

Enhancement of Removal of VOCs and Odors from Wood by Microwave-Activated Persulfate

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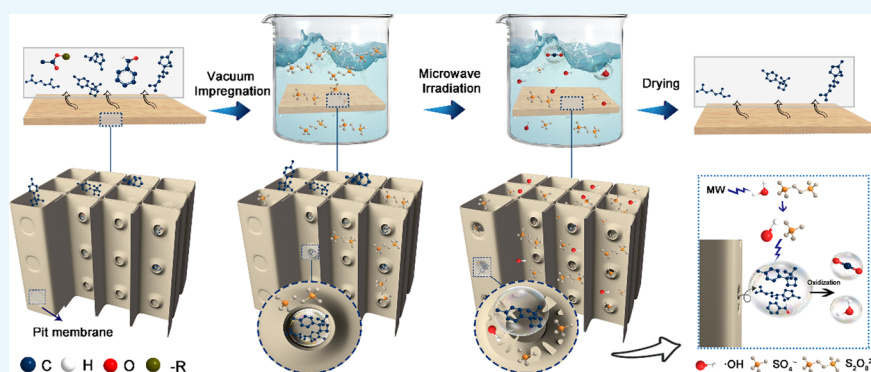
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ABSTRACT: Since traditional methods for removing volatile organic compounds (VOCs) from wood consume large amounts of energy and generate environmental pollution, it is desired to develop a convenient and green treatment method. Oxidation by microwave-activated persulfate (MW-PS) is a promising alternative method that has been used to eliminate VOCs from wood. The penetration of microwave energy can destroy the wood pit membranes and increase VOC emissions. The VOCs are further degraded by $\cdot\text{OH}$ and $\text{SO}_4^{\cdot-}$, which are generated *via* the activation of microwaves. This phenomenon can be detected by the electron paramagnetic resonance spectrometry. The 35 types of main VOCs of natural wood were determined, including alkanes/terpenes, alcohols/ethers, esters, aldehydes/ketones, and others. In the MW-PS system, 23 compounds were removed with an efficiency of 100%. Specifically, as one of the major compounds, the content of alkanes/terpenes was sharply decreased, and no alcohols/ethers and esters were detected. It was found that the optimal conditions of the MW-PS system for the minimum release of VOCs from wood were the microwave power of 462 W, irradiation time of 30 min, and PS dosage of 0.5 mmol/L.

1. INTRODUCTION

Wood has deservedly been popular as the main raw material used in furniture manufacturing and interior decoration and is increasingly popular because of its green characteristics, mild tactility, and visual effect; however, volatile organic compounds (VOCs) with unpleasant odors are emitted from wood, which contribute to indoor air pollution. The risk of sick building syndrome is increased by the polluted indoor air, which affects work efficiency and quality of life.^{1–3} Therefore, the removal of VOCs with unpleasant odors from wood is of great significance for furniture manufacturing and production.

The VOCs emitted from extractives in wood pits⁴ have a complex composition that includes alkenes, alcohols, aldehydes, ketones, and terpenes.^{5,6} Heat treatment and extraction methods are typically used to remove VOCs from wood. Heat treatment effectively removes some VOCs (such as terpenes and aldehydes), which are small molecules that are highly sensitive to temperature.^{7,8} However, these methods require high energy consumption, and it is difficult to remove all VOCs from wood. Extraction is often performed using an organic solvent (such as ethanol–toluene, ethanol–cyclo-

hexane, *etc.*) to extract VOC compounds from wood.⁹ Solvents are toxic and remain in the treated wood, and the liquid organic solvent waste is difficult to be disposed of in actual production processes. Therefore, it is necessary to develop environment-friendly and energy-conserving methods to remove VOCs from wood.

Advanced oxidation processes (AOPs) have been proposed as environment-friendly approaches to effectively and non-selectively eliminate a wide range of organic contaminants.^{10–12} AOPs have been used to treat aqueous solutions,¹³ polluted gases,¹⁴ and contaminated solid matrices.¹⁵ AOPs rapidly generate active free radicals, including sulfate radicals ($\text{SO}_4^{\cdot-}$),^{16,17} ($\cdot\text{OH}$),^{18,19} and ($\text{O}_2^{\cdot-}$),^{20,21} which can degrade

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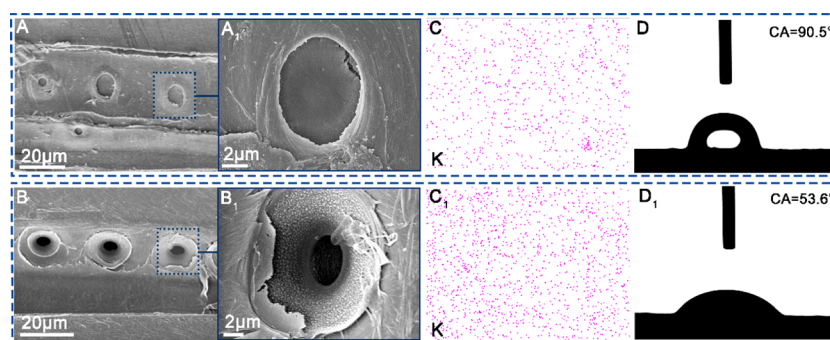


