Investigation of the factors contributing to malodorous gases emission during secondary fiber reuse

JINGJING CHEN, CHANG LIU, SHENG DOU, MINGQI DING, AND XINXING XIA

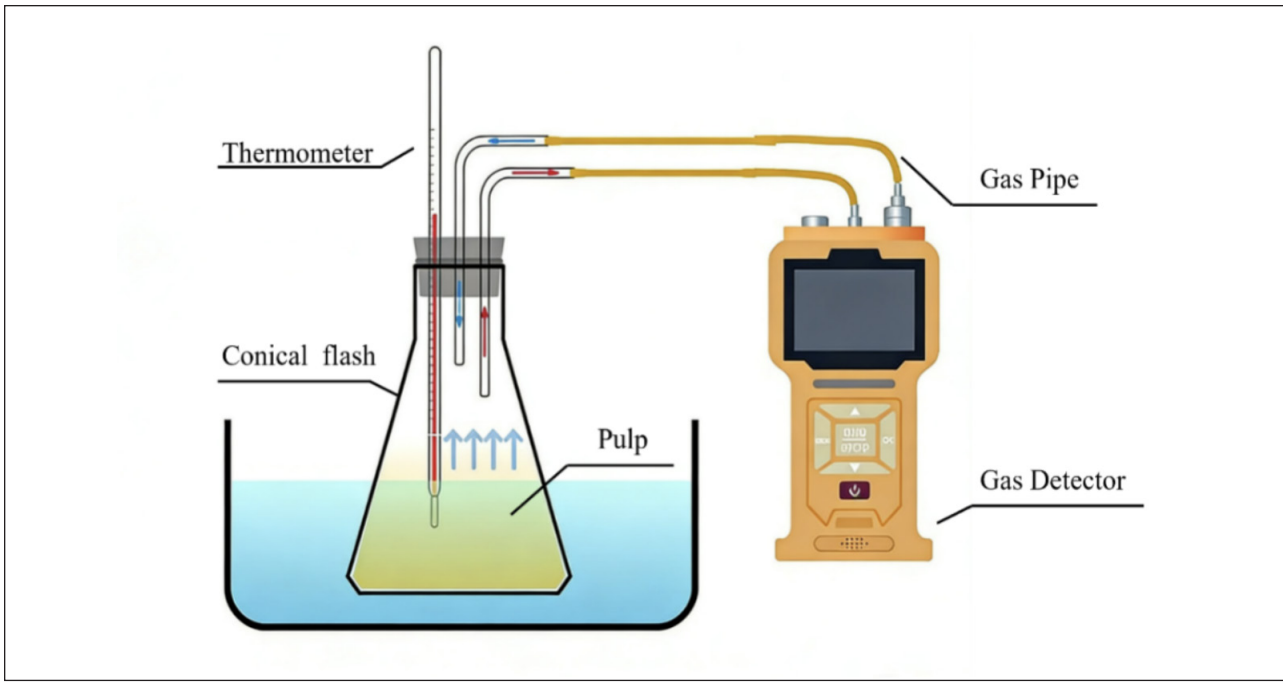
ABSTRACT: Malodorous gases are commonly produced during secondary fiber reuse, which is harmful to human health and causes environmental pollution. This paper investigated the influence of fiber type and concentration, temperature, and whitewater concentration on the malodorous gases. The results indicated that, in pulp prepared with fresh water, bleached hardwood kraft pulp (LBKP) did not produce malodorous gases after standing for five days. In contrast, the secondary fiber began to release substantial amounts of total volatile organic compounds (TVOC) on the third day and hydrogen sulfide (H_2S) and ammonia (NH_3) on the sixth day, and black substances began to appear in the pulp, which proved that the microorganisms began to proliferate. With the increase of the secondary fiber concentration, the release amounts of TVOC, H_2S, and NH_3 gradually rose, along with the black substances in the pulp. With increased temperature, the release of TVOC increased steadily, while the release of H_2S and NH_3 reached the maximum at about 45°C, and then began to decline. The decrease of the pulp freeness accelerated the generation of the malodorous gases, but the total release amounts of TVOC, H_2S, and NH_3 were basically the same. With the increase of white water concentration, the release of TVOC, H_2S, and NH_3 increased rapidly. When the white water/fresh water was 20 mL/80 mL, the slurry changed from pale yellow to aterrimus on the sixth day. Therefore, microorganisms in the secondary fiber caused pulp deterioration, while white water was the main reason for generating a large amount of malodorous gases.

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

Waste paper, known as secondary fiber, that is reused for papermaking significantly curtails the demand for virgin woods and conserves forest resources [1]. However, the recycling of secondary fiber brings the problem of environmental pollution, especially the emission of malodorous gases [2]. These malodorous gases, mainly including total volatile organic compounds (TVOC), hydrogen sulfide (H_2S), and ammonia (NH_3), have low olfactory threshold, direct stimulation, and harm to the human respiratory system, circulatory system, and nervous system [3]. They also diffuse into the atmosphere to aggravate air pollution, which is one of the precursors to form secondary particulate matter and ozone [4,5].

