Control of malodorous gases emission from wet-end white water with hydrogen peroxide

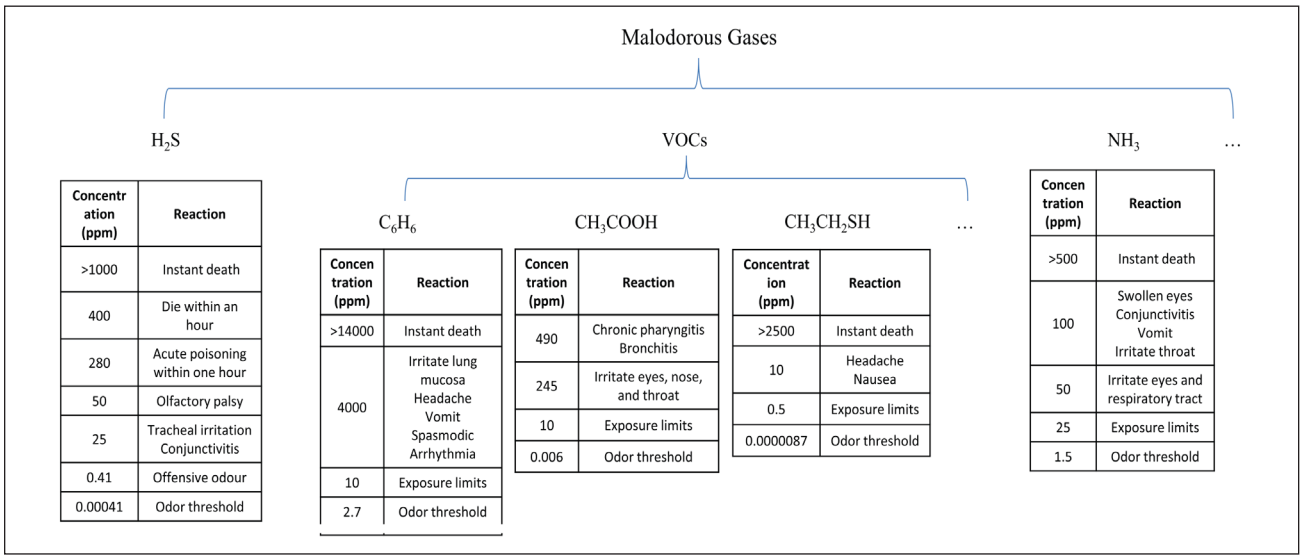
HUANG SHAN-CONG, LIU CHANG, DAI LEI, DOU SHENG, DING MING-QI, AND XIA XIN-XING

ABSTRACT: White water is highly recycled in the papermaking process so that its quality is easily deteriorated, thus producing lots of malodorous gases that are extremely harmful to human health and the environment. In this paper, the effect of hydrogen peroxide (H_2O_2) on the control of malodorous gases released from white water was investigated. The results showed that the released amount of total volatile organic compounds (TVOC) decreased gradually with the increase of H_2O_2 dosage. Specifically, the TVOC emission reached the minimum as the H_2O_2 dosage was 1.5 mmol/L, and meanwhile, the hydrogen sulfide (H_2S) and ammonia (NH_3) were almost completely removed. It was also found that pH had little effect on the release of TVOC as H_2O_2 was added, but it evidently affected the release of H_2S and NH_3. When the pH value of the white water was changed to 4.0 or 9.0, the emission of TVOC decreased slightly, while both H_2S and NH_3 were completely removed in both cases. The ferrous ions (Fe^{2+}) and the copper ions (Cu^{2+}) were found to promote the generation of hydroxyl radicals (HO•) out of H_2O_2, enhancing its inhibition on the release of malodorous gases from white water. The Fe^{2+}/H_2O_2 system and Cu^{2+}/H_2O_2 system exhibited similar efficiency in inhibiting the TVOC releasing, whereas the Cu^{2+}/H_2O_2 system showed better performance in removing H_2S and NH_3.

Application: This study shows that adding oxides to white water can control the release of malodorous gases. This has a positive effect on paper mills to limit the release of odor.

Air pollution is a serious global issue at present, and thus tremendous effort has been put into controlling gas emissions to reduce their impact on the environment and human health [1]. In particular, the paper industry emits lots of waste gas that is difficult to control, because these malodorous gases are of complex types, including volatile organic compounds (VOCs), hydrogen sulfide (H_2S), and ammonia (NH_3), etc. [2,3]. Emission of these gases causes different environmental problems, such as the greenhouse effect, acid rain, and ozone depletion. Human beings exposed to these gases for a long time may have symptoms such as headache, nausea, and vomiting, as shown in **Fig. 1**. [4-7].