Figure 1. (A) SEM image of the pits in the natural wood. (A₁) High-resolution SEM image of the pit membranes in the natural wood. (B) SEM image of the pits in the MW-PS treated wood. (B₁) High-resolution SEM image of the pit membrane in the MW-PS treated wood. (C, C₁) Elemental mappings of K in the wood immersed in PS without and with the MW treatment, respectively. (D, D₁) Contact angles of the natural wood and MW-PS-treated wood on the tangential section, respectively. The reaction conditions of the MW-PS system: [MW power] = 462 W, [time] = 30 min, and [PS dosage] = 0.5 mmol/L.

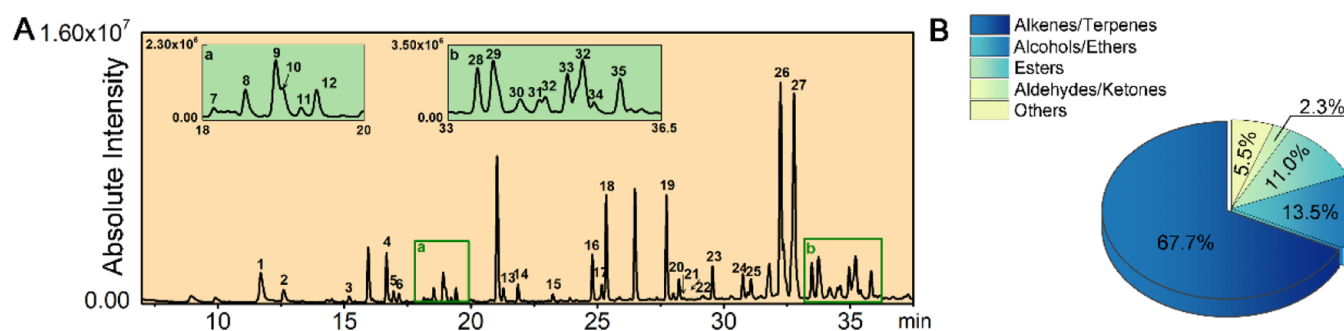


Figure 2. (A) GC-MS total ion chromatograms of natural wood. [insert: (a) detail image of ion chromatograms in 18–20 min and (b) detail image of ion chromatograms in 33–36.5 min] (B) Percentages of the main VOCs in natural wood.

many organic substances. Based on their powerful oxidative capacities, a higher redox potential (2.5–3.1 V), a longer lifetime (~ 4 s), and a wider pH range (2–8), $\text{SO}_4^{\bullet-}$ -based AOPs have gained attention as an alternative method. $\text{SO}_4^{\bullet-}$ is typically generated from persulfate (PS, $\text{S}_2\text{O}_8^{2-}$). Due to the excellent stability of PS, it can be activated in various ways, including heat,¹² microwave irradiation,^{15,22} ultrasonication,²³ and transition metals.²⁴ Meanwhile, PS stably exists in the matrixes until it is activated for *in situ* chemical oxidation.²⁵

Among the activation methods, microwave (MW) activation is highly energy-efficient and provides a rapid and selective method that can be used for volumetric heating.²⁶ MW irradiation, whose frequency ranges from 300 MHz to 300 GHz, is a band of the electromagnetic spectrum that directly interacts with materials using penetration, reflection, and absorption.²² Both H_2O and PS with high dielectric loss factor (ϵ'') values as the solvent and oxidant, respectively, have ultrafast realignment rates, which allows the temperature of a solution to be instantaneously and efficiently increased.²⁷ In addition, due to the dielectric properties of wood,²⁸ the MW technology is widely used to treat wooden materials because it increases the wood permeability by damaging the wood microstructure.^{29,30}

This study provides the strategy of using microwave-activated persulfate (MW-PS) as a green, low energy, and efficient method, to efficiently degrade VOCs that produce unpleasant odors in wood. The wood microstructures before and after the MW treatment were analyzed by scanning electron microscopy (SEM). Furthermore, the degradation effects of MW-PS using different processing factors (MW

power, treatment time, and PS dosage) were investigated by HS-SPME/gas chromatography–mass spectrometry (GC-MS). Thereafter, reactive radicals were identified by electron paramagnetic resonance (EPR) spectroscopy, and the properties of wood samples were discussed. The results of this study can significantly accelerate the development of green productions for the wood industry and the furniture manufacturing industry.