The source of waste paper is complex, and it is easily contaminated with impurities and microorganisms during collection, transportation, and storage. In addition, in the process of secondary fiber recycling, the closed white water circulation loop leads to accumulation of dissolved organic matters, fillers, and various chemical additives [6]. These are the causes of malodorous gases. In the early 21st century, Fourest et al. [7] investigated the source of odorous gases during secondary fiber recycling and showed that the production of hydrogen sulfide and volatile fatty acids was closely associated with the proliferation and metabolism of anaerobic microorganisms.

Researchers have proposed multiple technologies for handling industrial odorous gases in recent years. The addition of expansion agents and additives (such as biochar and zeolite) in manufacture could reduce the emission of malodorous gases [8]. Biochar, which possesses specific adsorption properties and various functional groups, could adsorb 60% to 70% of the odorous gases released during composting. Moreover, biofiltration emerged as a robust and cost-effective technique, which reduced the emission of odorous gases by as much as 90% at the end of pipeline [9]. The research by Huang et al. [10] demonstrated that low temperature inhibited the release of the malodorous gases from white water, while the emission significantly increased at elevated temperature. As the stirring speed increased, the rate of the malodorous gases emission from whitewater increased correspondingly. However, the total amount of the malodorous gases released were basically the same as the static state [10]. Huang et al. [11] also found that the released amount of the malodorous gases decreased gradually with the increase of H_2O_2 dosage. Ferrous ion (Fe^{2+}) and cupric (Cu^{2+}) promoted generation of hydroxyl radicals (HO•) of H_2O_2, enhancing its inhibition on the release of malodorous gases from white water. Liu et al. [12] found that the release of H_2S and NH_3 increased



1. Schematic of the experimental setup.

with the increase of starch concentration in pulp, whereas TVOC was basically the same as that without starch. Also, the release of malodorous gases of TVOC, H_2S, and NH_3 increased significantly with the increase of sodium sulfate (Na_2SO_4) concentration in the pulp.

In this paper, we studied of the influence of fiber types and concentration, pulp freeness and temperature, and white water concentration on the generation of malodorous gases to provide a theoretical basis for the malodorous gases emission control in the process of secondary fiber reuse.

MATERIALS AND METHODS

Materials

Secondary fiber (waste corrugated cardboard boxes, with a freeness of 250 mL), was gathered from the production line of corrugated base paper at Zhejiang Jingxing Paper Joint Stock Co. Ltd. (Zhejiang, China). Bleached hardwood kraft pulp (LBKP, with a freeness of 540 mL) and white water were all provided by Zhejiang Jingxing Paper Joint Stock Co. Ltd.

Procedure and apparatus

The setup is shown in **Fig. 1**. It comprised two main parts: (1) the left was the apparatus of malodorous gases generation, which contained a 250 mL sealed conical flask loaded with pulp, and (2) the right was a pumping gas detector (JA908-VOC-PID, JA908-H2S, JA908-NH_3, Shenzhen Ziyuan Environmental Protection Technology Co. Ltd.; Guangdong, China), detecting the concentrations of TVOC, H_2S, and NH_3, respectively.

