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INFLUENCE OF PARTICLE SIZE OF POWDERED PINE WOOD (*PINUS SYLVESTRIS*) ON ITS OXIDATION INTO VANILLIN*

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Catalytic oxidation of pine wood (*Pinus sylvestris*) with oxygen produces high yields of vanillin. The influence of mass transfer intensity on the oxidation process rate and selectivity has been little studied. It was shown that size of wood particles (fractions less than 2 mm) does not significantly affect the rate of oxidation or the maximum yield of vanillin. The highest recorded yields (up to 35–36 wt.% based on the initial lignin) exceeded those obtained from pine wood oxidation with nitrobenzene (23–27 wt.%). The high vanillin yields were confirmed by weight analysis, GLC, ¹H NMR methods. The results obtained indicate that in order to attain high vanillin yields from native lignin oxidation with oxygen it is sufficient to use sawdust with a fineness of 2 mm or less, and finer grinding the wood is not required. Vibrational wet milling disrupts the anatomic porous structure of wood, increases resistant to oxidation and decreases vanillin yield.

Keywords: Pine wood (*Pinus sylvestris*), vanillin, oxygen, nitrobenzene, wood particle size, diffusion.

For citing: Tarabanko V.E., Kaygorodov K.L., Smirnova M.A., Chelbina Yu.V., Vorobyev S.A., Mazurova E.V., Korsakov A.V. *Khimiya Rastitel'nogo Syr'ya*, 2026, no. 3, pp. 363–369. <https://doi.org/10.14258/jcprm.20260319369>.

Introduction

Finite nature of natural fossil resources leads chemical industry to inevitable gradual transition towards plant-based feedstocks. In this context, the “lignin-first” biorefining approach, in which lignin valorisation is considered in the design phase, has demonstrated the fullest utilisation of lignocellulose [1]. One tactical task within this strategy – production of vanillin, syringaldehyde, and cellulose from wood – is a subject of intense research.

Despite long history of studies on oxidation of lignins with molecular oxygen into the aromatic aldehydes (50–70 years) [2–4], influence of mass transfer intensity on the process rate and selectivity remained unexplored for a long time; sparse papers on this subject began appearing since the 2000's. Pacek showed that increasing the stirring intensity leads to higher rates of oxygen consumption and vanillin accumulation, which suggests diffusion-limited kinetics in the experiments on oxidation of lignosulfonates [5]. Another paper shows that vanillin yield in oxidation of pine wood depends on stirring rate [6], i.e. the process is diffusion-limited.

Quantitative study of how mass transfer intensity influences the rates of oxygen consumption and vanillin accumulation was first offered for a process using flax shives as the feedstock [7–9]. In a 1 L cylindrical reactor with a magnetic stirrer, the oxidation at 160 °C follows a linear dependency between logarithms of the stirrer rotation speed, and the rates of oxygen consumption and vanillin accumulation:

$$\ln W_{O_2} = 1.87 \ln N + a \quad (1)$$

$$\ln W_v = 1.86 \ln N + b \quad (2)$$

where W_{O_2} and W_v are the rates (mol/min) of O_2 consumption and vanillin accumulation respectively, and N is the stirring rate (min^{-1}). These dependencies correspond to previously established and recommended models for mass

* This article has electronic supplementary material (appendix), which is available to readers on the journal's website. DOI: 10.14258/jcprm.20260319369s

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transfer in gas-liquid systems [10]. They describe limiting of the overall process rate by diffusion of oxygen through the liquid diffusion layer at the gas-liquid or liquid-solid wood boundary, and are often interpreted using known model that links the rate of a diffusion-limited chemical process to the stirring power density [10]. It was also shown that the maximum vanillin yield is independent from the stirring power density in a wide range of values; i.e., the same yield can be attained under rate limitation by either external or internal diffusion [8, 9].

