



Emission characteristics of volatile compounds during sludges drying process

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ABSTRACT

The emission characteristics of volatile compounds (VCs) during municipal sewage sludge (MSS) and paper mill sludge (PMS) drying process were investigated through experiments conducted on a lab-scale tubular drying furnace and a pilot-scale paddle dryer, respectively. The result indicated that five kinds of VCs, i.e. CO₂, NH₃, C₇H₁₆ (*n*-heptane), volatile fatty acids (VFAs) and CH₄ were emitted during the drying process. It was found that the NH₃ and CO₂ were the primary compound released from the MSS drying process. In the case of the PMS, the VFAs and CO₂ were the main compounds released. The temperature and water content of sludge had great effects on the emission rates of NH₃, C₇H₁₆, CO₂ and VFAs. The pH and chemical oxygen demand (COD) of condensate from the paddle dryer were also studied. It showed that pH and COD of condensate from MSS were much higher than that from the PMS, and that the higher COD value of the MSS condensate interrelated to the higher ammonium and sulfur content of it.

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1. Introduction

The production of sludge from wastewater treatment plants has been continuously increasing for several years in China. In addition to this increase, Chinese legislation is being strengthened in regard to sludge dumping and its use in agriculture. For instance, in the near future and after a transition period, the deposition of sludge at dump sites will be permitted only if the water content of sludge is lower than 60 wt%.

Thermal drying of sludge is a necessary intermediate common to all disposal methods, as it makes it possible to stabilize the sludge, reduce its volume and hygienize the product. Numerous researches have been carried out in both lab and full scale to investigate various parameters that determine the sludge drying efficiency. But very few researches were focused on the emission characteristics of volatile compounds (VCs) during sludge drying process. Vesilind and Ramsey [1] indicated that the most common volatile emissions identified from sludge incineration in multiple hearth furnace (MHF) were benzene, toluene and acrylonitrile, and these VCs emitted from the drying hearths of the MHF (300–450 °C). However, this result addresses to incineration at high temperature. The published researches focused on VCs emissions at much lower temperature during sludge drying are still lacking. Reimann [2] showed that thermal conditioning of sludge led to partial release of its ammonia with water vapour, and another report [3] showed that the condensate from a sewage sludge dryer con-

tained 190 and 2310 mg/L of NH₃ during drying temperature of 150 and 350 °C, respectively. In fact, plenty of researches have been reported on the VCs emissions in wastewater treatment plants, and these reports are helpful for the study of VCs emissions in sludge drying. Many odorous compounds associated with wastewater treatment have been identified. Among them, reduced organic sulfides, hydrogen sulfide and amines are predominant [4,5]. Dincer and Muezzinoglu [6,7] studied odor determination at waste water collection systems and sludge treatment areas, and showed the relations between VCs concentration and odor problem of the wastewater plant. Gostelow et al. [5] reviewed odorants measurement and listed 39 typical kinds of odorants associated with sewage treatment works, including reduced sulfur bearing compounds, amine compounds, organic acids, aldehydes and ketones. However, detailed researches on the species and emission characteristics of VCs during sludge drying process were not investigated yet and these characteristics may be important. Analytical measurements are complicated by the large number of VCs present, often at concentrations close to detection limits. It is almost impossible to identify all VCs emissions by one-off measurement. Therefore, the main objectives of this study were to identify the VCs with high concentrations that can be easily identified during sludge drying process. Fourier transform infra-red spectroscopy (FTIR) can be used to prove the existence of some emissions. It provides plenty of information of mixed gases, and can be used to identify the composition of mixture. Online FTIR analysis is a useful method in dynamic analysis as it monitors continuously the time dependent evolution of the gases. It is commonly used for online, real-time measurement of volatile organic compounds from gaseous samples [8].