Different methods have been proposed to reduce the emissions of these malodorous gases, including physical (adsorption, condensation, washing, etc.); chemical (ther-



Concentration (ppm)	Reaction
>1000	Instant death
400	Die within an hour
280	Acute poisoning within one hour
50	Olfactory palsy
25	Tracheal irritation Conjunctivitis
0.41	Offensive odour
0.00041	Odor threshold

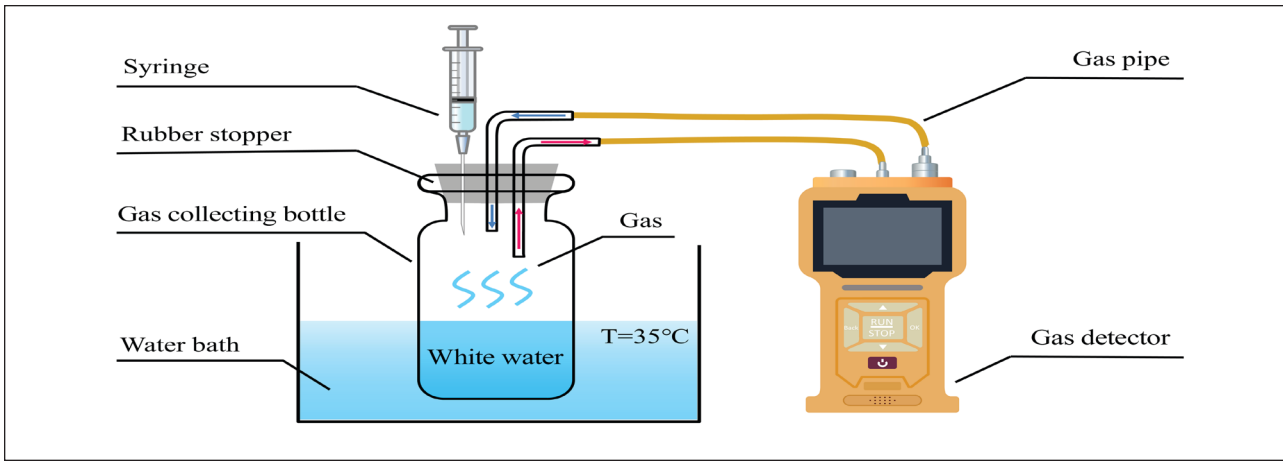
Concentration (ppm)	Reaction
>14000	Instant death
4000	Irritate lung mucosa Headache Vomit Spasmodic Arrhythmia
10	Exposure limits
2.7	Odor threshold

Concentration (ppm)	Reaction
490	Chronic pharyngitis Bronchitis
245	Irritate eyes, nose, and throat
10	Exposure limits
0.006	Odor threshold

Concentration (ppm)	Reaction
>2500	Instant death
10	Headache Nausea
0.5	Exposure limits
0.0000087	Odor threshold

Concentration (ppm)	Reaction
>500	Instant death
100	Swollen eyes Conjunctivitis Vomit Irritate throat
50	Irritate eyes and respiratory tract
25	Exposure limits
1.5	Odor threshold

1. Effect of malodorous gases concentration on human beings (VOCs = volatile organic compounds; H_2S = hydrogen sulfide; C_6H_6 = benzene; CH_3COOH = acetic acid; CH_3CH_2SH = ethanethiol; NH_3 = ammonia).



2. Schematic illustration of the experimental setup

mal oxidation, catalytic oxidation and oxidation, etc.); and biological (biofilters, bioscrubbers, and biotrickling filters, etc.) methods [8,9]. These malodorous gases can be largely removed by one of the previously mentioned methods or a combination thereof. However, these treatments are generally implemented at the tail end, which is risky. In this context, some researchers have tried to remove malodorous gases at the very beginning when they are just generated. Advanced oxidation processes (AOPs) relying on the generated hydroxyl radicals (HO•) are commonly used to treat industrial wastewater with the goal of improving the biodegradation capability of water. Generally, AOPs have good organic matter removal efficiency, with a significant decrease of chemical oxygen demand (COD) by over 40% [10], and they can also effectively remove malodorous gases. According to the research of Hohee et al. [11], the geosmin and 2-methylisopropanol (2-MIB) could be removed by 10% and 18%, respectively, via the ultraviolet (UV)/H_2O_2 treatment. In addition, the Fenton process, as one of the typical AOPs, not only reduces the chemical oxygen demand (COD) of wastewater, but also has a good control on VOCs emissions [12].

Due to the renewal of papermaking technology and the demand for sustainable development, the degree of closed circulation of white water is getting higher and higher. This leads to the continuous accumulation of organic matter, inorganic salts, and microorganisms in the white water, thus generating malodorous gases [13-16]. Although some researchers have proved that adding oxidants and biocides can control malodorous gases to some extent, the quantitative relationship between oxidants and malodorous gases has not been established [17,18].

Sodium hypochlorite (NaClO) and chlorine dioxide (ClO_2) are commonly used oxidants, but they will increase the chlorine content and conductivity of water, interfere with the chemical action of the system, and increase the corrosion of equipment. Hydrogen peroxide (H_2O_2) is an environmentally friendly oxidant with strong oxidative capacity. Therefore, H_2O_2 was used to treat white water, and the H_2O_2 dosage, pH, ferrous ions (Fe^{2+}), and copper ions (Cu^{2+}) were investigated to elucidate their influence on the control of malodorous gases and to establish the quantitative relationship between them. This work may provide guidance for controlling the release of malodorous gases from white water in the papermaking process.