Among the unexplored subjects with both theoretical and applied significance, one can point out the influence of particle size distribution of the wood feedstock on the effectiveness of its oxidation into vanillin and cellulose. The effect of the particle sizing on the kinetics is the most conclusive criterion for distinguishing the processes limited by internal diffusion or chemical reaction; while knowing the maximum suitable particle size of the lignocellulosic feedstock for vanillin production is important for developing the technology of the process.

Earlier, we studied influence of particle size in aspen sawdust on the oxygen consumption rate during oxidation of different size fractions at 110–120 °C [11]. At high stirring intensity in a shaking reactor (50 mL of reaction mass, engine power 100 W, reciprocating motion with frequency 4–6 s⁻¹) the oxygen consumption rate was independent from the stirring intensity and particle size of aspen sawdust in the range 0.5–5 mm. These results can be interpreted as signs of a process with reaction-limited rate, but they obtained at relatively low temperature and high stirring power density [11] compared to later study [8, 9].

The goal of the present paper is studying the effect that particle sizes of pine wood loaded into the oxidation reactor have on its mechanical disruption, the maximum vanillin yield, and its accumulation dynamics.

Materials and methods

Pine wood harvested in the vicinity of Krasnoyarsk city (Russia) was used in this work. Composition of the wood (wt.%): lignin 27.1, cellulose 49.5, extractives 4.63 [12, 13]. Separation of coarse size fractions in the range 0.5–2 mm was performed with agitated sieves. Finer fractions were obtained by milling of aqueous suspensions with a 1 kW vibration mill MV-2 (Russia) with steel cylinders grinding media (loaded wood sample 120 g, liquid-to-solid ratio 7, milling time 5–15 min with breaks for cooling of the mill and the suspension after every 5 min). Wet milling was used to prevent wood overheating. Iron impurities were extracted from the ground samples with 3% HCl for one hour at room temperature.

A reaction mass was prepared by adding the desired amounts of the components – the pine wood (15 g), NaOH solution (15 g), water (300 mL), and the catalyst solution (CuSO₄ · 5H₂O, 11.25 g) in the stated order into the reactor, stirring all the while. The catalyst under the process conditions eventually converts into copper oxide (12 g L⁻¹) dispersed on the wood surface and in the aqueous phase. The oxidation experiments were conducted in an autoclave of 1 L capacity at 160 °C and oxygen partial pressure 0.2 MPa for 30 minutes and stirrer speed 1200 rpm, unless specified otherwise (Fig. 1 of supplemental material). The majority of the oxidation experiments were performed twice, once to record the oxygen consumption dynamics, and once more for periodic sampling the reaction mass to determine the vanillin content. Samples of the reaction mass (13–15 mL) were taken from the reactor through a special valve during the oxidation process, and after the experiment. The aldehyde concentration was determined by GLC [7]. Wood particle size distribution before and after the oxidation was performed using laser diffraction with a Betsizer S 3 Plus unit (BetsizerInstruments LTD, China). The surface morphology was studied using a TM4000 Plus scanning electron microscope (Hitachi, Tokyo, Japan). The ¹H NMR spectra were measured using a BrukerAvance III 600 spectrometer at 300 K.

Results and discussion

In order to clarify the influence of mass transfer on the native lignin oxidation in the three-phase gas-liquid-solid system, it is important to obtain data on the initial state of the wood solid particles (principally, their size) and on their degradation during the oxidation. These results were produced by laser diffraction and scanning electron microscopy. Then, we will discuss the role of the wood powder particle size in the dynamics of oxygen consumption, and of vanillin accumulation during the oxidation.