RESULTS AND DISCUSSION

Effect of fiber on odorous gases generation

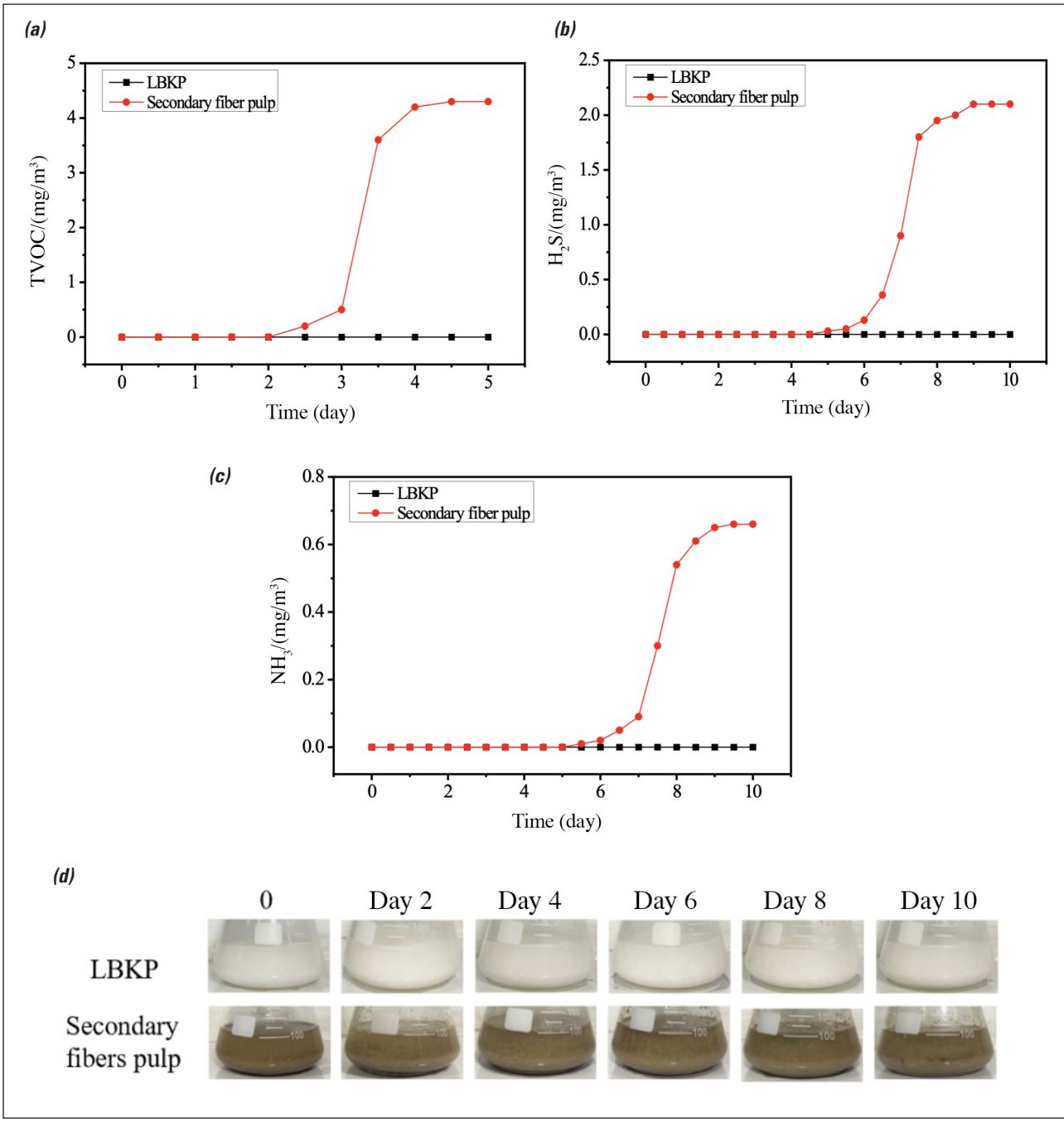
The effects of fiber types on the release of malodorous gases are shown in **Figures. 2a–2c**. Standing for five days in fresh water of the LBKP, the release amounts of TVOC, H_2S, and NH_3 from the slurry were almost zero, which proved that LBKP did not generate malodorous gases in fresh water. The TVOC began to release on day 2 from the secondary fiber, and rose to 4.2 mg/m³ on day 4, and then plateaued. The H_2S and NH_3 started to release on day 4.5, and reached the maximum values of 2.1 mg/m³ and 0.7 mg/m³ on day 9, respectively. At the same time, as shown in Fig. 2d, some black substances appeared in the slurry, which was attributed to the combination of metal ions with the sulfide ion (S^{2-}) in water to form black sulfides, and the metal ion was typically Fe^{2+} [13,14], as shown in Eq. 1:

$$X^{2+} + S^{2-} \rightarrow XS\downarrow \quad (1)$$

Attributed to impurities and microorganisms, secondary fibers are easy to generate malodorous gases. Shi et al. [15] found that secondary fiber contained other substances besides fiber, such as starch, plastics, metals, rubber, organic matter, dust, and bacteria. The complexity of secondary fiber composition created favorable conditions for microbial proliferation, leading to pulp decay and deterioration to generate malodorous gases and black substances [16].

