Investigation of the influencing factors in odor emission from wet-end white water

SHANCONG HUANG, SHENG DOU, MINGQI DING, AND XINXING XIA

ABSTRACT: Emission of malodorous gases, such as volatile organic compounds (VOCs), hydrogen sulfide (H_2S), and ammonia (NH_3) during pulping and papermaking has caused certain harm to the air environment and human health. This paper investigated the influencing factors of odor emission from wet-end white water during the production of bobbin paper in a papermaking mill using old corrugated containers (OCC) as raw material.

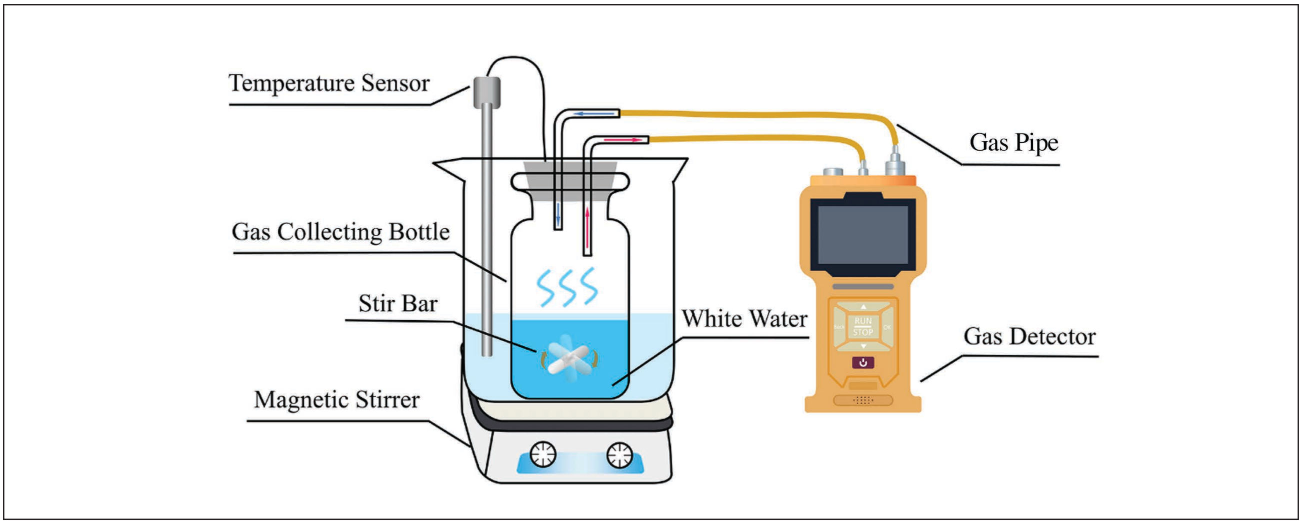
The concentration of malodorous gases emitted from wet-end white water was determined with pump-suction gas detectors. The results indicated that low temperature could limit the release of malodorous gases from white water. Specifically, no total volatile organic compounds (TVOC), H_2S, and NH_3 was detected at a temperature of 15°C. The concentrations of malodorous gases were slightly increased when temperature increased to 25°C. When temperature was 55°C, the released concentrations of TVOC, H_2S, and NH_3 were 22.3 mg/m^3, 5.91 mg/m^3, and 2.78 mg/m^3, respectively. Therefore, the content of malodorous gases significantly increased with the temperature increase. The stirring of white water accelerated the release of malodorous gases, and the release rate sped up as the stirring speed increased. However, the total amount of malodorous gases released were basically the same as the static state. Furthermore, the higher the concentration of white water, the greater the amount of malodorous gases released. The pH had little influence on the TVOC release, whereas it significantly affected the release of H_2S and NH_3. With the increase of pH value, the released amount of H_2S and NH_3 gradually decreased. When pH reached 9.0, the release amount of H_2S and NH_3 was almost zero, proving that an alkaline condition inhibits the release of H_2S and NH_3.

Application: The relevant information in this article can be used to control the release of malodorous gases from paper mills.

Malodorous gas is a type of gas that irritates the human olfactory organs. Total volatile organic compounds (TVOC) represent most of the malodorous gases. In addition, malodorous gas includes inorganic gases such as hydrogen sulfide (H_2S) and ammonia (NH_3). Their emission causes serious environmental issues, such as smog, greenhouse effect, acid rain, and ozone depletion [1]. Long-term human exposure to these gases can damage nerves and cause headaches, nausea, vomiting, etc. [2]. In severe cases, it may lead to memory loss and even cancers [3].