Influence of the initial wood particle size on size of the post-oxidation lignocellulosic residue (LCR) particles. To study the effect of the wood's extent of grinding, a wide assortment of size fractions was used, covering the range from 5 to below 0.5 mm (obtained by dry sieving), and also two finer fractions obtained by wet-milling of sawdust. Particle volume distribution functions for the powders after 5 and 15 minutes of wet-milling both exhibit

two maxima at diameters 1000 and 100–400 microns (Fig. 1a). The number distribution maximum for the wet-milled wood samples lies in a much finer range (5–20 microns, Fig. 1b) in comparison with the volume-based distribution. The number-based maximum shifts to a finer value with longer milling duration, while no such notable shift occurs in the volume-based distribution. The mass fraction of these micron-sized particles is minuscule (Fig. 1a), and therefore they cannot define the process kinetics. Considerable difference between the volume and the number distributions shows high polydispersity of the studied powders. Scanning electron microscopy (SEM) confirms the particle size data from the laser diffraction (Fig. 2 of supplemental material). SEM also shows that while native anatomical structure of wood is partially preserved after 5 minutes of wet-milling, 15 minutes of milling disrupts it practically completely.

Figure 2 presents data on size distribution of particles by volume (and by consequence, masses) in lignocellulosic residues (LCR) from oxidation of the different initial wood size fractions, as determined by laser diffraction. The LCRs from coarse initial fractions of the wood (from >0.5 to <2 mm) exhibit bimodal volume distribution with maxima at 20–80 and 500–1000 microns. The latter maximum represents the size of the initial wood particles, while the former represents the particles that appear during the oxidation because of chemical destruction and mechanical disruption of the feedstock. The fraction from thorough wet-milling (15 min) and the finest dry-sieved powder (<0.5 mm) produce LCRs with monomodal size distribution of the disrupted particles (the maximum is at 20–30 microns). Hence, these fractions mechanically destructed completely during the oxidation. Fraction obtained by wet milling during 5 minutes is the most recalcitrant for mechanical disruption during oxidation (Fig. 2, line 1).

In this way, except the 5 minutes wet-milled fraction, the exact distribution depends on the initial size: The smaller are the feedstock particles, the faster they disintegrate to the size 20–80 microns. Oxidation of the dry-sieved fractions (from <0.5 to 2 mm) produces LCRs with partially retained anatomical structure. The wet-milled (5 min) fraction loses this structure and is pressed after the oxidation (Fig 3 of supplemental material). This pressing and losing the wood anatomic structure explains the recalcitrant behaviour of this fraction during the oxidation, and it will appear and discuss later.

It should be noted that after only 2 min of the oxidation, the LCR from the 1–2 mm sawdust fraction comprises large enough particles with retained anatomical structure. Unlike the initial wood size fraction, this LCR divides into finer fractions (1–2 mm 20 wt.%, 0.5–1 mm 57%, 0.25–0.5 mm 15%, and <0.25 mm 8 wt.%). In other words, disintegration of the wood particles down to tens of microns observed at the late stage of the oxidation is not apparent at the early stage, and this disintegration develops over the entire process duration.

The results obtained show that during oxidation processes, significant destruction of the wood structure occurs under conditions of high mass transfer intensity. As a result, quite low yields of solid residue (15–22% by weight) and cellulose (30–36%, based on the initial content in wood) are obtained. The amount of lignin in the solid residue is of 4–6% by weight, based on the native one (Table 1). The optimum conditions for producing maximum yields of vanillin or cellulose may not coincide [14], and this study focuses on the maximum production of vanillin.

