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PRODUCTION OF PHENOL COMPOUNDS BY ALKALINE TREATMENT OF POPLAR WOOD BARK

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It is well known that, as a result of thermo-chemical treatment in a water-alkaline medium, lignin is destroyed and low-molecular-weight phenol compounds are obtained [1–4]. Low-molecular-weight phenol compounds (LMPC) 2-methoxyphenol, 2,6-dimethoxyphenol, vanillin, etc. were previously obtained from the alkaline destruction of technical hydrolysis lignin (THL), wood biomass, and poplar wood bark [5]. The optimal conditions for obtaining LMPC were lignin–5% NaOH ratio 1:10 and process duration 6 hours. The experiment has been carried out at 180°C [6].

Technical hydrolysis lignin is not as common a residue as wood bark. Moreover, treatment of the poplar wood bark leads to a considerably higher yield of low-molecular-weight compounds in comparison with those from THL treatment [5]. This statement is due to the more condensed THL structure. The aim of the present investigation is to carry out alkaline treatment of poplar wood bark under different conditions.

The lignin content in poplar wood bark is 37.40 mass % of initial bark.

It was found that with increasing process duration and concentration of NaOH (6 h, 7% NaOH) the highest quality of LMPC was obtained (Table 1). Moreover, at these conditions the influence of hydromodule on LMPC yield was studied. It was established that at h.m. 1:8 and 1:10 a similar yield of LMPC was obtained (Table 2).

Table 3 presents low-molecular-weight phenol compounds obtained from poplar wood bark under different conditions.

The possibility for obtaining low-molecular-weight phenol compounds through alkaline treatment of poplar wood bark was investigated. It was found out that alkaline treatment is an effective process, in which the main part of the lignin is destroyed, and as a result of this, low-molecular-weight phenol compounds are obtained. Using extraction with toluene from the liquid phase, the following compounds were isolated: 2-methoxyphenol, 2,6-dimethoxyphenol, 4-hydroxy-3-methoxybenzaldehyde, 4-hydroxy-3,5-dimethoxybenzaldehyde, 1-(4-hydroxy-3-methoxyphenyl) ethanone, etc.

For the purpose of this investigation, poplar wood bark was previously powdered.

The process and extraction conditions were determined based on the results of our previous investigations [5, 6].

The alkaline treatment of bark was carried out in stirring autoclaves made of stainless steel, heated in a bath of polyethyleneglycol at 180°C for 2, 4, and 6 hours. A water solution of NaOH (3, 5, and 7%) was used at different bark/water solution of NaOH ratios (hydromodule) – 1:6, 1:8, and 1:10.

The amounts of obtained extracts, insoluble residue, and precipitated and nonprecipitated lignin were determined.

The LMPC were identified by GC-MS analysis [5, 6].

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TABLE 1. Influence of Treatment Duration on Lignin Product Yield, $\%_{mass}$ of Initial Bark

Duration of alkaline destruction, h	Concentration of NaOH, $\%_{mass}$	Undissolved residue	Dissolved part		
			Precipitated lignin	Unprecipitated part in the water phase	Low-molecular-weight phenol compound
2	3	55.78	7.12	37.10	0.44
	5	30.91	16.05	53.04	0.91
	7	27.03	16.97	56.00	1.26
4	3	53.12	7.32	39.56	0.67
	5	28.98	17.79	53.23	1.40
	7	24.05	18.15	57.80	1.82
6	3	52.60	7.91	39.49	0.71
	5	28.65	18.80	52.55	1.48
	7	23.65	19.25	57.10	2.05

TABLE 2. Influence of Hydromodule on Lignin Product Yield (6 h, 180°C, 7% NaOH), $\%_{mass}$ of Initial Bark

Hydromodule	Undissolved residue	Dissolved part		
		Precipitated lignin	Unprecipitated part in the water phase	Low-molecular-weight phenol compound
1:6	25.24	18.31	56.45	1.93
1:8	23.65	19.25	57.10	2.05
1:10	21.75	19.42	58.83	1.99

Bark – 100.00% $_{mass}$.

TABLE 3. Compounds Identified in Toluene Extract of Destroyed Lignin

Compound	Retention time, min	Area, %*	Area, %**	Compound	Retention time, min	Area, %*	Area, %**
Phenol	2.91	3.85	2.58	2,6-Dimethoxyphenol	6.07	37.84	29.38
Unidentified	3.51	3.03	3.63	3-Hydroxy-4-methoxybenzaldehyde	6.53	4.48	6.67
2-Methoxyphenol	3.82	16.44	15.86	4-Hydroxy-3-methoxybenzoic acid	6.88	0.64	–
Unidentified	–	–	1.95	1-[4-Hydroxy-3-methoxyphenyl] ethanone	7.26	10.21	10.52
Unidentified	4.24	0.81	1.40	Unidentified	7.52	3.97	6.71
Unidentified	4.28	2.86	4.51	Unidentified	7.60	0.72	–
Unidentified	5.05	0.53	–	4-Hydroxy-3,5-dimethoxybenzaldehyde	8.76	1.92	3.59
4-Ethyl-2-methoxyphenol	5.45	1.42	3.17	1-[4-Hydroxy-3,5-dimethoxyphenyl] ethanone	9.49	11.28	10.04

*4 h, 5% NaOH, h.m. – 1:8; **6 h, 7% NaOH, h.m. – 1:10.

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