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Table 1
Proximate and ultimate analyses of the MSS and PMS (dry basis)

Species (mass fraction)	MSS	PMS
Ash content (%)	52.86	49.73
Volatile content (%)	43.15	42.37
Fixed carbon content (%)	2.52	3.94
C (%)	26.05	16.94
H (%)	4.19	3.64
N (%)	2.83	1.07
S (%)	3.09	1.66
O (%)	11.1	23.00
Cl (mg/kg)	63.6	915.5

In this study, the main volatile products from municipal sewage sludge (MSS) and paper mill sludge (PMS) drying were identified by an online FTIR analyzer, the formation mechanisms of these compounds were determined, and the effects of water content and drying temperature on emission rate of these compounds were evaluated.

2. Materials and methods

2.1. Materials

Two very different kinds of mechanically dewatered sludge were used in this study, i.e. MSS and PMS, with water content of 3.69 kg H₂O kg⁻¹ dry solid (DS) for the MSS and 4.85 kg H₂O kg⁻¹ DS for the PMS, respectively. The MSS was sampled from QIGE municipal sewage treatment plant in Hangzhou city of Zhejiang province, and the PMS was sampled from a paper-making factory in Pinghu city. Both kinds of sludge were produced from mechanical dewatering of activated sludges which were produced from aeration basins of anaerobic/oxic (A/O) wastewater treating system. The results of the proximate and ultimate analyses of the both sludges are given in Table 1. Large amounts of the organically bound fuel-nitrogen, sulfur, volatile matter and ash were observed.

2.2. Apparatus

Gaseous samples emitted from sludge drying were continuously measured by Gasetm DX-4000 Gas Analyzer which was composed of two units: the sampling unit for gases sampling and cleaning, and the measuring unit for continuous analysis with FTIR analyzer. Infra-red spectra obtained were processed by Calcmeter Software 2005. Metrohm-792 Basic Ion Chromatography was used for ammonium and aqueous sulfate determination. The pH and chemical oxygen demand (COD) of condensate were measured by LP115FK PH meter and 5B-3 COD meter, respectively.

2.3. Experiment procedure

2.3.1. Batch drying test

Batch drying test was conducted on a tubular drying furnace (the length of heating zone was 400 mm). The length and inner diameter of the quartz tube were 1150 and 24 mm, respectively (Fig. 1). The experiment commenced by introducing about 5 g of sludge sample into the center of the drying chamber where the temperature kept constant at 160 °C, and drying took place until sludge was dried completely. The sludge was placed in a silica boat until the container edge and sludge surface were level. A depth of sludge of 1 cm was used and the dry air flowrate in the drying chamber was 3.2 L/min. Gaseous samples were continuously outleted into gas analyzer for components analysis. Duplicated experiment was conducted to confirm the reproducibility of the result.

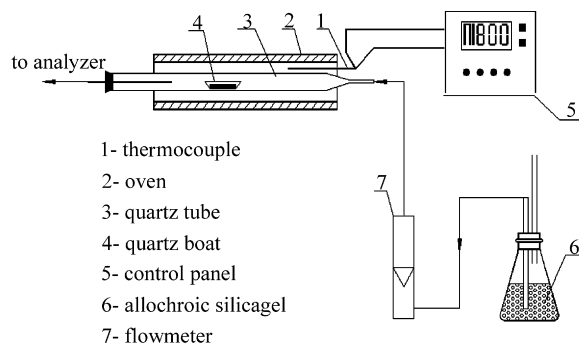


Fig. 1. Schematic diagram of batch drying test.

2.3.2. Continuous drying test

Continuous drying test was carried out on a paddle dryer. The dryer was constructed with paddles and two hollow shafts. Fig. 2 shows the flow diagram of the test. The heat-transfer oil was electrically heated in the oil tank, and then passed through the shafts and paddles as well as the jacket of the shell walls. Sludge was thermally dried by the heat transferred from the oil. The heating power of the electrical heater was 18 kW and drying capacity of the paddle dryer was about 30 kg/h. During the drying process, the dewatered sludge cakes were continuously supplied to one end of the dryer and was dried while being transferred to the other end. Water evaporated during the drying process was extracted outside the dryer with carrier gas (dry air) by an induced fan. Wet air was introduced into a condenser and condensate was collected for further pH and COD measurement. VCs were measured at points A and B (Fig. 2), respectively.