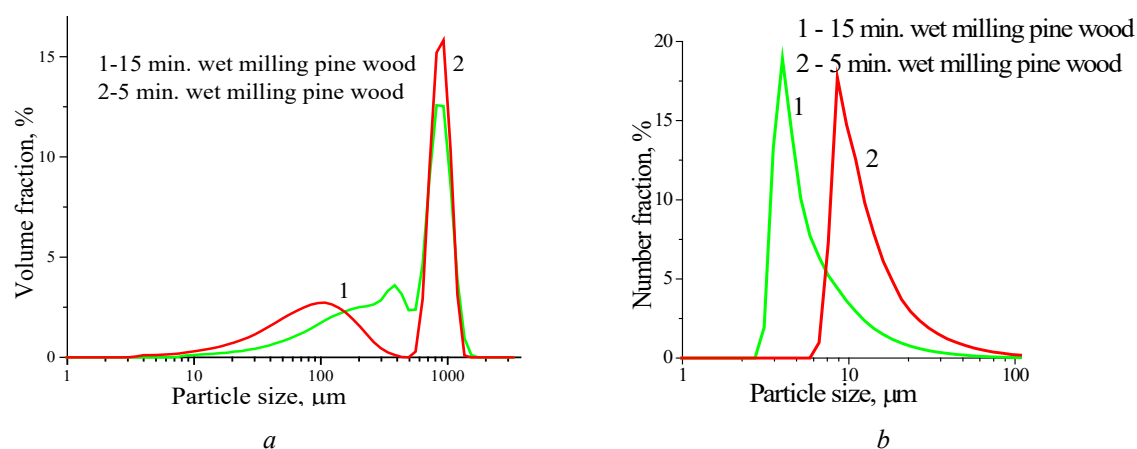


Fig. 1. Distribution of solid volume (a) and particle number (b) by the particle diameters of pine wood powder, wet-milled for 5 and 15 minutes

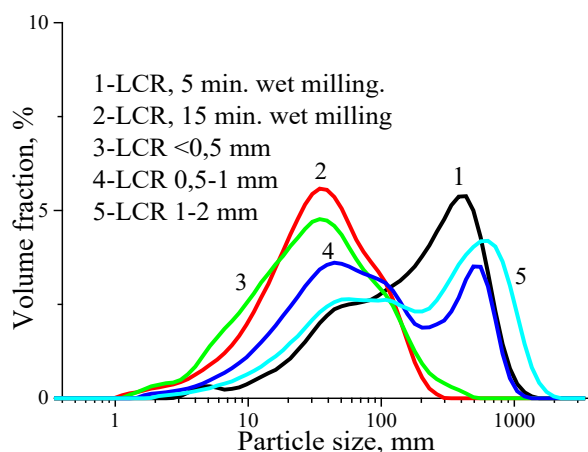


Fig. 2. Distribution of solid volume by the particle diameters in lignocellulosic residues after the oxidation, for different size fractions of the initial wood

Influence of wood's particle size distribution on kinetics of its oxidation. Figure 3 presents a series of dependences of vanillin accumulation, oxygen consumption, and pH change versus oxidation duration for different size fractions of pine wood, at 160 °C in the reactor with intense stirring.

The maximum vanillin concentration (4.5–5 g/L) is attained at 12–15 minutes and has almost no dependency on the size fraction in the range 0.1–2 mm (Table 1). It is noteworthy that there is a small but reproducible difference of vanillin yields between the coarse feedstocks (36.5 wt.% based on the initial lignin) and the wet-milled ones (32.6 wt.%). This may be caused by disruption of the wood anatomical structure, its partial pressing and loss of porosity during wet milling, and this is the only observed influence of the wood particle size on the process kinetics.

Vanillin yield at zero-time moment is relatively high, approximately a half of the maximum yield. Vanillin formation proceeds without oxidants as a result of alkaline hydrolysis of lignin. The ratio of vanillin yields obtained by the hydrolysis and oxidation was studied by Pacek [5], and similar results were obtained. Alkalinity of the reaction mass decreases below pH 13 after the vanillin concentration maximum is attained, and this corresponds to complete alkali consumption by the oxidation products. A pH above 11–12 is required for the formation of vanillin during oxidation [3]. This means that the amount of alkali used in the experiment is close to the minimum required. It should be strongly noted that the vanillin yields (32–37 wt.% based on the initial lignin) are relatively high (Tables 1, 2), compared to results of nitrobenzene oxidation of fir (28%) [15] and loblolly pine (30–34%) [16]. Oxidation of pine wood (*Pinus sylvestris*) with nitrobenzene with *o*-phenanthroline gave 36% of vanillin based on the initial lignin [17]. The herein obtained vanillin yields also exceed the earlier published data on three-stage oxidation of prehydrolyzed pine wood with oxygen (25–28%) and coincides with the maximum selectivity of vanillin formation at the first two stages of oxidation (35–37%) [18]. Usual concentrations (0.3–0.6 g/L) of impurities of acetovanillone and vanillic acid are registered; Together with vanillin they give the total yield of monomeric phenols of 38–42% (Table 1). This result exceeds a little the recently obtained yield of monomeric phenols in the oxidation of pine lignin with oxygen, 34% [19].