The water consumption and gas emission in the pulp and papermaking industry are huge. The fresh water consumption accounts for about 8.7% of total industry, and the direct carbon dioxide (CO_2) emission accounts for about 2% of total industry [4,5]. In addition to the wastewater and sludge treatment process, the papermaking production process also accounts for generation of malodorous gases. Specifically, due to the close circulation of white water, organic matter, chemicals, and microorganisms accumulate continuously in white water, resulting in the formation of a large amount of malodorous gases in white water. The water consumption in the papermaking section accounts for about 70%–80% of the total pulp and papermaking water consumption. The violent turbulence in the papermaking process accelerates the odor emission. Tong et al. [6] found emissions of malodorous gases from the wet end where TVOC possessed higher concentration than other gases (i.e., formaldehyde [HCHO], H_2S, hydrocarbon compounds [C_xH_y], etc.). Addition of chemicals, poor ventilation, high temperature, and high humidity are the main reasons for the high concentration of TVOC.

White water system closure provides favorable conditions for the growth and reproduction of microorganisms [7]. The source of malodorous gases was mainly anaerobic bacteria in microorganisms. These anaerobic bacteria liberate energy from molecules using anaerobic respiration. The end products of anaerobic respiration are usually nitrogen, oxides of nitrogen, H_2S, or methane [8]. The NH_3 is regarded as a typical malodorous gas that is usually derived from the ammonification of microorganisms. Microorganisms in white water transform organic nitrogen into NH_4^+ or NH_3 to promote the NH_3 emission [9]. The H_2S is a product of sulfate-reducing bacteria decomposing sulfur-containing compounds under anaerobic conditions. High temperature during the first stage of anaerobic biological treatment generally increases H_2S emission [10]. A small amount of sludge in white water releases some volatile organic gases (VOCs). Terpenes with predominantly α-pinene were the main components of VOCs, accounting for between 70% and 80% [11]. Organic matter, such as secondary fiber and starch in white water, provides sufficient nutrition for the first stage



1. Schematic of the experimental setup.

of anaerobic digestion. At this stage, microorganisms secrete extracellular enzymes, breaking them down into simple soluble monomers. During the second stage (the acid-generating stage) of anaerobic digestion, the formation of volatile fatty acids (VFAs) is mainly caused by acidogenic bacteria [12].

The chemicals added in the papermaking process are also one of the reasons for emission of the malodorous gases from white water. Most of the papermaking chemicals, such as sizing agent, wet strength agent, dry strength agent, defoaming agent, and felt conditioning agent, usually have a special smell. In addition, a variety of impurities are brought into the system by the waste paper pulping process, such as hot melts, plastics, metals, stickies, paints, etc., and soluble substances may enter the white water circulation in the paper machine. Stickies usually contain styrene-butadiene rubber (SBR), which is thermally decomposed into styrene and butadiene. They can be easily detected by humans due to their strong volatility [13]. It is worth noting that the coatings used in the papermaking process mainly consist of fossil-based chemicals containing lots of hydrocarbon substances (such as thiols, thioethers, and carboxylic acids) that have a strong pungent odor [14].

Davis and Smith [15] found that the odor in recycled paper mills mainly came from VFAs, which were produced by anaerobic bacteria metabolism. In order to effectively inhibit the generation of VFAs, comprehensive methods were used, such as monitoring technology, new bacterial culture methods, improved operating procedures, germicidal agent, and water-based oxidation treatment. Gudlauski [7] believed that the production of flammable gases could be controlled by inhibiting the growth of anaerobic bacteria. Control methods, such as mechanical aeration of the water, ventilation, and biocide treatment, will reduce populations of anaerobic bacteria to manageable levels. Wiik and Helle [16] found that wood extractives contained unsaturated fatty acids that were oxidized to odorous aldehydes. In food packaging, these aldehydes may cause tainting. Their study indicated that aldehydes were formed during the analytical procedure and that paper appeared virtually unable to store volatile aldehydes, either adsorbed, absorbed, or trapped in voids. Also, the release of VFAs from the papers has a certain relationship with the concentration of VFAs in white water [17]. This paper investigated the influencing factors of odor emission from wet-end white water to guide the control of the malodorous gases from wet-end white water.