Effect of secondary fiber concentrations on odorous gases generation

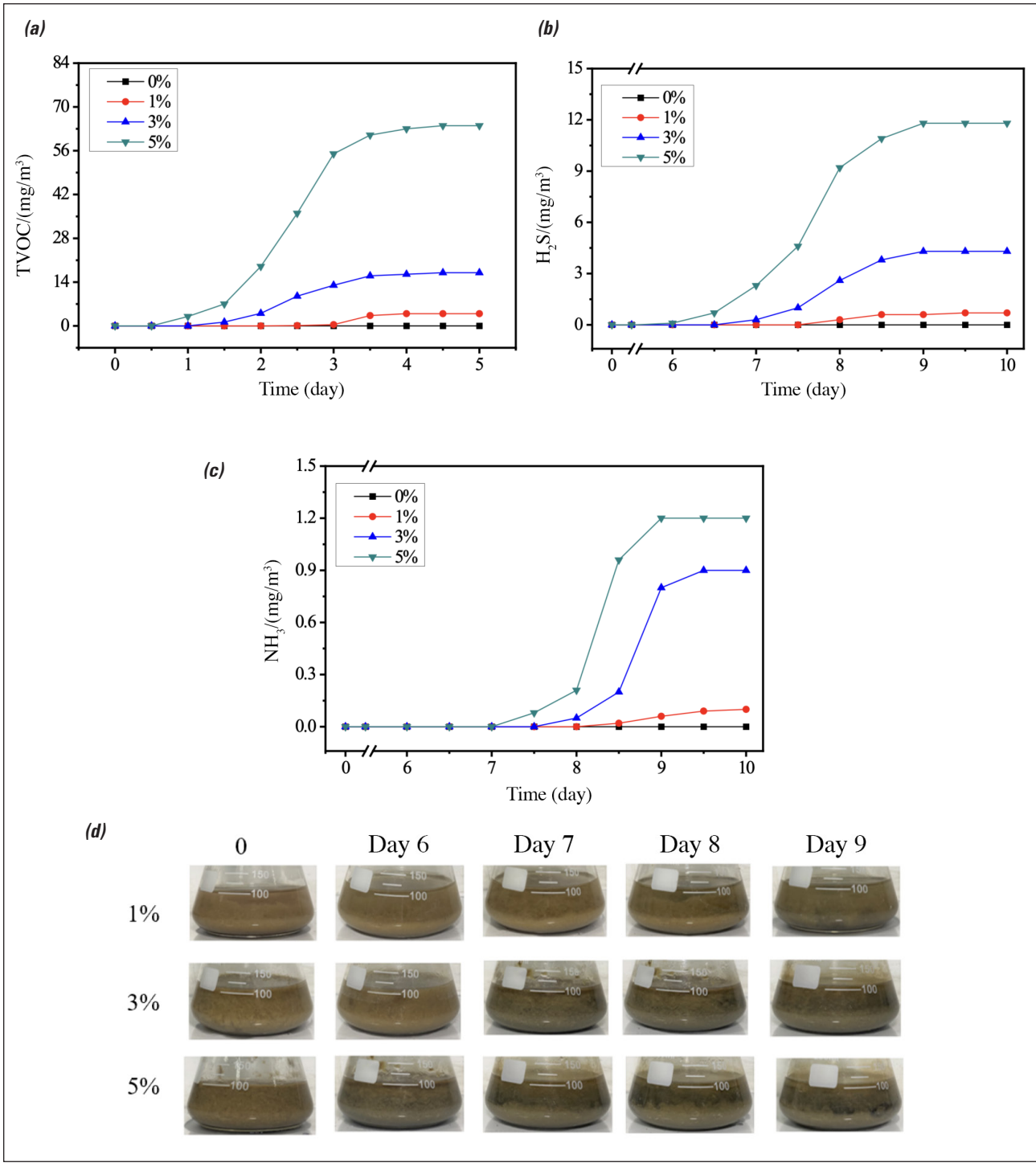
Figures 3a–3c show the effects of secondary fiber concentrations on the release of malodorous gases from the pulp slurry. The release amounts of TVOC, H_2S, and NH_3 exhibited a positive correlation with the secondary fiber concentration. No malodorous gases were detected in the absence of secondary fiber. At the concentration of 1% secondary fiber, the maximum TVOC release was 3.8 mg/m³, and that of 5% secondary fiber was 63.7 mg/m³. Correspondingly, H_2S and NH_3 concentrations were 0.7 mg/m³ and 0.1 mg/m³ at 1% secondary fiber, and rose significantly to 11.8 mg/m³ and 1.2 mg/m³ at 5% concentration, respectively. As shown in **Fig. 3d**, with the increase of the pulp concentration, the amount of black substances increased and appeared earlier, which was because with the increase of secondary fiber concentration, the contents of cellulose, organic matter, microorganisms, and other impurity substances increased [17].



2. (a) Effect of fiber type on total volatile compounds (TVOC) release (LBKP: bleached hardwood kraft pulp); (b) effect of fiber type on hydrogen sulfide (H_2S) release; (c) effect of fiber type on ammonia (NH_3) release; and (d) effect of standing time on color changes in LBKP and secondary fiber. Experimental conditions were: fresh water, temperature = 38°C, slurry concentration = 1.5%.