The obtained results of pine wood oxidation (38–42 wt.% of phenolic monomers) may be compared to the modern results on oxidation of hardwood lignins with oxygen. Oxidation of birch wood released 28.7 wt.% monophenols [20], red oak gave around 40% of phenolic monomers, mainly syringaldehyde and vanillin [21], and monophenolic yield was 48.6 wt.% for eucalyptus oxidation [19]. Usually nitrobenzene oxidation produces less aromatic aldehydes from softwood (20–27%) compared to hardwood (42–50%) [3], but the results listed above show that the yields of these products in the oxidation of softwood and hardwood with oxygen get principally closer.

Table 1. Yield of oxidation products from different size fractions of powdered pine wood. Pine wood (15 g), NaOH solution (15 g), water (300 mL), and the catalyst ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, 11.25 g), 160 °C, stirrer speed 1200 min^{-1} . Time to attain the maximum vanillin yield, 12 min

Size fraction	Vanillin (<i>V</i>), g/L	Acetovanillone (<i>AV</i>) + vanillic acid (<i>VA</i>), g/L	Oxygen consumption, mmol	Vanillin, wt.% based on the initial lignin	<i>V+AV+VA</i> , wt.% based on the initial lignin	Lignin in the wood sample, wt.%	Residual lignin, based on the native, %
1–2 mm	4.97	0.30 + 0.32	540	36.7	41.2	27.1	5.3
0.5–1 mm	4.90	0.42 + 0.41	565	36.2	42.3	27.1	6.0
wet-milled for 5 min	4.70	0.37 + 0.54	437	32.2	37.9	29.2	5.7
wet-milled for 15 min	5.08	0.43 + 0.58	457	33.0	39.6	30.8	4.3