MATERIALS AND METHODS

Materials

Wet-end white water was kindly provided by Zhejiang Jingxing Paper Joint Stock Co. Ltd. (Zhejiang, China), which uses OCC as raw material to produce bobbin paper. Sodium hydroxide (NaOH, AR grade), hydrochloric acid (HCl, AR grade), and other chemical reagents used in the experiment were purchased from Damao Chemical Reagent Factory (Tianjin, China).

Procedure and apparatus

The collected wet-end white water was stored at 5°C. For each analysis, 100 mL of wet-end white water was placed in a 250 mL gas collecting bottle. The influence of temperature, time, stirring speed, white water concentration, and pH on the concentration of malodorous gases released from white water was investigated. The setup is shown in **Fig. 1**. A magnetic stirrer was used to control the temperature of white water and the speed of the stir bar. The original wet-end white water was diluted with fresh water to adjust the white water concentration. The pH value was adjusted with sodium hydroxide and hydrochloric acid.

Analytical method

The gas detectors (JA908-VOC-PID, JA908-H2S, JA908-NH3) from Shenzhen Ziyuan Environmental Protection Technology Co. Ltd. (Guangdong, China) were used to de-

Temperature, °C	Concentration, %	pH	Conductivity, mS/cm	CODcr, mg/L	VFAs, mmol/L
42	0.574	6.5	4.79	8080	34.6
CODcr = chemical oxygen demand; VFAs = volatile fatty acids.					

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

termine the gas concentrations. The pH meter (PHS-25, Shanghai Yidian Scientific Instrument Co. Ltd.; Tianjin, China) and conductivity meter (DDS-307, Shanghai Yidian Scientific Instrument Co. Ltd.) were used to measure the pH and conductivity of white water. The white water concentration was measured using a 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 obtained quality was the white water concentration. 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 VFAs were analyzed as follows: a certain amount (V, mL) of supernatant resulting from the centrifugation of white water was taken and diluted to 100 mL with deionized water. The solution was titrated with 0.100 mol/L HCl to pH 3.0. The solution was then moved to an Erlenmeyer flask with condensing reflux and heated to boil for 3 min to remove CO_2. Reflux was then continued for 2 min without heating, and the solution was quickly cooled to room temperature. After cooling, the solution was titrated with 0.100 mol/L NaOH to a pH of 6.5, and the amount of consumed NaOH was recorded (A, mL). The VFAs calculation formula is shown in Eq. 1:

$$\text{VFAs}\left(\frac{mmol}{L}\right) = \frac{A \times c_a}{V} \times 1000 \quad (1)$$

where:
A = amount of NaOH standard solution, mL
c_a = concentration of standard NaOH solution, mol/L
V = white water sample volume, mL

RESULTS AND DISCUSSION

Analysis of the wet-end white water

The basic properties of wet-end white water are shown in **Table I**. The white water was weakly acidic, since it contained 34.6 mmol/L VFAs generating hydrogen ions. Various chemicals added in the papermaking process and the highly closed circulation of white water caused the electrolyte accumulation. Therefore, the conductivity of white water reached 4.79 mS/cm. In addition, white water exhibited a high concentration, because it contained various substances, such as fine fibers, starch, latex, and inorganic minerals. The CODcr of the white water was determined to be 8080 mg/L.

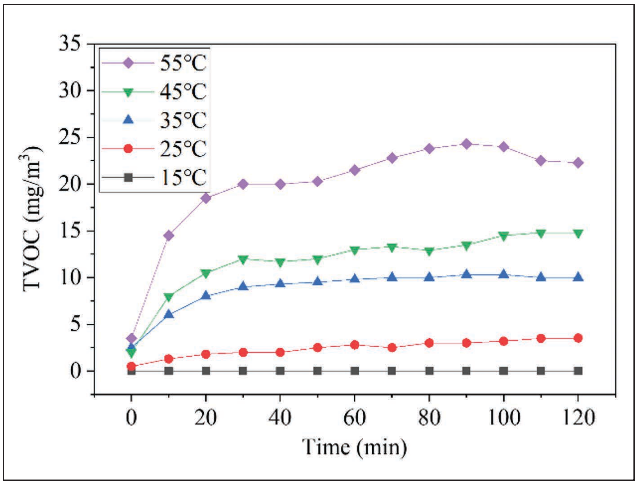
Effect of temperature on the release of malodorous gases