3. Results and discussion

3.1. Emission characteristics of VCs during batch drying process

In this test, the sludge drying process was described as follows: firstly, the temperature of the sludge sample increased rapidly

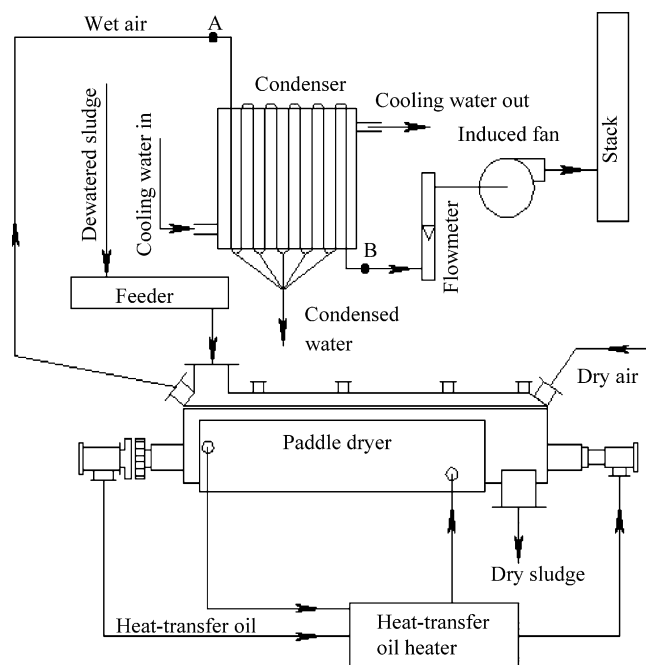


Fig. 2. Flow diagram of continuous drying test.

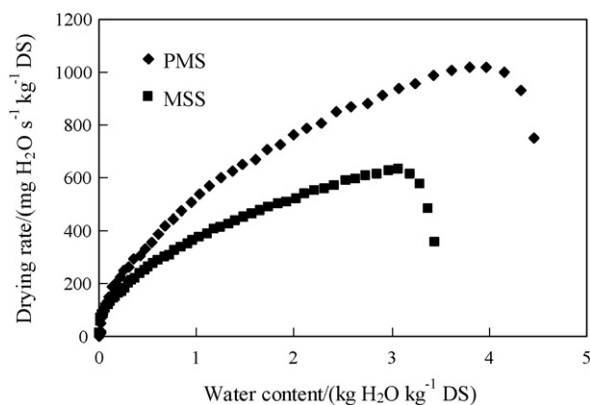


Fig. 3. Drying rate curves of PMS and MSS (drying temperature $T = 160^\circ\text{C}$, air velocity $V_a = 0.108\text{ m/s}$).

from ambient to experimental temperature. Then, the evaporation surface shrank from sludge surface to the interior of the sludge, and the sludge volume was reduced with the loss of moisture and volatile matters. The rate of water vapour evolution was measured by measuring the air humidity discharged from the drying chamber by FTIR analyzer, and the rate of moisture loss (Fig. 3) was back-calculated. As shown in Fig. 3, the drying rates of both sludges markedly increased at the beginning of the drying process due to the increase of the sludge temperature. The drying rate of the PMS was higher than that of the MSS for the same water content, because of different physical properties.

As suggested by Vesilind and Ramsey [1], sludge sample would lose 10% of high heating value when the drying temperature was around 150°C . This indicated that the drying and loss of volatile substances occurred in parallel in sludge drying process, and that the loss of volatile substances played an important role when the drying temperature was higher than 150°C [9]. In this test, the drying temperature was electrically controlled at 160°C . Duplicated experiment was conducted to confirm the reproducibility of the result. The differences for the results of two parallel tests was existent which was caused by inhomogeneity of the sludge samples, but the percentage error of the tests were below 5%, and the result discussed below came from one of the two tests. As shown in Fig. 4, FTIR spectra of gaseous samples were measured, and four kinds of volatile compound, i.e. NH_3 , C_7H_{16} , CO_2 and volatile fatty acids (VFAs), were clearly identified.