2. RESULTS AND DISCUSSION

2.1. Effect of MWs on Wood. The changes in pit membranes after the MW treatment were verified by SEM. Intact pit membranes in natural wood were formed by amorphous materials, which encrusted and blocked the margo (Figure 1A, A₁). Thereby, the migration of the VOCs and PS was hindered. As presented in Figure 1B, B₁, the pit membranes after the MW treatment were severely damaged and ruptured. This was attributed to the evaporation of water in the wood cells due to the instantaneous increase in the temperature by MWs,³¹ which generated a steam pressure that destroyed the pit membranes; thus, there were no barriers to transfer the liquids. From the energy-dispersive X-ray spectroscopy (EDS) elemental mappings (Figure 1C, C₁), the concentration of K^+ was higher than that in the non-treated wood, indicating that more PS was immersed into the wood. Therefore, once activated, the PS produced more free radicals to promote *in situ* chemical oxidation inside the wood, which helped degrade the VOCs. This was further verified by the surface wettability measured by the contact angles (CAs) (Figure 1D, D₁) and the permeability expressed by the curves

Table 1. Compounds in Samples Detected with SPME/GC–MS^a

peak number (no.)	compounds	peak area		
		control	MW	MW-PS
1	toluene	16,554,755	-	-
2	hexanal	5,302,709	1,258,401	1,297,210
3	1,3,5,7-cyclooctatetraene	2,086,032	-	-
4	tri(propylene glycol)propyl ether	14,064,500	-	-
5	butanoic acid, 3-hydroxy-, ethyl ester	3,664,735	-	-
6	β -myrcene	2,457,581	1,485,968	394,840
7	5-hepten-2-one, 6-methyl-	686,079	-	-
8	butanoic acid, 4-methoxy-, methyl ester	3,725,129	-	-
9	D-limonene	8,551,164	14,420,323	-
10	<i>o</i> -cymene	3,728,945	4,864,375	-
11	bicyclo[3.1.0]hexane, 4-methylene-1-(1-methylethyl)-	1,148,663	1,552,723	-
12	1-hexanol, 2-ethyl-	3,799,591	-	-
13	<i>p</i> -(1-propenyl)-toluene	3,624,495	-	2,399,221
14	nonanal	4,663,082	1,566,191	2,178,299
15	fenchol	2,224,215	-	-
16	3-cyclohexen-1-ol, 4-methyl-1-(1-methylethyl)-, (R)-	11,802,444	-	-
17	endo-borneol	3,386,530	786,544	-
18	α -terpineol	27,258,169	1,212,044	-
19	cyclohexanol, 5-methyl-2-(1-methylethyl)-, acetate	27,924,312	-	-
20	bicyclo[2.2.1]heptan-2-ol, 1,7,7-trimethyl-, acetate, (1S-endo)-	1,671,263	505,377	-
21	methyl 6,6-dimethylbicyclo[3.1.1]hept-2-ene-2-carboxylate	5,790,067	811,588	-
22	tetradecane	1,348,504	-	370,581
23	α -terpinyl acetate	8,690,474	721,914	-
24	cyclohexane, 1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-, [1S-(1 α ,2 β ,4 β)]-	7,715,820	1,869,403	655,595
25	di-epi- α -cedrene-(1)	6,673,312	6,015,937	3,727,860
26	1H-3a,7-methanoazulene, 2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3 α ,3 $\alpha\beta$,7 β ,8 $\alpha\alpha$)]-	96,758,918	37,696,192	35,659,334
27	bicyclo[4.4.0]dec-1-ene, 2-isopropyl-5-methyl-9-methylene-	91,294,126	15,688,141	-
28	benzene, 1-(1,5-dimethyl-4-hexenyl)-4-methyl-	11,856,528	15,688,141	431,954
29	(1R,4R,5S)-1,8-dimethyl-4-(prop-1-en-2-yl)spiro[4.5]dec-7-ene	48,154,130	3,269,618	1,146,267
30	bicyclo[7.2.0]undec-4-ene, 4,11,11-trimethyl-8-methylene-	6,275,903	-	-
31	α -muurolene	379,1428	-	-
32	(1R,5S)-1,8-dimethyl-4-(propan-2-ylidene)spiro[4.5]dec-7-ene	4,593,770	1,704,517	-
33	naphthalene, decahydro-4a-methyl-1-methylene-7-(1-methylethenyl)-, [4aR-(4 $\alpha\alpha$,7 α ,8 $\alpha\beta$)]-	11,418,933	1,237,510	377,171
34	naphthalene, 1,2,3,4,4a,5,6,8a-octahydro-7-methyl-4-methylene-1-(1-methylethyl)-, (1 α ,4 $\alpha\beta$,8 $\alpha\alpha$)-	3,400,089	-	-
35	benzene, 1-methyl-4-(1,2,2-trimethylcyclopentyl)-, (R)- TVOCs	9,750,346 46,124,2971	1,572,828 113,927,735	439,704 49,078,036