The effect of temperature on the release of malodorous gases from wet-end white water is shown in **Figs. 2**, **3**, and **4**. When the temperature was 15°C, the concentration of TVOC, H_2S, and NH_3 was not detected. At 25°C, the concentration of malodorous gases was slightly increased, proving that low temperature can limit the release of malodorous gases from white water. When the temperature was further increased to higher than 35°C, the release amount of malodorous gases greatly increased. At 120 min, the concentrations of TVOC H_2S and NH_3 increased from 10 mg/m³, 1.18 mg/m³, and 1.02 mg/m³ at 35°C to 22.3 mg/m³, 5.91 mg/m³, and 2.78 mg/m³ at 55°C, respectively, which showed that high temperature promotes the release of malodorous gases. This was similar to the conclusion of Thurnau and Clark's [18] study, which was consistent with Henry's law. The saturated vapor pressure of the gas increases as the temperature increases, leading to the decrease of gas solubility in water. Therefore, the volatility increases and the gas concentration increases as well. These results proved that the temperature was an important factor that affects the release of malodorous gases [19,20].

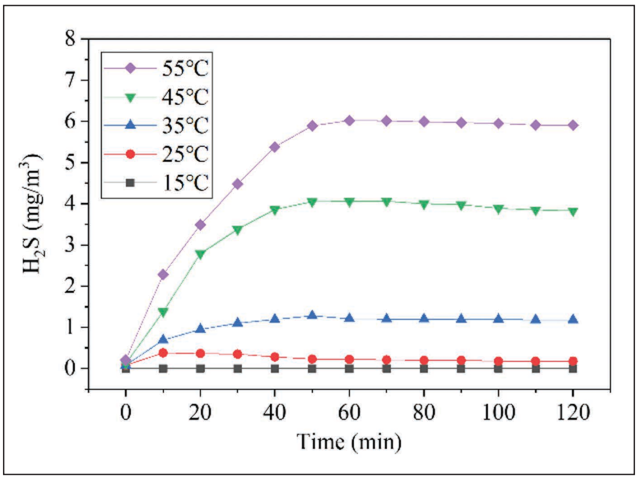
The malodorous gases emission from white water could be divided into two stages: the rapid rise stage and the stable stage. Figure 2 shows that the concentration of TVOC almost reaches the maximum value at 30 min, while Fig. 3 and Fig. 4 show that the concentrations of H_2S and NH_3 reached the maximum at 60 min. The gas amount of TVOC was larger than that of H_2S and NH_3. This was probably because in the papermaking process, the contact of white water with air increased the dissolved oxygen in water and the formation of VFAs in white water was not affected by the oxygen content [21], but the generation of H_2S and NH_3 required bacteria working under anaerobic conditions [10].

Effect of stirring on the release of malodorous gases

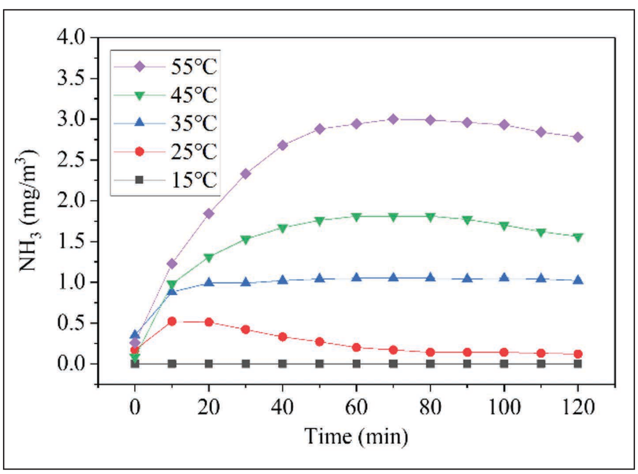
As shown in **Figs. 5**, **6**, and **7**, the release amount of malodorous gases increased quickly in the initial stirring stage, and the gas release rates also increased as the stirring speed increased. At 20 min, the concentrations of TVOC, H_2S, and NH_3 increased from 8 mg/m³, 0.95 mg/m³, and 0.99 mg/m³ at 0 rpm stirring speed to 21.3 mg/m³, 3.78 mg/m³, and 2.47 mg/m³ at 1000 rpm stirring speed, respectively. This can be attributed to the increased contact of the two phas-