3.1.1. NH_3 emission

As shown in Fig. 5, the NH_3 emission rates of the MSS and PMS passed through three stages in the drying process, i.e. the rising rate stage which interrelated to the increase of sludge temperature, then the constant rate stage which was between $0.75\text{--}2.33\text{ kg H}_2\text{O kg}^{-1}\text{ DS}$ for the MSS and $0.49\text{--}1.63\text{ kg H}_2\text{O kg}^{-1}\text{ DS}$ for the PMS, and finally the decreasing rate stage where the NH_3 emission rate dropped rapidly from the high level. In this study, the NH_4^+ concentrations of the MSS and PMS solutions were measured by ion chromatography. The NH_4^+ concentration was $5.46\text{ g kg}^{-1}\text{ DS}$ for the MSS and $0.28\text{ g kg}^{-1}\text{ DS}$ for the PMS, respectively. The higher NH_4^+ concentration of the MSS may contribute to the higher NH_3 emission during the sewage sludge drying process. It has been reported that the NH_3 emitted from sludge drying was formed through hydrolysis of protein [10]. When the protein in sludge dissolves, it hydrolyzes to form multipetide, dipeptide and amino acid. The amino acid further hydrolyzes to form organic acid, NH_3 and CO_2 [11].

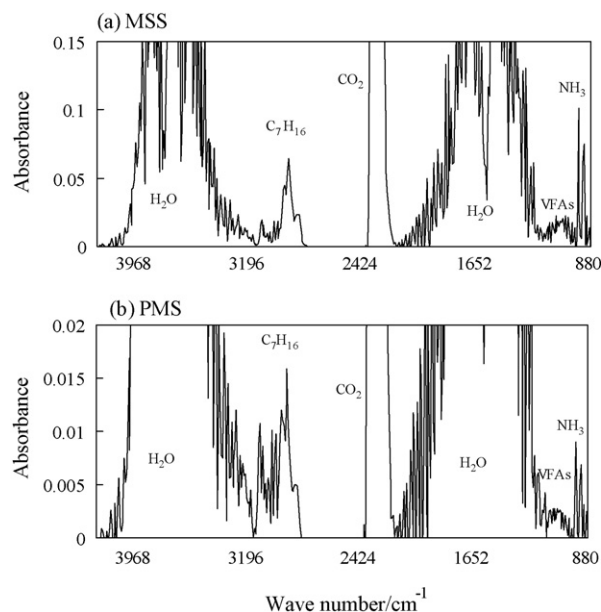


Fig. 4. Infra-red spectra of gaseous samples from the drying of (a) MSS (at $0.11\text{ kg H}_2\text{O kg}^{-1}\text{ DS}$) and (b) PMS (at $0.02\text{ kg H}_2\text{O kg}^{-1}\text{ DS}$) (drying temperature $T = 160^\circ\text{C}$, air velocity $V_a = 0.108\text{ m/s}$).

3.1.2. C_7H_{16} emission

The emission rate curves of the C_7H_{16} shown in Fig. 5 were much different than that of the NH_3 . The distribution of the water in sludge plays an important role in the emission of volatile