MATERIALS AND METHODS

Materials

Wet-end white water was kindly provided by Zhejiang Jingxing Paper Joint Stock Co. Ltd. (Zhejiang, China), which uses old corrugated containers (OCC) as raw material to produce bobbin paper. Hydrogen peroxide (AR grade), ferrous sulfate heptahydrate ($FeSO_4{\cdot}7H_2O$, AR grade), copper sulfate pentahydrate ($CuSO_4{\cdot}5H_2O$, AR grade), sodium hydroxide (NaOH, AR grade), hydrochloric acid (HCl, AR grade), and other chemical reagents were purchased from Sinopharm Chemical Reagent Co. Ltd. (Shanghai, China).

Experimental methods and devices

The white water from the wet end was collected and stored at 5°C. For each analysis, 100 mL of white water was placed in a 250 mL gas collecting bottle. The effect of H_2O_2 dos-

Temperature, °C	Concentration, wt%	pH	Conductivity, ms/cm	CODcr, mg/L	VFA, mmol/L
45	0.498	6.35	5.37	8110	61.6

CODcr = chemical oxygen demand; VFA = volatile fatty acids.

I. Physical and chemical properties of the wet-end white water.

age, pH, Fe^{2+}, and Cu^{2+} on the amount of malodorous gases released from white water was investigated. The setup is shown in **Fig. 2**. The temperature was maintained at 35°C with water bath. A syringe was used to add reagents. The pH value was adjusted with HCl and NaOH.

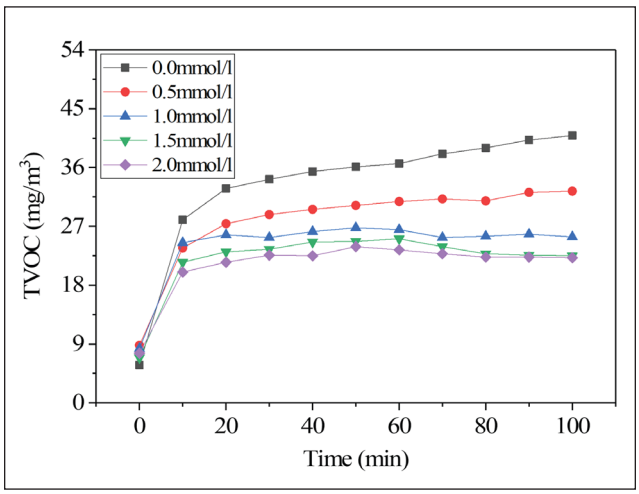
Analytical method

The white water concentration was measured with the filter paper filtration method. Specifically, 100 mL of white water was filtered with filter paper to obtain a filterable solid. The filtered solid was dried at 105°C, and the collected solid was used to calculate the white water concentration. The pH and conductivity of white water were measured with a pH meter (PHS-25, Shanghai Yidian Scientific Instrument Co. Ltd.; Tianjin, China) and conductivity meter (DDS-307, Shanghai Yidian Scientific Instrument Co. Ltd.). The measurement of volatile fatty acids (VFA) was implemented according to the method reported by Huang et al. [19]. Chemical oxygen demand (CODcr) was measured according to Chinese National standard HJ 82-2017 "Water quality—determination of the chemical oxygen demand—dichromate method." The gas detector (JA908-VOC-PID, JA908-H2S, JA908-NH3, Shenzhen Ziyuan Environmental Protection Technology Co. Ltd.; Guangdong, China) was used to determine the gas concentration.

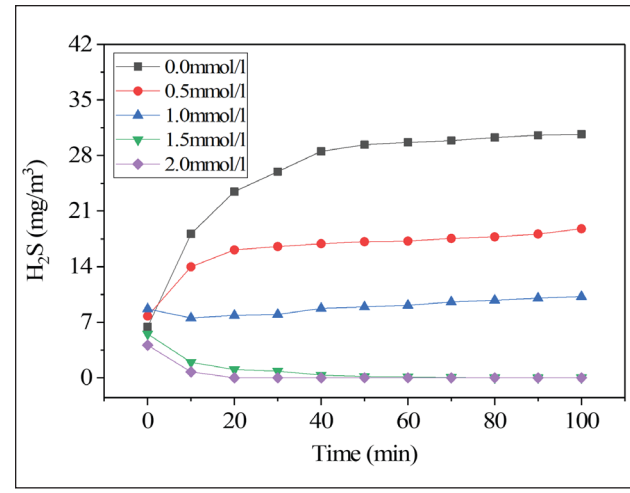
RESULTS AND DISCUSSION

Analysis of wet-end white water

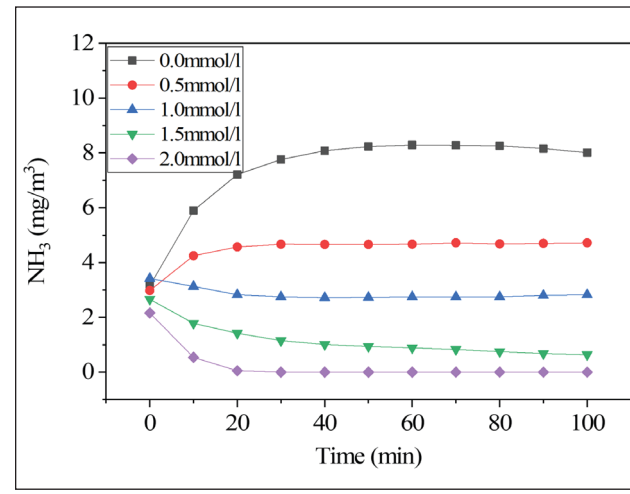
The basic properties of wet-end white water are shown in **Table I**. Secondary fibers, fillers, and soluble substances are the main components of white water, accounting for 0.498%. The pH of white water was 6.35, which is weakly acidic. The conductivity of white water was as high as 5.37 ms/cm, due to the inorganic salts in the soluble substance. The contents of CODcr and VFA were 8110 mg/L and 61.6 mmol/L, respectively, which are mainly attributed to the organic compounds. The malodorous gases are mainly derived from these organic and inorganic compounds in white water.