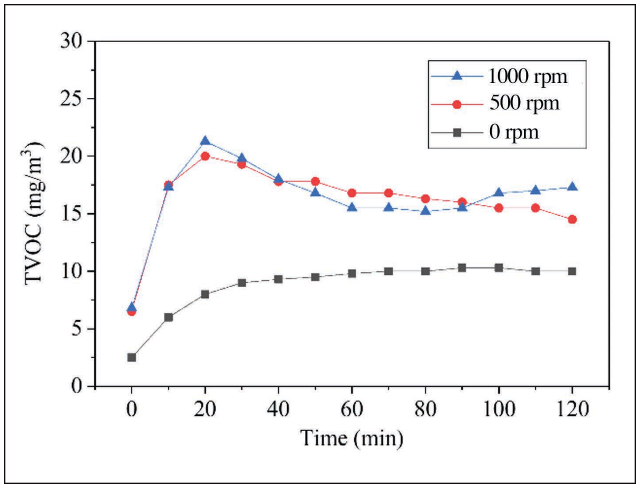
2. Effect of temperature on the release of total volatile organic compounds (TVOC) from wet-end white water. Experimental conditions: concentration = 0.574%, pH = 6.5, no stirring.



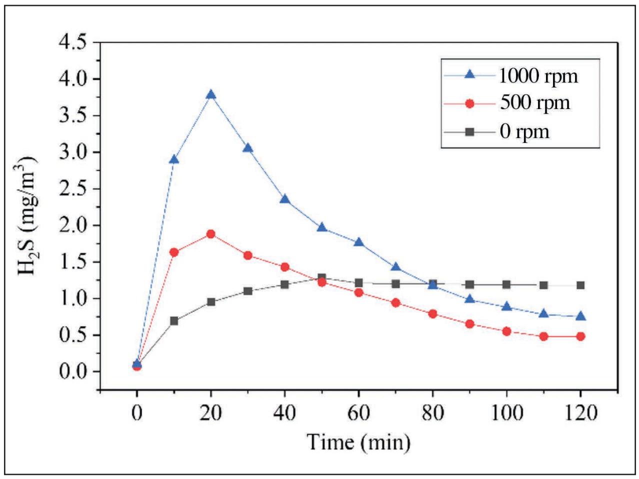
3. Effect of temperature on the release of hydrogen sulfide (H_2S) from wet-end white water. Experimental conditions: concentration = 0.574%, pH = 6.5, no stirring.



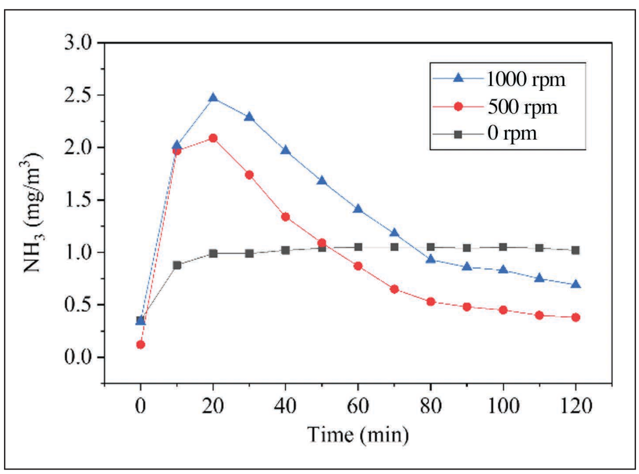
4. Effect of temperature on the release of ammonia (NH_3) from wet-end white water. Experimental conditions: concentration = 0.574%, pH = 6.5, no stirring.



5. Effect of stirring speed on the release of TVOC from wet-end white water. Experimental conditions: temperature = 35°C, concentration = 0.574%, pH = 6.5.



6. Effect of stirring speed on the release of H_2S from wet-end white water. Experimental conditions: temperature = 35°C, concentration = 0.574%, pH = 6.5.



7. Effect of stirring speed on the release of NH_3 from wet-end white water. Experimental conditions: temperature = 35°C, concentration = 0.574%, pH = 6.5.

es of gas-liquid upon stirring, which sped up the mass transfer efficiency and eventually led to the rapid release of malodorous gases [22].

