residue from extraction processing of the bark. High yield of active coal (from 50 to 80 % from the mass of coal-raw) is attained through its activation at 700 °C for 15–30 min. Active coals from bark

comply in their characteristics (total pore space of $0.44-0.55 \text{ cm}^3/\text{g}$, iodine capacity of 50-70 %, 40-85 mg/g capacity with respect to the methylene dark blue) with the commercial powder sorbents, which are produced from high-quality wood.

Selection of cultivation conditions for *Trichoderma* fungi species on the bast of birch bark has been performed, these conditions providing production of biological products or humic substances.

To produce biological preparations like "Trikhodermin", which are applied in protection against plant diseases, it is advisable to cultivate fungi with ammonium nitrogen nutrition. Upon 5 days of cultivation, the yield of fungi spores increases 42 times as compared to their sown quantity. Under these conditions, water-soluble matter and some part of readily hydrolysable polysaccharides are predominantly subject to biological degradation.

In the presence of nitric nitrogen, intensification of the processes of biological degradation of water-soluble matter and readily hydrolysable polysaccharides from bast is observed, and hardly hydrolysable polysaccharides and lignin matter get partially involved in the bioprocessing. The loss of mass of bast becomes as great as 19 % on the 19th day; however, in so doing the yield of fungi spores increases only by a factor of 21 as compared to the sown quantity. Biological degradation of bast in the presence of nitric nitrogen is followed by its huminification and the content of humin substances amounts to 17.6 mass % on the 4th day of cultivation. Thus, nitric nitrogen should be used for bioprocessing of bast into humin substances and fertilizers when cultivating fungi.

Results of the completed research give evidence for a variety of possible ways to recover the waste products from birch bark processing to produce energy, carbon sorbents, biological products, and humic fertilizers. When choosing feasible technologies for recovery of bark waste, one should start from the fact that their energetic application does not involve any additional investment cost. However, the processes for waste utilization to yield carbon sorbents and biological products are more favoured as regards manufacturing of products with higher added value.

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