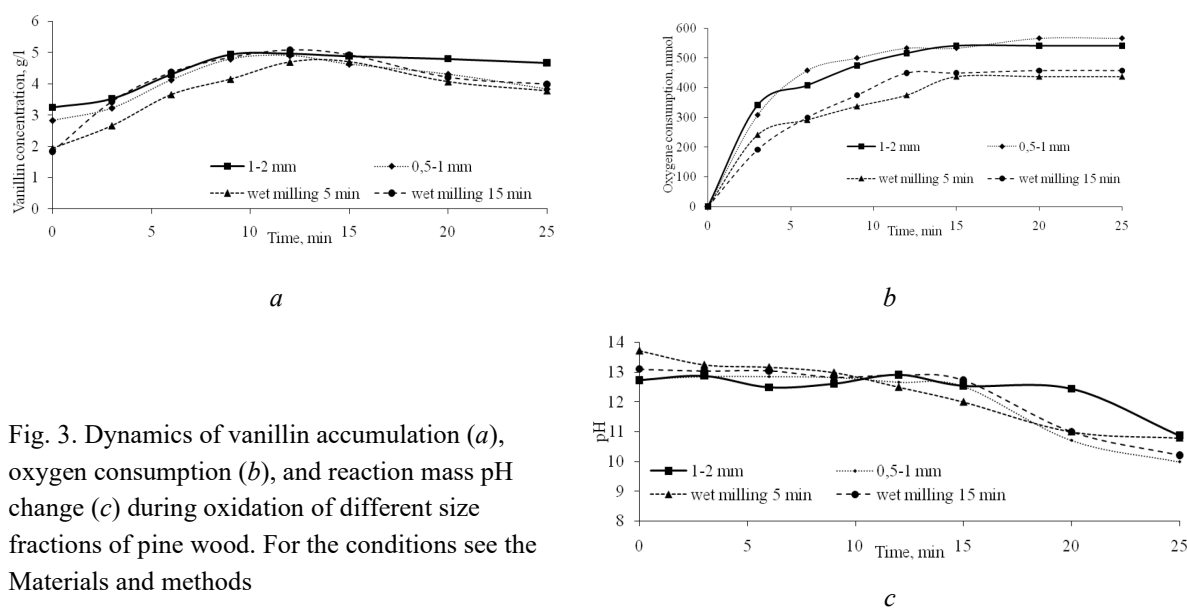


Fig. 3. Dynamics of vanillin accumulation (a), oxygen consumption (b), and reaction mass pH change (c) during oxidation of different size fractions of pine wood. For the conditions see the Materials and methods

Table 2. The maximum yields of vanillin in the oxidation of pine lignin with nitrobenzene and oxygen

References	2	17	16	20	6	18	19	This paper
Oxidant	Nitrobenzene			Molecular oxygen				
Vanillin yield based on the native lignin, wt. %	27	36	32–34	23	21.1	25–28	31.8	32–36
Vanillin concentration, g/L		9.5		4.5	0.97	–	1.5	5.0
Total monomers yield based on the initial lignin, %	–	–	36	–	26	30–32	33.8	38–42
Time of attaining the maximum vanillin yield, minutes	180	180	90–150	20	60	–	60	9–15

To confirm the high yield of the product, we extracted vanillin from the entire volume of the reaction mixture, and then performed independent analyses using weight analysis, GLC, ^1H NMR. The results were slightly lower than the expected yield calculated from regularly withdrawn samples (1–3% based on lignin, as shown in Table 3). This difference may be due to partial evaporation of water during sampling of the hot reaction mixture from the reactor for analysis.

There is one probable reason for the yields exceeding the previously reported data: a high oxidation rate, which is of 10–20 times higher than the oxidation rate with nitrobenzene (Table 2). Native lignins are well known to be condensed during various processes of delignification and destruction, as well as during its isolation. Therefore, there is no reason to assume that destructive oxidation of lignins is not accompanied by side condensation reactions (Fig. 4). The scheme shows a 5-5 condensation, and any other possible type of condensation at the 5-position would decrease vanillin yield.

To explain the high yield of vanillin, one may assume that the rate of condensation is not strongly depended on the origin of the oxidant in the alkaline media and, therefore, is relatively low compared to oxidation to vanillin under the same conditions. Based on this assumption, we may conclude that the degree of lignin condensation decreases while decreasing the process duration and, hence, increasing the rate of oxidation. This decrease attained by using oxygen as an oxidant instead of nitrobenzene at high mass transfer intensity (stirring rate 1200 min^{-1}). As a result, the yield of vanillin, a competitive product, is higher than that obtained from nitrobenzene oxidation, due to high rate of oxidation. This conclusion is supported by a slight increase in maximum vanillin yield during flax shives oxidation with oxygen while increasing the stirring rate [11].

Table 3. Yield of vanillin extracted from the full volume of the reaction solution and quantified by GLC and ^1H NMR. Oxidation duration 15 min, for other conditions see Table 1

Size fraction	Extract mass after evaporation, g	Vanillin content (GLC), %	Vanillin yield (GLC), wt. % based on the initial lignin	Vanillin content (NMR), %	Vanillin yield (NMR), wt.% based on the initial lignin
0.5–1 mm	1.828	84.5	36.8	81.1	35.3
wet-milled for 5 min	1.791	71.7	28.9	72.3	29.2
wet-milled for 15 min	1.838	79.6	31.7	81.4	32.4