The concentrations of malodorous gases decreased and gradually reached a stable value after they reached a highest value. At this time, the stirring speed had little effect on the concentration of the gases. This phenomenon may be due to the rapid release of malodorous gases from the water in a short time, causing the unbalance of gas-liquid phase in the container. The mass transferred from the high-concentration gas phase to the low-concentration liquid phase occurred; therefore, the gas concentration slowly decreased. Compared with static state, the concentration of TVOC with stirring was always higher, while the concentrations of H_2S and NH_3 with stirring were slightly lower after 60 min. According to the principle of similar miscibility, the released H_2S and NH_3 redissolved in white water because they had higher solubility in water than TVOC and were polar molecules [23].

Therefore, we can deduce that in real paper production, due to the accumulation of rotten pulp and bacterial reproduction in the system and violent turbulence in the wet end, most malodorous gases will be quickly released in the initial stage. However, after a certain period of production, the released amount of malodorous gases significantly decreases because most malodorous gases have been released. Also, turbulence size has no obvious influence on the released amount of malodorous gases.

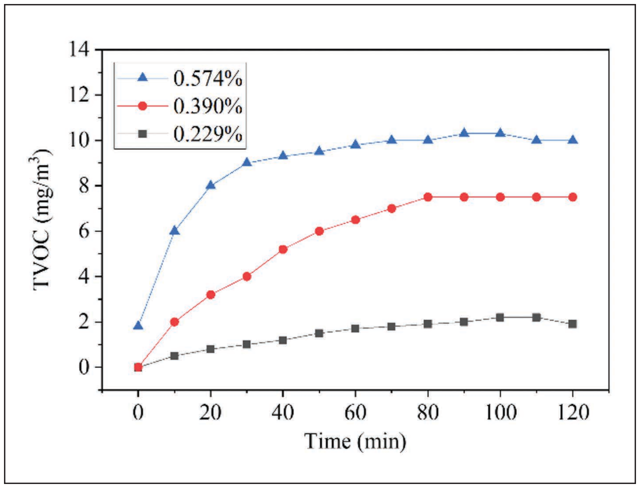
Effect of white water concentration on the release of malodorous gases

The effect of wet-end white water concentration on the release of malodorous gases is shown in **Figs. 8**, **9**, and **10**. As the concentration of white water increased, the concentrations of TVOC, H_2S, and NH_3 increased. The concentrations of TVOC, H_2S, and NH_3 reached 10 mg/m^3, 1.18 mg/m^3, and 1.02 mg/m^3, respectively, at 120 min, while the concentration of white water was 0.574%. The higher the concentration of white water, the more fibers, fillers, and impurities it contained. Therefore, there were more nutrients in white water that resulted in a significant increase in malodorous gases [24].

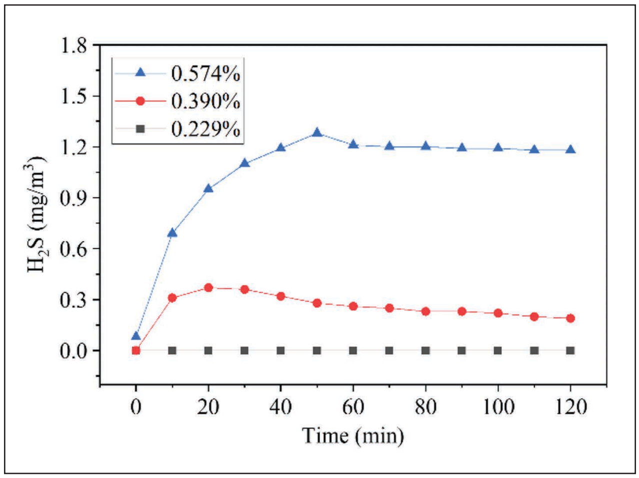
Effect of pH on the release of malodorous gases

Figures 11, **12**, and **13** illustrate the minor effect pH had on TVOC release and the significant effect it had on H_2S and NH_3 release. The release of H_2S and NH_3 gradually decreased with the increase of pH. When the pH value was 9.0, the release of H_2S and NH_3 was almost zero; therefore, alkaline conditions could inhibit the release of H_2S and NH_3.