^aNote: “-” indicates that the relative content of the compound is lower than the detection limit.

of dynamic CAs over time (Figure S1). As a porous material, natural wood exhibits a wettability with an initial CA of $\sim 90^\circ$ and completely absorbs the water in about ~ 12 s. After the MW treatment, the surface wettability was improved with a CA of $\sim 53^\circ$. Additionally, the CAs on the tangential section of the MW-PS sharply decreased in less than 1 s. These findings demonstrated that the MW-PS made the wood sample with excellent surface wettability and permeability; therefore, the released VOCs were concurrently oxidized by MWs into carbon dioxide and water. The tiny cracks in the wood sample increased the wood permeability during the MW process, which accelerated the transfer of more free radicals to the wood interior for oxidation.

2.2. Identification of VOCs Emitted from Natural Wood. The VOC composition in wood is complex, and the limitations of the detection method prevented some substances from being determined; therefore, the common VOCs in many samples were selected as the main VOCs in the following analysis. The main VOCs, about 35 compounds, were

identified in the control (Figure 2A) and were numbered according to their name (Table 1). As shown in Figure 2B, 35 main VOCs were collected, including alkanes/terpenes, alcohols/ethers, esters, aldehydes/ketones, and others. Alkenes/terpenes were the major components and occupied 67.7% of the main VOCs, which was coincident with the previous study.²⁸ Some terpenes were odorous, such as no. 6 (β -myrcene), no. 9 (D-limonene), no. 24 (cyclohexane, 1-ethenyl-1-methyl-2,4-bis(1-methylethenyl)-, [1S-(1 α ,2 β ,4 β)]-), no. 26 (1H-3a,7-methanoazulene, 2,3,4,7,8,8a-hexahydro-3,6,8,8-tetramethyl-, [3R-(3 α ,3 $\alpha\beta$,7 β ,8 $\alpha\alpha$)]-), and no. 30 (bicyclo[7.2.0]undec-4-ene, 4,11,11-trimethyl-8-methylene-). They also pose increased health risks.^{32,33} Alcohols/ethers (13.5% of main VOCs) and esters (11.0% of main VOCs) also emit unpleasant odors and cause physiological effects. These compounds included no. 4 (tri(propylene glycol) propyl ether), no. 12 (1-hexanol, 2-ethyl-), and no. 17 (endo-borneol). Aldehydes/ketones (5.5%

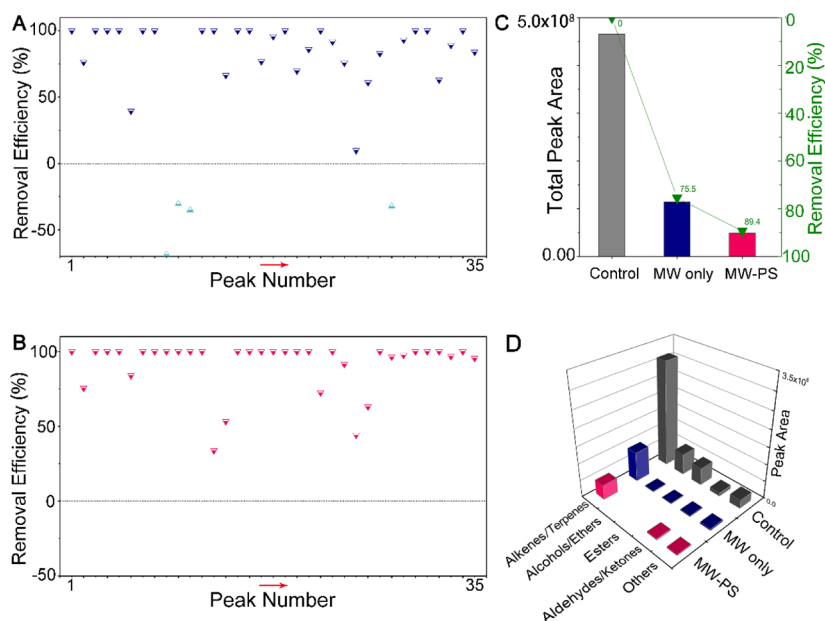


Figure 3. Removal efficiency of each compound in the samples treated by MW only (A) and MW-PS (B). (C) Comparison of the total contents of the main VOCs and the removal efficiency of major compounds in the control, MW-only, and MW-PS samples using the base peak area. (D) Bar graph comparison of the categories of main VOCs in the control, MW-only, and MW-PS samples based on the peak areas.

of main VOCs) and others (2.3% of main VOCs) accounted for a smaller percentage.