3. Effect of hydrogen peroxide (H_2O_2) dosage on the release of total volatile organic compounds (TVOC) from wet-end white water. Experimental conditions: pH = 6.35.



4. Effect of H_2O_2 dosage on the release of hydrogen sulfide (H_2S) from wet-end white water. Experimental conditions: pH = 6.35.



5. Effect of H_2O_2 dosage on the release of ammonia (NH_3) from wet-end white water. Experimental conditions: pH = 6.35.

Effect of H_2O_2 dosage on the release of malodorous gases

The effect of H_2O_2 dosage on the release of malodorous gases from wet-end white water is shown in **Figs. 3, 4, and 5**, which indicate that H_2O_2 had a good effect on malodorous gases control. Figure 3 shows that the amount of released total VOCs (TVOC) decreases with the increase of H_2O_2 dosage, and it finally reaches a stable value. The TVOC concentration reached the minimum value of 22.5 mg/m³ at 100 min when the amount of H_2O_2 increased to 1.5 mmol/L.

The H_2O_2, which degrades some organic compounds in water as a result of its high oxidation potential (OP, 1.78V), is often used to reduce the COD in wastewater [20]. None-

theless, H_2O_2 is ineffective in treating some high-concentration non-biodegradable pollutants, such as high-chlorinated aromatic compounds and inorganic compounds (cyanide, etc.) [21]. Therefore, these pollutants surviving from the H_2O_2 treatment are still released in the form of TVOC gas.

Figures 4 and 5 show the effects of H_2O_2 dosage on the release of H_2S and NH_3 from wet-end white water. It can be seen from the figures that the amounts of H_2S and NH_3 reduce with the increase of the H_2O_2 amount. When the dosage of H_2O_2 was increased from 0 to 1.0 mmol/L, the H_2S concentration significantly decreased from 30.67 to 10.24 mg/m³ at 100 min, and the NH_3 concentration decreased from 8.01 to 2.83 mg/m³. As the dosage of H_2O_2 was further increased to 1.5 mmol/L, the concentrations of H_2S and NH_3 dropped to 0 and 0.64 mg/m³, respectively. Therefore, when H_2O_2 was added at 1.5 mmol/L, TVOC emission reached the minimum, and meanwhile, H_2S and NH_3 were almost completely removed.

According to previous research [22], H_2O_2 can oxidize H_2S to generate sulfur (S^0) through the following reactions (Eq. 1 and Eq. 2). This explains the H_2S removal as the H_2O_2 dosage increases, and finally H_2S is removed:

$$H_2O_2 + H_2S \rightarrow 2H_2O + S^0 \downarrow \quad (1)$$

$$H_2O_2 + HS^- \rightarrow H_2O + S^0 \downarrow + OH^- \quad (2)$$

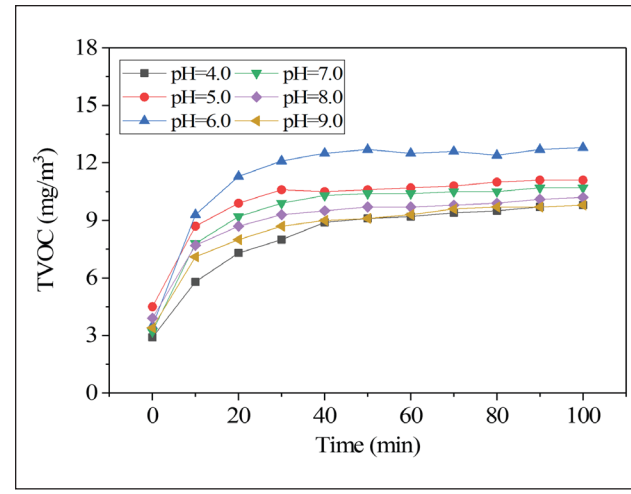
According to the research by Wang et al. [23], NH_3 was more reactive than ammonium (NH_4^+) when reacting with HO•, thus ammonia/ammonium (NH_3/NH_4^+) could be completely converted to nitrogen oxide (NO_x^-) with the addition of H_2O_2 in a stepwise way. This is one of the reasons why NH_3 was removed.

In addition, microorganisms are one of the important factors that generate malodorous gases. The addition of H_2O_2 deactivates microorganisms, decreasing the malodorous gases release from white water [24].