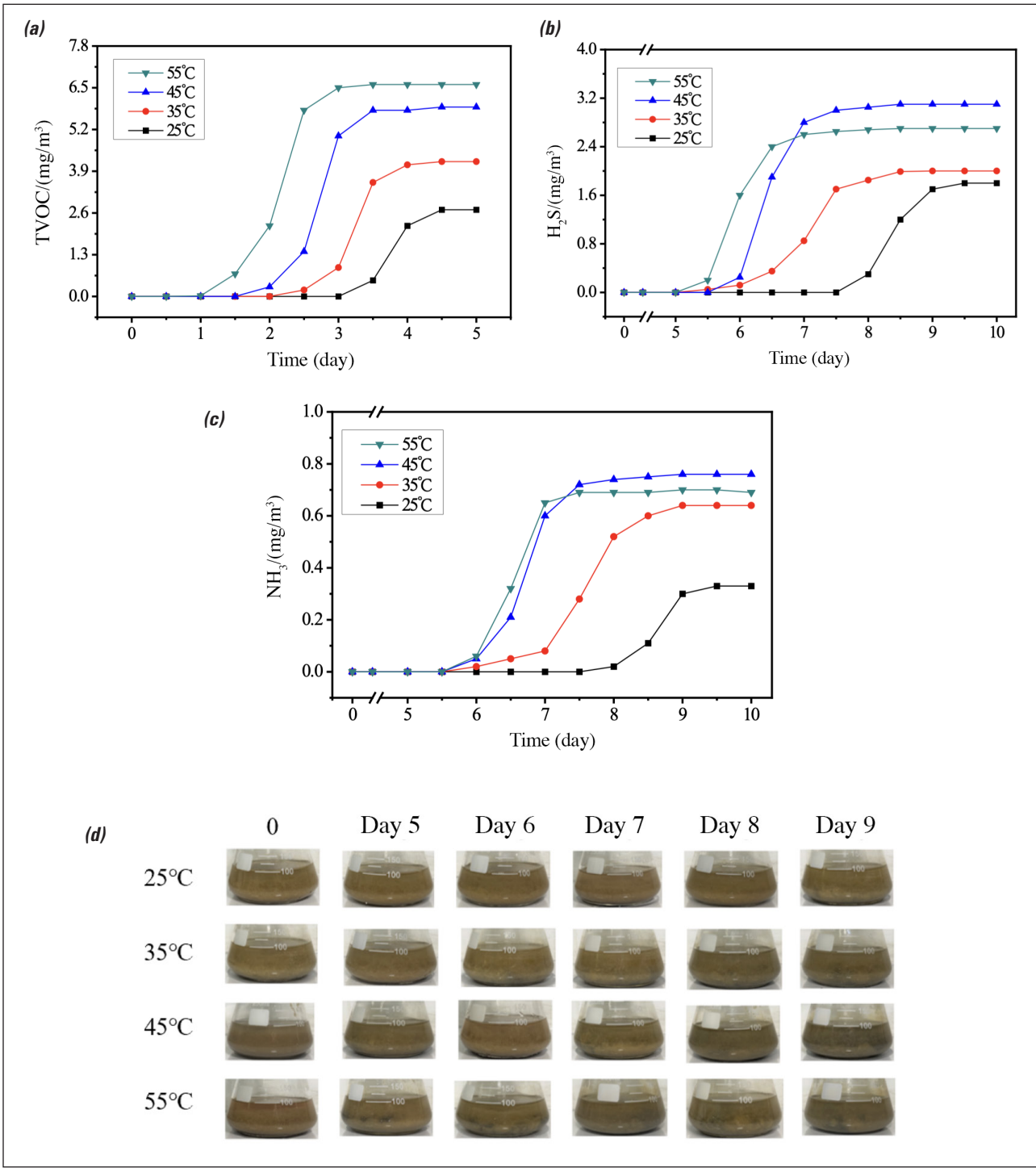
Effect of temperature on odorous gases generation from secondary fiber

Figures 4a–4c illustrate the influence of temperature on malodorous gases emissions from secondary fiber. When the temperature increased from 25°C to 35°C, the release time of TVOC was advanced from day 3 to day 2, and the maximum concentration of the TVOC raised from 2.7 mg/m³ to 4.2 mg/m³. When the temperature rose to 55°C, TVOC began to release on day 1 and reached the maximum value of 6.5 mg/m³ on day 3. This was because the increase in temperature promoted the growth and reproduction of microorganisms, thus producing more TVOC.



3. (a) Effect of secondary fiber concentration on TVOC release; (b) effect of secondary fiber concentration on H_2S release; (c) effect of secondary fiber concentration on NH_3 release; and (d) effect of pulp concentration on secondary fiber pulp color at different time points. Experimental conditions were: fresh water, temperature = 38°C.

In addition, high temperature led to the rapid decline of dissolved oxygen in the pulp slurry, establishing anaerobic conditions conducive to the growth of sulfate-reducing bacteria and other microorganisms [18]. The accelerated anaerobic microbial activity promoted the release of H_2S and NH_3 [19]. On day 10, the concentrations of H_2S and NH_3 at 25°C, 35°C, 45°C, and 55°C were 1.8 mg/m³, 2.1 mg/m³, 3.1 mg/m³, 2.7 mg/m³ and 0.3 mg/m³, 0.6 mg/m³, 0.8 mg/m³, 0.7 mg/m³, respectively. The concentrations of H_2S and NH_3 at 55°C were lower than that at 45°C, which was because most microorganisms, such as sulfate-reducing bacteria and denitrifying bacteria, had high biological activity at 20°C~50°C.