2.3. Effect of the MW-PS System. According to the chromatograms, the VOCs treated by MW only (Figure S2) were efficiently removed after the 30 min treatment. Specifically, 100% of 15 compounds in the MW-only samples were removed, and almost 50% of 14 other compounds were removed. In contrast, the content of four compounds increased after the MW-only treatment (Figure 3A). Meanwhile, the peak area of the main VOCs was used to compare the total VOCs (TVOCs) among the samples. It was observed that the TVOC removal of the MW-only sample was 75% in Figure 3C; meanwhile, as illustrated in Figure 3D, the content of each category decreased. As the major reaction medium, the polar structure of H₂O molecules, which have a large dielectric constant, can efficiently absorb microwaves to rapidly increase the temperature at a rate of billions of degrees per second, causing the emission of temperature-sensitive VOCs, such as no. 2 (hexanal), no. 14 (nonanal), and some terpenes.³⁴ The higher temperature induced by the MW can increase the collision of molecules and may also accelerate evaporation or oxidation by damaging the pits and rearranging molecules.³⁵ Specifically, the athermal effect of the MW promoted the realignment of molecules within 30 min; thus, alkenes and terpenes were readily emitted from the wood without a high temperature. Moreover, H₂O molecules could generate •OH as the major reactive species by the MW irradiation. It was further verified by EPR spectroscopy that the characteristics of •OH using the reagent of DMPO produced strong lines in the four-line EPR spectra with an intensity ratio of 1:2:2:1 (Figure 4, eq 1). The •OH radical performed H-atom abstraction, isomerization, and double bond cleavage, which were responsible for the sharp decline in the alkene and terpene contents;³⁶ however, no. 10 and no. 11 were detected in higher amounts due to the degradation of limonene.³⁵

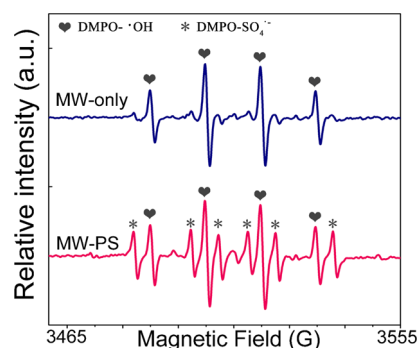
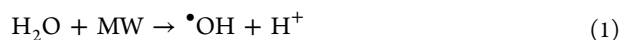


Figure 4. EPR spectra of MW-activated water and MW-activated PS with DMPO.

As expected, when the PS was activated by the MW, the removal efficiency of each VOC further increased (Figure S3). As presented in Figure 3B, the removal efficiency of all main VOCs (35 compounds) was above 33%. Specifically, seven compounds were removed up to 75%, and 23 compounds were removed up to 100%. Furthermore, the TVOCs of MW-PS was calculated as shown in Figure 3C, demonstrating that the TVOCs in the MW-PS system decreased to 89.4%. As shown in Figure 3D, MW-PS improved the degradation of alkanes/terpenes, aldehydes/ketones, and others. No alcohols/ethers and esters were detected. As for the odorous compounds mentioned above, MW-PS exhibited an excellent removal capacity. The contents of no. 2 (hexanal) and no. 14 (nonanal) increased due to the further oxidation. The O–O bond of PS, with a bond length of 1.497 Å, was easily ruptured by the MW to generate SO₄^{•-} via eqs 2 and 3.³⁷ As shown in Figure 4, it was further verified by EPR spectroscopy that the characteristics of SO₄^{•-} using the reagent of DMPO produced strong lines with an intensity ratio of 1:1:1:1:1. Because of its higher redox potential, SO₄^{•-} possessed a better degradation ability and continuously eliminated VOCs.³⁸ It was shown that the MW-PS system can efficiently eliminate VOCs from wood.