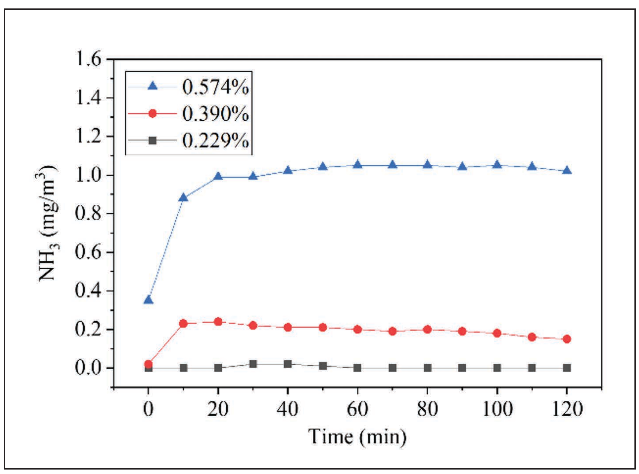
The TVOC concentration exhibited no significant change as the pH of white water was between 5.0 and 9.0 (Fig. 11), because most of the VOCs had stable chemical properties and hydrogen ion (H^+) and hydroxide (OH^-) in



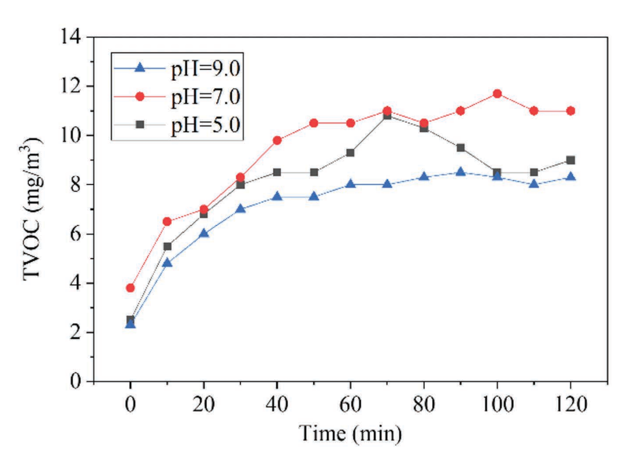
8. Effect of concentration on the release of TVOC from wet-end white water. Experimental conditions: temperature = 35°C, pH = 6.5, no stirring.



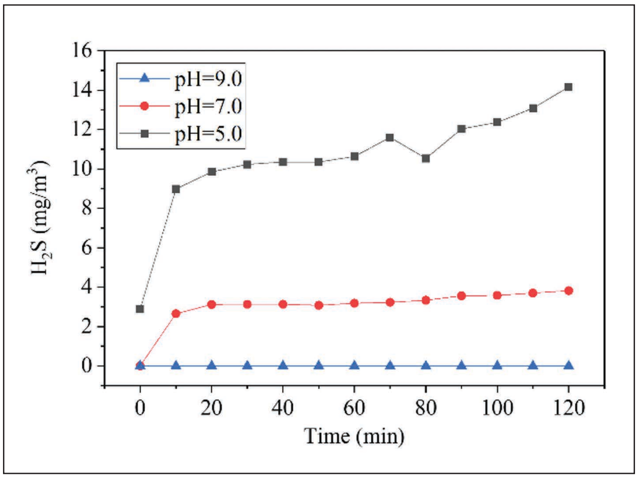
9. Effect of concentration on the release of H_2S from wet-end white water. Experimental conditions: temperature = 35°C, pH = 6.5, no stirring.



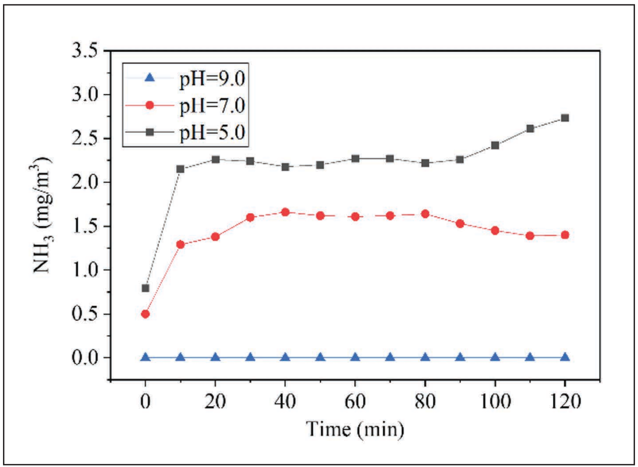
10. Effect of concentration on the release of NH_3 from wet-end white water. Experimental conditions: temperature = 35°C, pH = 6.5, no stirring.