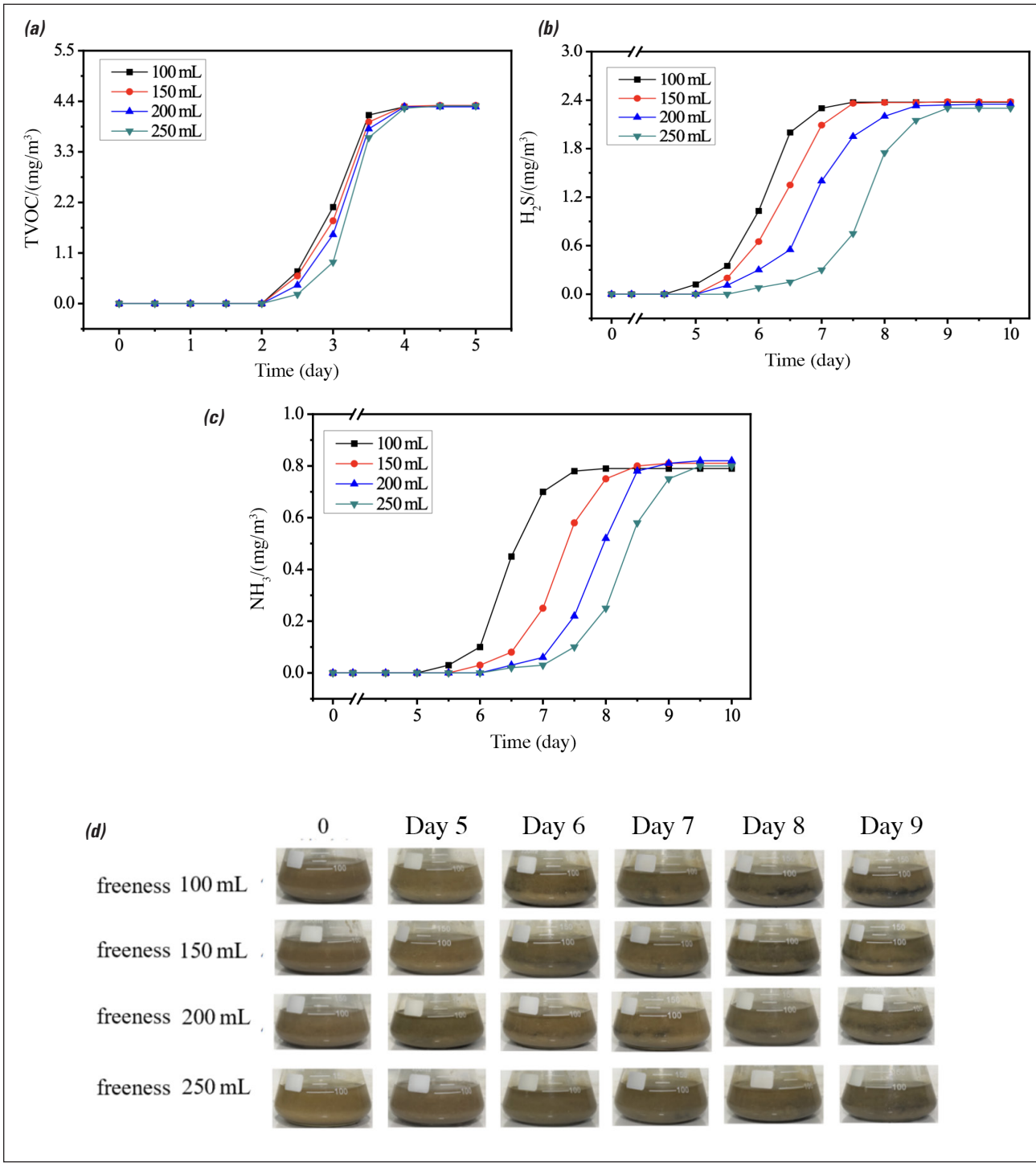


4. (a) Effect of temperature on TVOC release from secondary fiber; (b) effect of temperature on H_2S release from secondary fiber; (c) effect of temperature on NH_3 release from secondary fiber; and (d) effect of temperature on secondary fiber pulp color at different time points. Experimental conditions were: fresh water, secondary fiber concentration = 1.5%.

When it exceeded 50°C, the biological activity of the microorganisms decreased, and the growth and reproduction were inhibited [20]. Consequently, in a suitable temperature range, microorganisms multiplied severely, which promoted the generation of malodorous gases and intensified the blackening of the pulp slurry, as shown in **Fig. 4d**.