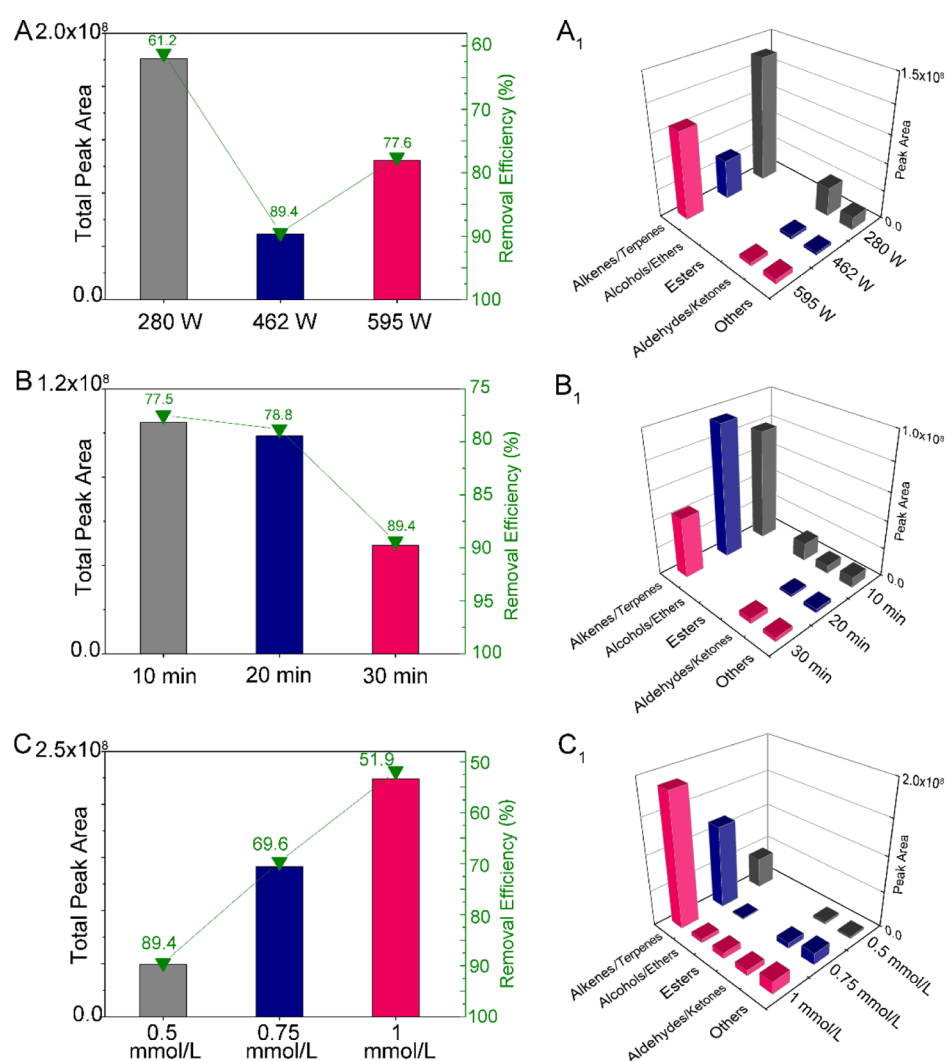
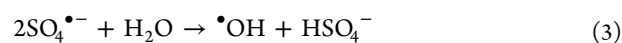


Figure 5. Effect of the MW-PS system with different MW powers on the TVOC removal efficiency (A) and the removal of different categories (A₁). The effect of the MW-PS system with different treatment times on the TVOC removal efficiency (B) and the removal of different categories (B₁). The effect of the MW-PS system with different PS concentrations on the TVOC removal efficiency (C) and the removal of different categories (C₁). The specific reaction conditions are shown in Table S1.



2.4. Effect of Different Reaction Conditions. Since the removal efficiency of VOCs was significantly influenced by the MW power, treatment time, and PS dosage, the effect of different conditions was investigated, and the results are shown in Figure 5 and Tables S2–S4.

The effect of the MW-PS system was identified by the previous analysis to control the VOC emission. Furthermore, the VOC removal under different MW powers was investigated, and the results are presented in Figure 5A,A₁. The various MW powers led to different removal efficiencies. The removal efficiency increased from 61.2 to 89.4% upon the increase of the MW power from 280 to 462 W. Meanwhile, the emission of alkenes/terpenes, aldehydes/ketones, and VOCs obviously declined. The concentration of alcohols/ethers and esters was lower than the detection limit. Nevertheless, as the power further increased, the TVOC emission increased, and the removal efficiency reached 77.6%. When the MW power was 280 W, the pits were inadequately damaged (Figure S4)

and the VOCs were not sufficiently exposed, which prevented them from being degraded by radicals. Moreover, the better removal of TVOCs at a power of 462 W was partly ascribed to the dielectric loss tangent. An increase in the MW power enhanced the degradation efficiency because the material rapidly converted the MW into heat.³⁹ However, a superabundance of sulfate radicals was generated at a high MW power (595 W) in a short period of time, which led to the self-decomposition of sulfate radicals.⁴⁰

The reaction time played a key role in promoting VOC removal, as shown by the results in Figure 5B,B₁. The VOC emission declined upon an increase in the irradiation time, with removal efficiencies of 77.5% ($t = 10$ min), 78.8% ($t = 20$ min), and 89.4% ($t = 30$ min). The alkenes/terpenes increased after 20 min and then sharply decreased after 30 min. Additionally, the ester content decreased as the time increased. The removal of aldehydes/ketones and other VOCs followed a similar trend to that of alkenes and terpenes. It was ascribed to the fact that free radicals were continuously generated and the pits were destroyed, thereby promoting the degradation of TVOCs. The slow degradation of TVOCs within 20 min was attributed to the fact that the water and wood absorbed the