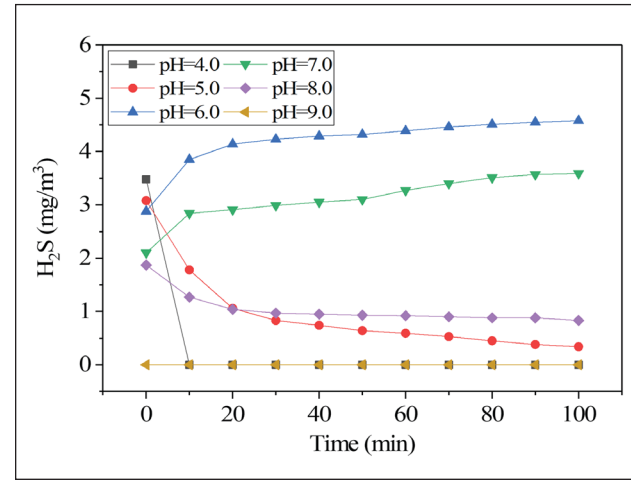
Effect of pH on the release of malodorous gases

Figure 6, 7, and 8 show the effect of pH on the release of malodorous gases from wet-end white water with the addition of H_2O_2. As shown in Fig. 6, the TVOC concentration is the highest (12.8 mg/m³ at 100 min) when pH is 6.0. The TVOC concentration decreases slightly as the pH either increases or decreases. Figures 7 and 8 indicate that the concentrations of H_2S and NH_3 are also the highest (4.58 mg/m³ and 1.49 mg/m³, respectively, at 100 min) at pH 6.0. The H_2S and NH_3 can be completely removed by adjusting the pH to 4.0 or 9.0, respectively.

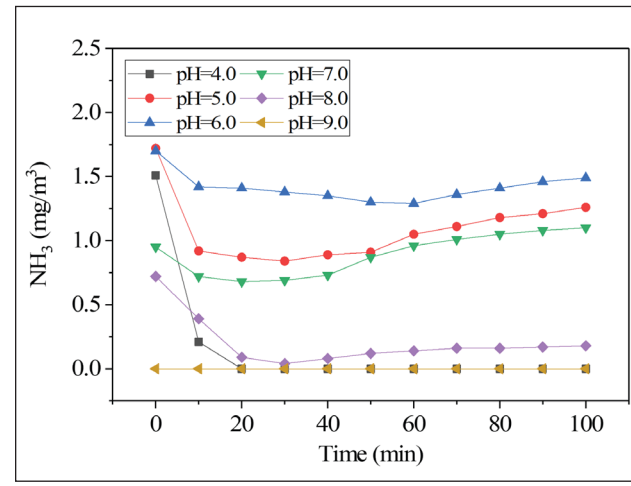
Hydrogen peroxide is a strong oxidant, with its oxidative capacity greatly affected by pH. When the pH drops from 14 to 0, its standard potential increases from 0.87 V to 1.80 V, so H_2O_2 appears to have stronger oxidative ca-



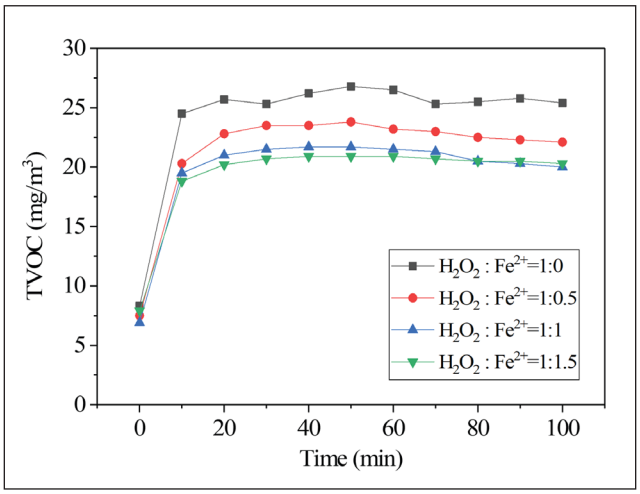
6. Effect of pH on the release of TVOC from wet-end white water. Experimental conditions: H_2O_2 = 1.0 mmol/L.



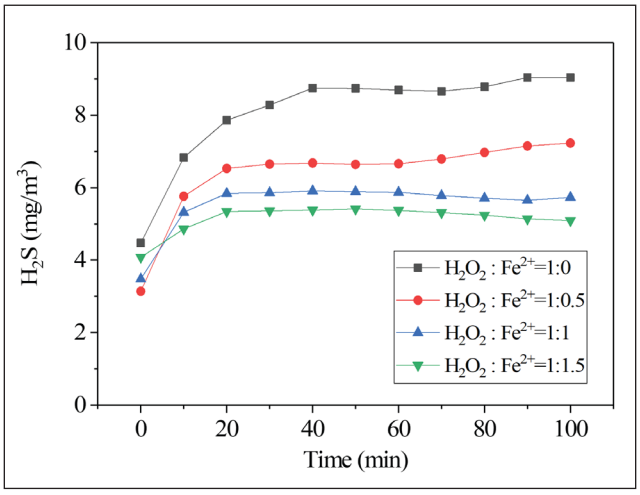
7. Effect of pH on the release of H_2S from wet-end white water. Experimental conditions: H_2O_2 = 1.0 mmol/L.