11. Effect of pH on the release of TVOC from wet-end white water. Experimental conditions: temperature = 35°C, concentration = 0.574%, no stirring.



12. Effect of pH on the release of H_2S from wet-end white water. Experimental conditions: temperature = 35°C, concentration = 0.574%, no stirring.



13. Effect of pH on the release of NH_3 from wet-end white water. Experimental conditions: temperature = 35°C, concentration = 0.574%, no stirring.

water had little influence on their emission [25]. However, the TVOC concentration had a slight decrease at pH of 9.0 that was due to the reaction between OH^- and acetic acid, propionic acid, etc., in some VFAs, limiting emission of the VFAs [21].

As shown in Fig. 12 and Fig. 13, the released amount of H_2S and NH_3 was the highest at pH of 5.0, followed by the data obtained at pH of 7.0, and no H_2S and NH_3 were detected at pH of 9.0. Therefore, the weak acidic condition of white water promotes the release of H_2S and NH_3, whereas a weak alkaline condition inhibits their release.

According to the dissociation equation of H_2S:

$$H_2S \leftrightarrow H^+ + HS^- \tag{2}$$

At the pH of 5.0, the dissociation reaction goes towards the left and generates H_2S. When pH was 9.0, the dissociation reaction goes towards the right; thereby, the concentration of HS^- ion in water increased and the amount of H_2S decreased [26].

Nitrifying bacteria in white water can convert ammonia and ammonium salts to nitrate under aerobic conditions. The pH had a great influence on the nitrification process, which not only affects the growth rate of nitrobacteria, but also affects the acid-base balance of nitrogen dioxide/nitrous acid (NO_2/HNO_2) and ammonium/ammonia (NH_4^+/NH_3) [27]. It was found that weakly alkaline conditions and temperature between 30°C and 40°C were the most suitable for the nitrifying bacteria growth [28,29]. At the pH of 5.0, ammonia nitrogen in white water mainly existed in the form of NH_4^+/NH_3, resulting in the release of NH_3. However, NH_3 was not detected while the pH of the white water was 9.0. The main reason may be that the weak alkaline condition was suitable for survival of nitrifying bacteria, which is beneficial to the conversion of ammonia to nitrate.

CONCLUSIONS

Conclusions from this study include the following:

1. Low temperature limited the release of malodorous gases from white water. When the temperature was 15°C, the concentrations of TVOC, H_2S, and NH_3 were not detected. At 25°C, the concentrations of malodorous gases were slightly increased. With further temperature increase, the released content of malodorous gases from wet-end white water increased significantly. When the temperature was 55°C, the released amounts of TVOC, H_2S, and NH_3 reached 22.3 mg/m^3, 5.91 mg/m^3, and 2.78 mg/m^3, respectively.
2. The stirring of white water accelerated the release of malodorous gases in a short time. With the increase in stirring speed, the release rate of malodorous gases increased, but the total released amounts of malodorous gases were basically the same as the static state of white water.

3. The higher the concentration of white water, the greater the amount of malodorous gases released.
4. The pH had little influence on the release of TVOC, but had an obvious influence on the H_2S and NH_3 release. With the increase in pH value, the released amounts of H_2S and NH_3 gradually decreased. However, the released amounts of H_2S and NH_3 were almost zero at the pH of 9.0, proving that alkaline conditions inhibit the release of H_2S and NH_3.

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We chose this topic to research because most paper mills still have problems with release of malodorous gases, which brings certain harm to the air environment and human health. Previous studies have found that the odor of paper mills mainly comes from volatile fatty acids in white water. This research differed from previous work in that we have established a relationship between white water and malodorous gases.

The most difficult part of this research was the sampling and storage of white water. Fortunately, our experimental point was not far from the sampling point. After sampling, we stored it at 5°C and completed the experiment in a short time.

We discovered that low temperature can inhibit the release of malodorous gases, and the intense turbulence of white water will aggravate the release of malodorous gases. It was most interesting to find that no total volatile organic compounds (TVOC), hydrogen sulfide (H_2S), or ammonia (NH_3) were detected at temperature of 15°C. The concentrations of malodorous gases were slightly increased when temperature increased to 25°C. Also, the pH had little influence on the release of TVOC, but it had an obvious influence on release of H_2S and NH_3.

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 is to use chemical methods to control release of malodorous gases in white water.

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