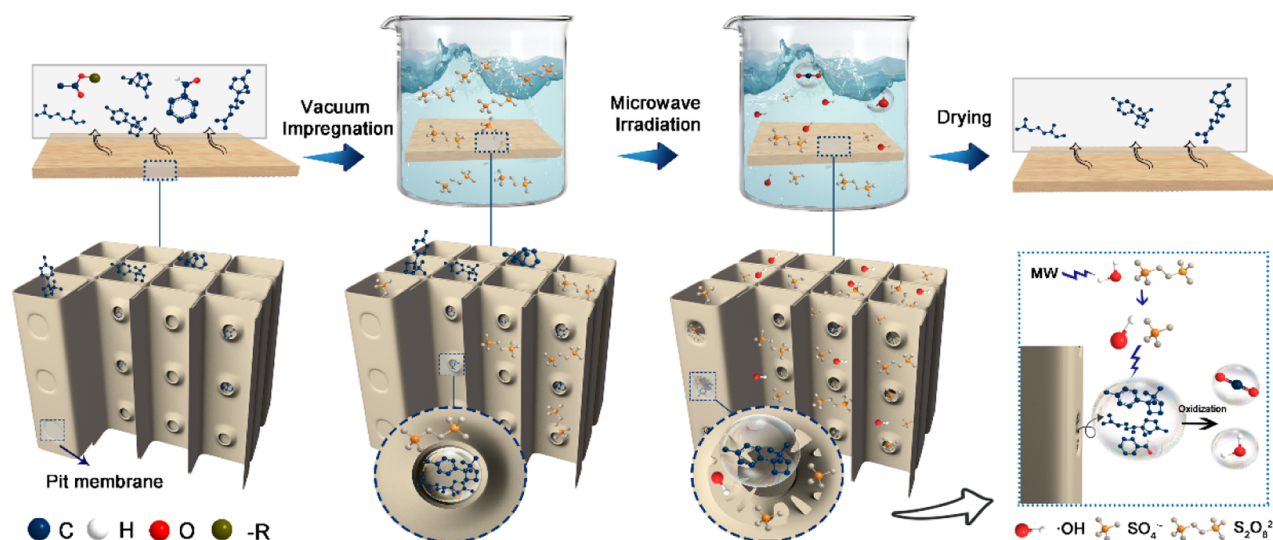
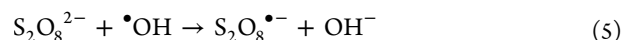


Figure 6. Schematic diagram of the MW-PS process used to degrade VOCs in wood.

MWs and converted them into thermal energy, the radicals were only partly generated, and the pit membranes could not be efficiently damaged. Therefore, the TVOCs were inadequately oxidized.

Besides the MW power and reaction time, the PS dosage also plays a significant role in removing VOCs using the MW-PS system. Figure 5C,C₁ illustrate that the PS concentration remarkably influenced the VOC release from wood. The TVOCs increased upon the addition of PS. Overall, 89.4% of the ethyl parathion was removed at a PS concentration of 0.5 mmol/L, compared with 69.6 and 51.9% after the addition of 0.75 and 1 mmol/L PS, respectively. The 3D column visually indicated that the content of alkenes/terpenes increased at higher PS concentrations. Besides, the increase in PS dosage increased the emission of alcohols, ethers, and esters. The decomposition of PS was affected by its concentration under the microwave irradiation. Once activated, PS reached saturation at higher PS dosages, which quenched sulfate radicals (eq 4). Consequently, excess sulfate radicals decreased the oxidation rate of VOCs. Simultaneously, based on eq 5, the sulfate radicals quenched and further consumed the $\cdot\text{OH}$.



2.5. Degradation Mechanism of VOCs from Wood in the MW-PS System. Based on the observations from the SEM, EPR, and GCMS, the degradation mechanism is illustrated in Figure 6. The wood sample was preliminarily impregnated in the PS solution by a vacuum impregnation process; thus, the $\text{S}_2\text{O}_8^{2-}$ remained in the wood. Furthermore, MW irradiation generated the “thermal” and “athermal” effects to promote the reaction. To be specific, (1) the pit membranes of the wood were damaged, causing VOCs to be released from pits and the permeability of wood to be enhanced; (2) the O–O bond of $\text{S}_2\text{O}_8^{2-}$ was easily ruptured to generate $\text{SO}_4^{\bullet-}$; (3) H_2O molecules can absorb the MW to instantaneously and efficiently increase the temperature and generate $\cdot\text{OH}$; and (4) VOCs absorbed the MW energy to accomplish bond breaking of molecules. Therefore, the MW-PS system achieved *in situ* chemical oxidation by means of $\text{SO}_4^{\bullet-}$ and $\cdot\text{OH}$ (H-atom abstraction, electron transfer, and addition reaction). In

addition, the increasing permeability accelerated the infiltration of free radicals in the solution to enhance oxidation efficiency. Consequently, VOCs accomplished self-degradation and mineralization by molecule realignment and free radical oxidation.