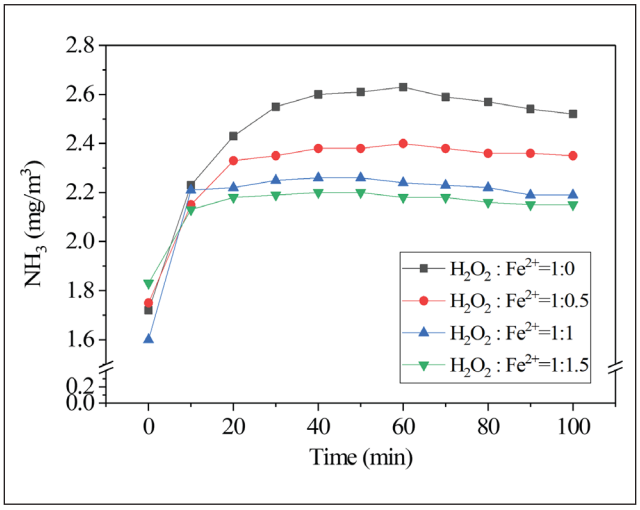
8. Effect of pH on the release of NH_3 from wet-end white water. Experimental conditions: H_2O_2 = 1.0 mmol/L.



9. Effect of Fe^{2+} on the release of TVOC from wet-end white water. Experimental conditions: pH = 6.35, H_2O_2 = 1.0 mmol/L.



10. Effect of Fe^{2+} on the release of H_2S from wet-end white water. Experimental conditions: pH = 6.35, H_2O_2 = 1.0 mmol/L.



11. Effect of Fe^{2+} on the release of NH_3 from wet-end white water. Experimental conditions: pH = 6.35, H_2O_2 = 1.0 mmol/L.

pacity under acidic conditions than it does under alkaline conditions. Accordingly, it has a better degradation effect on malodorous gases under acidic conditions [25]. However, under alkaline conditions, hydroxide (OH^-) can react with acetic acid and propionic acid, etc., in VFA, which limits the emission of the VFA and eventually leads to a slight decrease in TVOC content. According to our previous research, H_2S in white water would be converted to bisulfide (HS^-) while NH_3 converts to nitrate under alkaline conditions, which limited the release of H_2S and NH_3 [19]. Particularly after the pH was higher than 9.0, the concentrations of H_2S and NH_3 dropped to 0 mg/m³. This further proved the potential of using H_2O_2 to control the release of malodorous gases from white water between pH 4.0 and 9.0.

Effect of Fe^{2+} on the release of malodorous gases

Figures 9, 10, and 11 show the effect of Fe^{2+} on the release of malodorous gases from wet-end white water while H_2O_2 is added. The addition of Fe^{2+} promotes the effect of H_2O_2 on the control of malodorous gases releasing from white water. When the concentration of H_2O_2 was 1 mmol/L and the molar ratio of H_2O_2 to Fe^{2+} increased from 1:0 to 1:1.5, the concentrations of TVOC, H_2S, and NH_3 decreased from 25.4, 9.04, and 2.52 mg/m³ to 20.3, 5.09, and 2.15 mg/m³ at 100 min, respectively. Therefore, when the molar ratio of H_2O_2 to Fe^{2+} was 1:1.5, the concentration of malodorous gases reached the minimum.

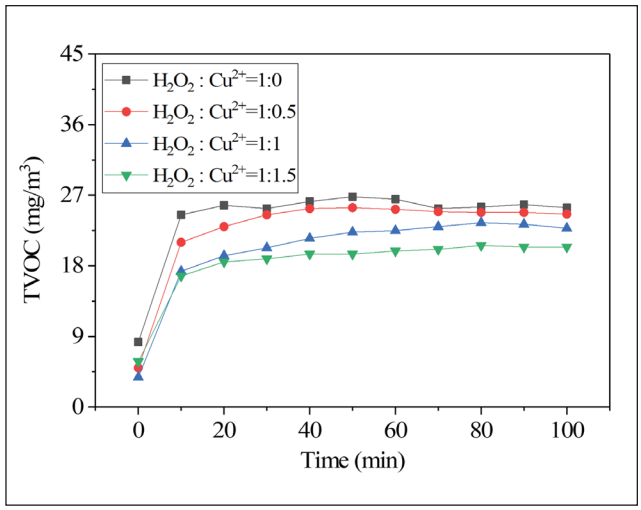
The AOPs degrade bio-refractory pollutants with the generated HO• [26]. Equation 3 shows that H_2O_2 releases HO• under the action of Fe^{2+}, and its oxidation potential is 2.8 V, which is obviously higher than that of H_2O_2 (1.78 V). Therefore, the Fe^{2+}/H_2O_2 system degrades organic compounds more efficiently than H_2O_2 alone, thereby limiting the generation of malodorous gases [27]. The catalytic effect of Fe^{2+} on H_2O_2 can be enhanced by increasing the Fe^{2+} concentration. Therefore, with the increase of Fe^{2+} concentration, the released amount of malodorous gases decreases. However, when the content of Fe^{2+} exceeds the optimal dosage, excessive Fe^{2+} consumes HO• (Eq. 4), thus limiting the H_2O_2 activity [28].