Effect of pulp freeness degree on odorous gases generation

As shown in **Figs. 5a–5c**, with the decrease of pulp freeness, the production rates of TVOC, H_2S, and NH_3 all increased gradually; however, their total release amounts were basically the same. Freeness reflects the degree of



5. (a) Effect of pulp freeness on TVOC release.; (b) effect of pulp freeness on H_2S release; (c) effect of pulp freeness on NH_3 release; and (d) effect of pulp freeness on secondary fiber pulp color at different time points. Experimental conditions were: fresh water, temperature = 38°C, pulp concentration = 1.5%.

fiber fibrillation. As the freeness of the pulp decreases, the degree of fiber fibrillation rises. This was attributed to the enhanced fibrillation and resultant larger specific surface area of the fiber at lower freeness degree, which accelerated the microbial reaction rates and the emission of malodorous gases. Nevertheless, the contents of secondary fiber, microorganisms, and other nutrients in the same pulp concentration were basically the same, so the total amounts of malodorous gases released were basically the same [21].

Effect of white water on odorous gases generation

The basic properties of the white water are shown in **Table I**. The white water was collected at the wet-end of a corrugated base paper production line. Its consistency was 0.518%, and the chemical oxygen demand via dichromate (CODcr) was 9320 mg/L, indicating that the white water contained a substantial amount of organic matter, such as fine fibers and starch. In addition, owing to the addition of a certain amount of aluminum sulfate during the secondary fiber recycling process, the concentration of sulfate (SO_4^{2-}) in the white water reached 674 mg/L.

Figures 6a–6c illustrate the influence of white water on malodorous gases emission. In the absence of white water (0 mL), the concentrations of TVOC, H_2S, and NH_3 remained negligible. With the white water/fresh water of 5 mL/95 mL, the malodorous gases concentrations increased slightly. With the white water/fresh water of 20 mL/80 mL, the TVOC release began on day 0.5 and reached 680.3 mg/m³ on day 3.5. With the white water/fresh water of 100 mL/0 mL, the TVOC release began to rise rapidly on day 0.5 and reached 929.6 mg/m³ on day 2, and the trends of H_2S and NH_3 were the same as TVOC.

Figure 6d demonstrates that slurry coloration directly correlated with white-water addition levels. When no white water was added, the slurry color remained essentially unchanged. With the white water/fresh water of 5 mL/95 mL, a small amount of black material appeared at the bottom of the conical flask on day 8. At a ratio of 20 mL/80 mL, the slurry color changed from light yellow to deep black on day 6. While at 100 mL/0 mL, the color of the slurry changed to deep black on day 4. This phenomenon illustrated that white water could promote the generation of malodorous gases and black substances. Granhall et al. [22] found that there were six kinds of microorganisms in white water, such as proteobacteria and firmicutes. Therefore, the microbial activity in white water was the main reason for the production of malodorous gases in pulp [23].

CONCLUSIONS

The following conclusions were reached through this study:

(1) LBKP generated almost no malodorous gases in fresh water. On the other hand, the secondary fiber began to generate TVOC by day 3, with formation of H_2S and NH_3 by day 6. Some black substances were formed in the slurry.

(2) With the increase of the secondary fiber slurry concentration, the release of TVOC, H_2S, and NH_3 gradually increased, and the quality of black matter in the slurry also gradually increased.

(3) With the increase of temperature, the release of TVOC increased steadily, while the releases of H_2S and NH_3 reached the maximum at about 45°C, and then began to decline.

(4) The decrease of the pulp freeness degree accelerated the generation of malodorous gases, but the total amounts of TVOC, H_2S, and NH_3 remained basically unchanged.

(5) With the increase of the white water concentration, the release of TVOC, H_2S, and NH_3 increased rapidly. When the white water/fresh water was 20 mL/80 mL, the slurry changed from light yellow to dark black on day 6, which proved that a large number of microorganisms were produced, and the slurry began to rot, resulting in malodorous gases generation.