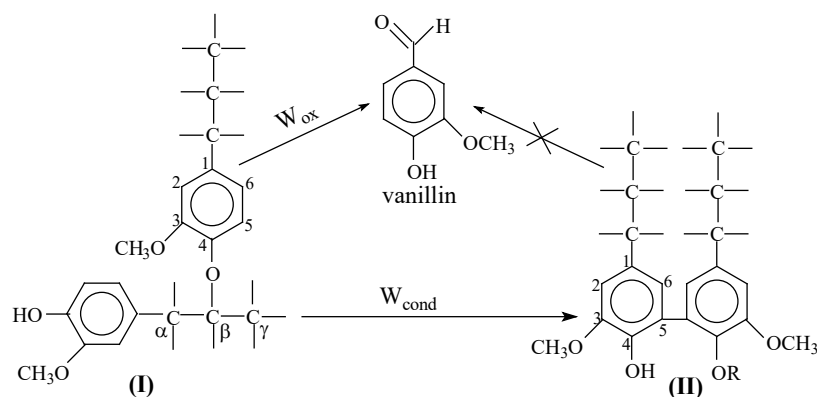


Fig. 4. Competition of the processes of destruction into vanillin and condensation during oxidation

The herein above described mechanical disintegration of the wood particles proceeds concurrently with vanillin formation over the entire course of the oxidation process. The disintegration proceeds faster as the initial wood particle size becomes smaller, while the vanillin formation rate in the time interval 3–10 minutes and its ultimate yield are practically independent of the wood size fraction (Fig. 3a). Such dynamics of the solid particle disruption and of vanillin accumulation do not contradict the concept of reaction-controlled kinetics: there is no simple linear connection between mechanical disintegration dynamics and the kinetics of vanillin accumulation or oxygen consumption.

Conclusions

In order to clarify the influence of mass transfer on the native lignin oxidation in the three-phase gas-liquid-solid system, the data on the initial state of the wood solid particles (principally, their size) and their degradation during the oxidation to vanillin were obtained. The present study demonstrated the absence of influence of the wood particle size (fractions less than 2 mm) upon the rate of their oxidation into vanillin. Chemical destruction and mechanical disruption of the wood particles during the oxidation proceeds concurrently with formation of vanillin during the entirety of the process, and the disintegration is faster for finer size fractions of the initial wood.

Final vanillin yield in oxidation of powdered pine wood (up to 35–36 wt.% based on the initial lignin) and the total yield of monomeric phenols of 38–42% are practically independent of the feedstock particle size distribution (<2 mm). The highest recorded vanillin yields exceed the classic and modern results of pine wood oxidation with nitrobenzene, and the main reason of this advantage is a high oxidation rate with oxygen, which is of 10–20 times higher than the rate of nitrobenzene oxidation. Such ratio of the rates decreases parallel condensation of lignin into structures which cannot produce vanillin under the conditions of fast oxidation with oxygen and, hence, increases the vanillin yield compared to one of slow oxidation with nitrobenzene. The presented results indicate that in order to attain high vanillin yield through native lignin oxidation with oxygen, it is not necessary to thoroughly grind the wood. It is sufficient to use sawdust with a fineness of 2 mm or less. Moreover, vibratory or ball mills are not recommended for wood grinding to produce vanillin, as the wet-milled wood powders can lose their porous anatomical structure and therefore become resistant to oxidation.

Supplementary Information

The electronic supplement to the article (DOI: <http://www.doi.org/10.14258/jcprm.20260319369s>) provides additional experimental material that reveals the main points set out in the article.

Acknowledgments

The equipment of Krasnoyarsk Regional Research Equipment Centre of SB RAS was used in the experiments. The authors thank Nikolay Tarabanko for valuable technical assistance.

Funding

This research was supported by the Russian Science Foundation, grant No. 25-26-20104, and by Krasnoyarsk Regional Fund of Science (No. 42).

Conflict of Interest

The authors of this work declare that they have no conflicts of interest.

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Received May 7, 2026

Revised September 10, 2026

Accepted September 14, 2026

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