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + HO\cdot + OH^- \quad (3)$$

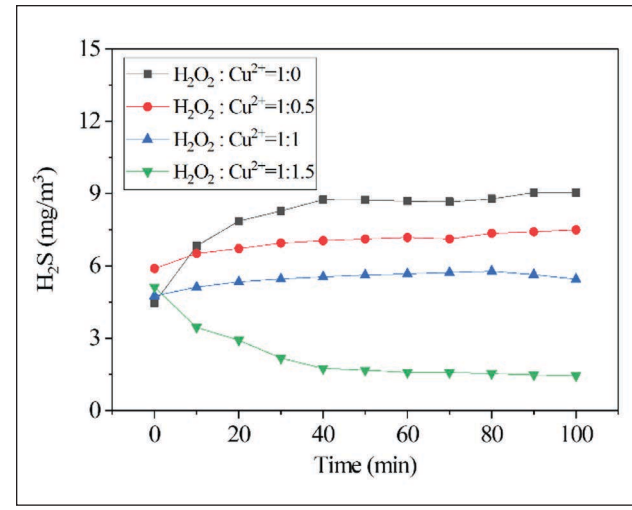
$$Fe^{2+} + HO\cdot \rightarrow Fe^{3+} + OH^- \quad (4)$$

Effect of Cu^{2+} on the release of malodorous gases

Figure 12 shows the effect of Cu^{2+} on the release of malodorous gases while H_2O_2 is added. It can be seen that the addition of Cu^{2+} limits the TVOC generation as H_2O_2 is added. The Cu^{2+} can also activate H_2O_2 and release HO• because it is a transition metal ion (Eqs. 5, 6, and 7), thus inhibiting the release of TVOC [29].



12. Effect of Cu^{2+} on the release of TVOC from wet-end white water. Experimental conditions: pH=6.35, H_2O_2 = 1.0 mmol/L.



13. Effect of Cu^{2+} on the release of H_2S from wet-end white water. Experimental conditions: pH=6.35, H_2O_2 = 1.0 mmol/L.

$$Cu^{2+} + H_2O_2 \rightarrow [Cu(HO_2)]^+ + H^+ \quad (5)$$

$$[Cu(HO_2)]^+ \rightarrow Cu^+ + HO_2 \cdot \quad (6)$$

$$Cu^+ + H_2O_2 \rightarrow Cu^{2+} + HO \cdot + OH^- \quad (7)$$

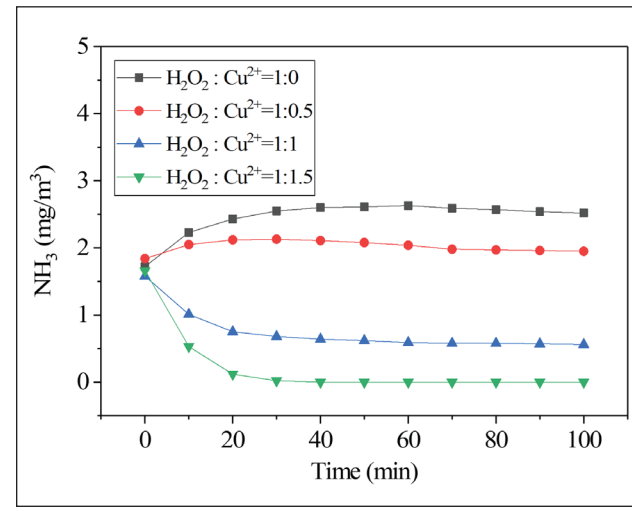
Figures 13 and 14 show the effect of Cu^{2+} on the release of H_2S and NH_3 as H_2O_2 is added. When the molar ratio of H_2O_2 to Cu^{2+} increased from 1:0 to 1:1.5, the content of H_2S and NH_3 gradually decreased. The Cu^{2+} reacts with H_2S (Eq. 8), generating copper(II) sulfide (CuS) precipitate [30]. Also, Cu^{2+} reacts with NH_3 forming $Cu^{2+}(NH_3)n$ (n=1–10) complexes, of which the $Cu^{2+}(NH_3)_4$ ion is the most stable [31,32]. Therefore, with the addition of Cu^{2+}, H_2S and NH_3 were well controlled.

$$H_2S + Cu^{2+} \rightarrow CuS \downarrow + 2H^+ \quad (8)$$

Furthermore, the addition of Cu^{2+} also inactivates the microorganisms in white water, thereby further limiting the generation of malodorous gases [33].

Comparison of Fe^{2+}/H_2O_2 system and Cu^{2+}/H_2O_2 system

Figure 15 shows the concentrations of malodorous gases after the H_2O_2, Fe^{2+}/H_2O_2 systems, and Cu^{2+}/H_2O_2 systems were respectively added to white water for 100 min. The effects of the Fe^{2+}/H_2O_2 system and Cu^{2+}/H_2O_2 system on controlling the malodorous gases were obviously better than those of H_2O_2 alone. Metal ions (Fe^{2+}, Cu^{2+}) catalyze H_2O_2 to release HO• (Eqs. 3–7), thus promoting the degradation of organic matter and removing more malodorous gases. The HO• has been reported to even react with a series of methoxy aromatic compounds [34].