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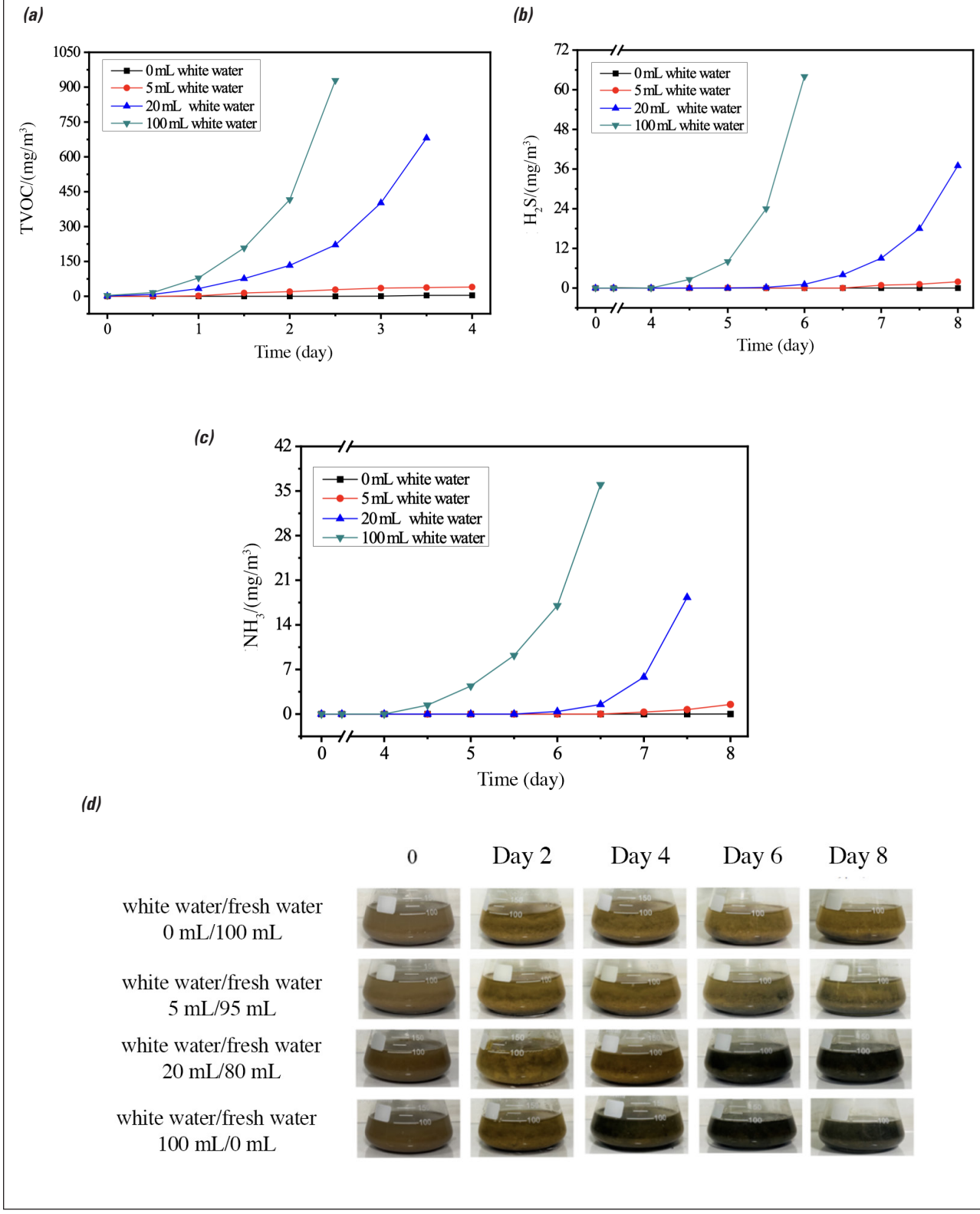
Temperature, °C	Concentration, %	pH	Conductivity, ms/cm	CODcr, mg/L	SO_4^{2-} Concentration, mg/L
46.1	0.518	6.4	5.47	9320	674

CODcr: chemical oxygen demand via dichromate; $SO4^{2-}$: sulfate.

I. Physical and chemical properties of the white water.



6. (a) Effect of white water addition on TVOC formation during secondary fiber recycling; (b) effect of white water addition on H_2S formation during secondary fiber recycling; (c) effect of white water on NH_3 formation during secondary fiber recycling; and (d) effect of white water addition on the color evolution of secondary fiber pulp over different time periods. Experimental conditions were: temperature = 35°C, secondary fiber concentration = 1%.

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ABOUT THE AUTHORS

We choose this topic to research because secondary fibers are prone to generate malodorous gases during the recycling process, which brings certain harm to human health and the environment. We further studied the changes in process conditions, such as pulp concentration and freeness, to investigate the causes of malodorous gases generation during the secondary fiber process. The most challenging part of this study was that due to the experimental instruments requiring regular inspection and calibration by dedicated personnel, our experimental cycle was relatively long.

Through this study, we discovered that in fresh water, bleached hardwood kraft pulp did not produce malodorous gases, whereas secondary fiber began to release malodorous gases around the third day. Furthermore, as the white water concentration gradually increased, a large amount of malodorous gases was released.

It was most interesting to discover that in the pulp containing secondary fiber, total volatile organic compounds (TVOC) began to be released around day 3, while hydrogen sulfide (H_2S) and ammonia (NH_3) began to be released in large quantities around day 6. In addition, the color change of the pulp was not obvious. When white water was added to fresh water, malodorous gases were rapidly released, and the pulp color changed from light yellow to deep black; as the concentration gradually increased, the time for the pulp color to change was further shortened.

This study demonstrated that mills could change the relevant process conditions to control the release of malodorous gases during the recycling process of secondary fibers and promote clean production in the paper industry. Our next step is to use chemical methods to control the release of malodorous gases during the recycling process of secondary fiber.

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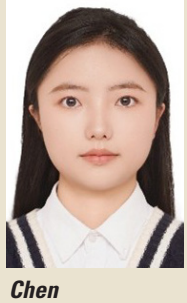
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