3. CONCLUSIONS

This study presented that MWs can destroy the pit membranes in wood to promote VOC emissions and PS permeation. Furthermore, $\cdot\text{OH}$ and $\text{SO}_4^{\bullet-}$ are the major radicals produced by MW activation, and they further oxidized the VOCs in the wood. The VOC content was efficiently decreased using the MW-PS system with a removal efficiency of 89.4% compared with that of natural wood. Meanwhile, the 23 compounds of main VOCs were removed by up to 100%. The MW-PS system with various conditions (MW power, reaction time, and PS dosage) was investigated. Increasing the MW power caused the TVOCs to increase at first and then decrease. A reduction in the TVOCs was facilitated by the increase in irradiation time; however, a higher PS concentration hindered the degradation of TVOCs due to the scavenging termination of radicals. Therefore, the optimal parameters were the MW power of 462 W, irradiation time of 30 min, and PS dosage of 0.5 mmol/L.

4. MATERIAL AND METHODS

4.1. Materials. Chinese fir [*Cunninghamia lanceolata* (Lamb.) Hook] lumber was obtained from the Henan Province. Potassium persulfate ($\text{K}_2\text{S}_2\text{O}_8$, AR, 99.5%), 5,5-dimethyl-1-pyrroline N-oxide (DMPO, AR, 97%), potassium iodide (KI, AR, 99%), and sodium hydrogen carbonate (NaHCO_3 , AR, 99.8%) were purchased from McLean Biochemical Technology Co., Ltd. (Shanghai, China). All chemicals were dissolved in deionized water [deionized (DI) water].

4.2. Methods. The wood bulk (10 g, $30 \times 20 \times 5$ mm) was weighed in a 500 mL beaker, followed by the addition of 300 mL of different amounts of $\text{K}_2\text{S}_2\text{O}_8$ (0.5, 0.75, and 1 mmol/L) dissolved in DI water. The samples were stirred at a 200 rpm, and the beaker was placed into a vacuum oven for impregnation (DZF-6050, Made in Yiheng Co., Shanghai China) at 0.1 MPa and room temperature for 2 h. After the impregnation, the beaker was treated in a MW oven

(MM721AAU-PW, Made in Media Co., China) operated at different output powers (280, 462, and 595 W) with a frequency of 2.45 GHz for 10, 20, and 30 min, respectively. After the MW irradiation, the treated wood was freeze-dried to absolute dryness. The different treatment conditions of samples are shown in Table S1.

4.3. HS-SPME/GC–MS Analysis. The VOCs were analyzed by an agitator/incubator with an automatic headspace sampler (GCMS-TQ8040 NX, Shimadzu, Japan). A 1 g sample of wood was placed into a 20 mL sealed headspace vial, which was incubated at 70 °C for 30 min and then collected with a DVB/CAR/PDMS 50/30 μm fiber with a length of 10 mm (Supelco, Bellefonte, USA). After collection, the fiber was then inserted into the inlet of the GC–MS system, and a desorption step was performed at 250 °C for 3 min. The volatiles were separated on a 30 m × 0.25 mm × 0.25 μm DB-624 capillary column (Agilent, CA, USA) with a carrier gas at a constant flow rate of 1.0 mL/min. The heating program was as follows: 60 °C held for 1 min, heated to 140 °C at a rate of 20 °C/min, held for 5 min, heated to 230 °C at a rate of 10 °C/min, and held for 10 min. MS was performed in the EI mode over the *m/z* range of 30–550. The volatile components were identified by the GCMS Postrun Analysis software by matching mass spectra using commercial libraries (NIST17s, NIST17-1, and NIST17-2). Each peak was checked by determining the area on the GC–MS chromatogram.^{41–43}

4.4. Characterization. Before and after treating the wood, the micromorphologies were characterized by SEM (NovaTM Nano SEM 250, FEI, USA). EDS was used to determine the surface elemental distribution of samples. Free radicals were trapped with DMPO and detected with EPR (EMXPLUS10/12, Bruker, USA). The water CAs and permeability of samples were dynamically measured within 30 s by a CA goniometer (OCA20, DataPhysics, Germany) at ambient temperature, using a droplet volume of 3 μL.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acsomega.1c00126>.

Reaction conditions of the MW-PS system, tables of GCMS, and figures of characterizations (PDF)

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Notes

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