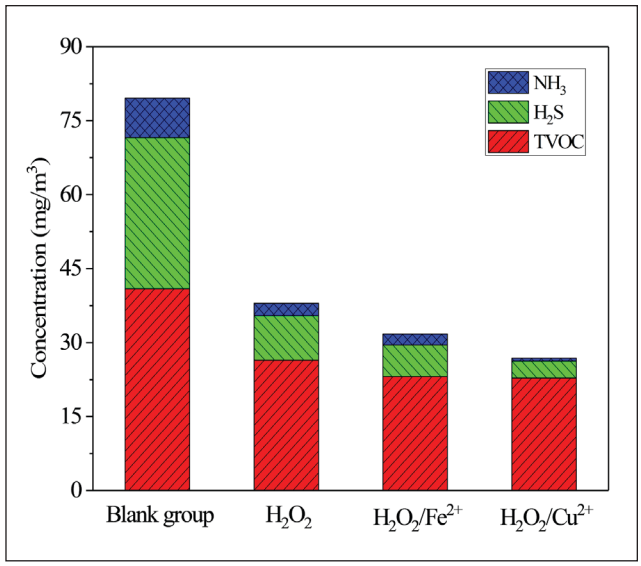
14. Effect of Cu^{2+} on the release of NH_3 from wet-end white water. Experimental conditions: pH=6.35, H_2O_2 = 1.0 mmol/L.

It was found that the Fe^{2+}/H_2O_2 system had nearly the same TVOC degradation efficiency as the Cu^{2+}/H_2O_2 system, whereas the Cu^{2+}/H_2O_2 system had higher removal efficiency for H_2S and NH_3. This was probably due to the similar activation capacity of Fe^{2+} and Cu^{2+} towards H_2O_2 to generate HO•. However, as to the reaction of H_2O_2 with H_2S and NH_3, Cu^{2+} also reacts with them, forming precipitate (CuS) and complexes ($Cu^{2+}(NH_3)n$, n=1–10); therefore, the Cu^{2+}/H_2O_2 system exhibited better control of H2S and NH_3 [30]. In summary, the Cu^{2+}/H_2O_2 system had the best effect on the control of the malodorous gases released from wet-end white water.

CONCLUSIONS

Key findings from this study include:

1. The H_2O_2 limits the release of malodorous gases from white water, and the concentration of malodorous gases



15. Comparison of ferrous ion/hydrogen peroxide (Fe^{2+}/H_2O_2) system and copper ion/hydrogen peroxide (Cu^{2+}/H_2O_2) system. Experimental conditions: pH = 6.35, H_2O_2 = 1.0 mmol/L, Fe^{2+} = 1.0 mmol/L, Cu^{2+} = 1.0 mmol/L.

decreased with an increased amount of H_2O_2. As the amount of H_2O_2 increased from 0 to 1.5 mmol/L, the TVOC concentration decreased from 40.9 to 22.5 mg/m³ at 100 min, which basically reached the maximum removal efficiency. Meanwhile, H_2S and NH_3 were almost completely removed.

2. The pH had little effect on the release of TVOC when H_2O_2 was added, but it evidently affected the release of H_2S and NH_3. When the pH was 6.0, the amount of malodorous gases released was the largest, and the concentrations of TVOC, H_2S, and NH_3 were 12.8, 4.58, and 1.49 mg/m³, respectively, at 100 min. When the pH value of the white water was adjusted to 4.0 or 9.0, the emission of TVOC decreased slightly, while H_2S and NH_3 were completely removed.
3. The Fe^{2+} and Cu^{2+} promote H_2O_2 to produce the hydroxyl radical (HO•), enhancing its inhibition on the release of malodorous gases from white water. The efficiency of the Fe^{2+}/H_2O_2 system and the Cu^{2+}/H_2O_2 system was almost the same in inhibiting TVOC release, but the latter has better performance in removing H_2S and NH_3. **TJ**

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About this journal

ABOUT THE AUTHORS

We chose this topic to research because most paper mills still have problems with release of malodorous gases, which harm the air environment and human health. We studied the relationship between the amount of oxidant added in white water and the concentration of malodorous gases.

Due to the difficulty of sampling and storing white water, we completed the experiment in a relatively short time. We discovered that hydrogen peroxide can be used to limit the release of malodorous gases from white water. An interesting finding was that the Cu^{2+}/H_2O_2 system had the best effect on control of the malodorous gases released from the wet-end white water.

Mills could use this concept to control the release of malodorous gases in white water and promote clean production in the paper industry. Our next step willl be to suppress microorganisms in order to control the release of malodorous gases in white water.

Huang and Liu are graduate students and and Xia is professor at Zhejiang Sci-Tech University in Hangzhou, Zhejiang, China. Dai is associate professor at Shaanxi University of Science and Technology in Xian, Shaanxi, China. Dou is general manager at Shanghai Jinhua Industrial Co. Ltd. in Shanghai, China. Ding is general manager at Zhejiang Jingxing Paper Joint Stock Co. Ltd. in Jiaxing, China. Email Xia at xinxingxia2000@hotmail.com.

Shan-Cong

Chang

Lei

Sheng

Ming-Qi

Xin-Xing