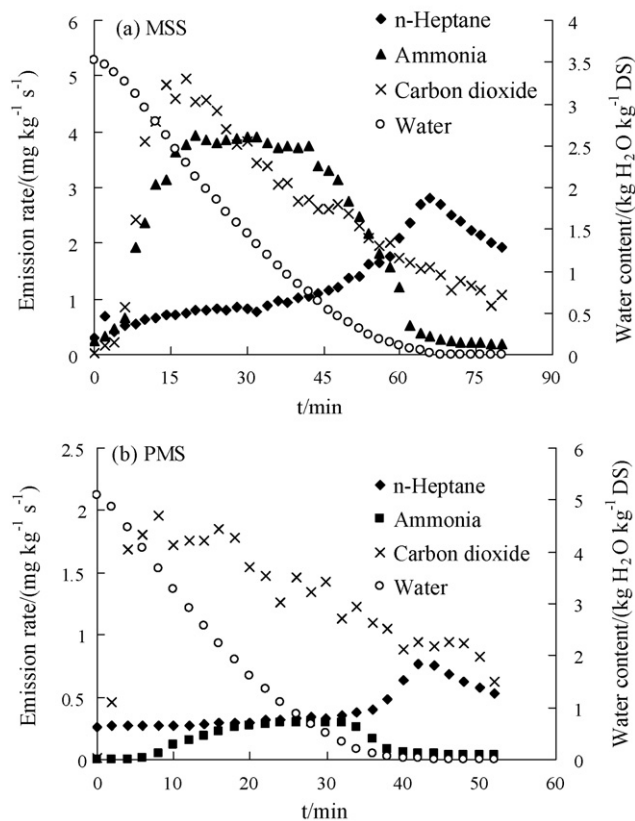


Fig. 5. Time-resolved C_7H_{16} and NH_3 emission rate curves and water content of (a) MSS and (b) PMS during drying experiments (drying temperature $T = 160^\circ\text{C}$, air velocity $V_a = 0.108\text{ m/s}$).

organic compounds. Rudolfs and Baumgartner [12] assumed that volatile matters were not driven from a sludge sample until 80–90% by weight of the original moisture content of the sludge had evaporated given that residual moisture was evenly distributed throughout the sludge cake. However, the sludge surface temperature was higher than the interior of the sludge cake before the sludge was totally dried, and the exposed sludge surface areas tend to dry faster than those buried within the sludge cake. Therefore, the C_7H_{16} was firstly driven from dry sludge surface. As shown in Fig. 5, the C_7H_{16} emission rate of the MSS moderately increased before the water content reaching to about $0.43 \text{ kg H}_2\text{O kg}^{-1} \text{ DS}$, and then followed by a marked increase. The C_7H_{16} emission rate reached a peak value of $2.80 \text{ mg kg}^{-1} \text{ s}^{-1} \text{ DS}$ when the MSS was totally dried. After that, it decreased rapidly until reached a constant value. In the case of the PMS, the C_7H_{16} emission rate started a marked increase until the water content decreased to about $0.11 \text{ kg H}_2\text{O kg}^{-1} \text{ DS}$, and then reached a peak value of $0.76 \text{ mg kg}^{-1} \text{ s}^{-1} \text{ DS}$ which was much less than that of the MSS. It was obvious that the increase of the C_7H_{16} emission rate was followed by the decrease of sludge drying rate. Since C_7H_{16} contributes to the calorific value of sludge, the loss of calorific value would become more and more significant in the drying process.

There are two possible ways for the C_7H_{16} formation. It might be present as individual component or be formed from the thermal degradation of more complex organics. C_7H_{16} is a volatile compound, with the boiling point 98.5°C . Thus, it should be expected that most of C_7H_{16} would be evaporated with water at the beginning of the drying process, given that C_7H_{16} was present in large quantities as individual component in sludge. However, Fig. 5 shows that the C_7H_{16} emission rate kept at relatively low level and increased mildly between the time of 0–40 min, and it reached the maximum until the sludge was completely dried. So it was reasonable to conclude that the C_7H_{16} was mainly formed from the thermal degradation of more complex organics.

3.1.3. VFAs emission

Three kinds of VFAs, i.e. propionic acid, acetic acid and formic acid, were detected during the drying process (Fig. 6). Fig. 6a shows the VFAs emission rates of the MSS. It was found that the VFAs emission rates markedly increased at the beginning of the drying process. After that, their emission rates gradually decreased with the loss of moisture content. As for the PMS, the emission rate of formic acid was negligible compared with the other two acids, and the propionic acid had a constant emission rate between sludge moisture contents of $0.8\text{--}3.8 \text{ kg H}_2\text{O kg}^{-1} \text{ DS}$.

It has been widely reported that the VFAs could be produced from hydrothermal treatment of organic matters [13–15]. In these reports, the VFAs were formed from hydrolysis of MSS, PMS, and plastic waste, etc. However, only a few researches about the formation of VFAs from thermal drying of sludge have been reported [10]. It indicated that the VFAs released from thermal drying process were also formed through the hydrolysis of organic matters. Because of the substantially lower temperature and pressure under which the drying test was conducted, the VFAs emission rates were

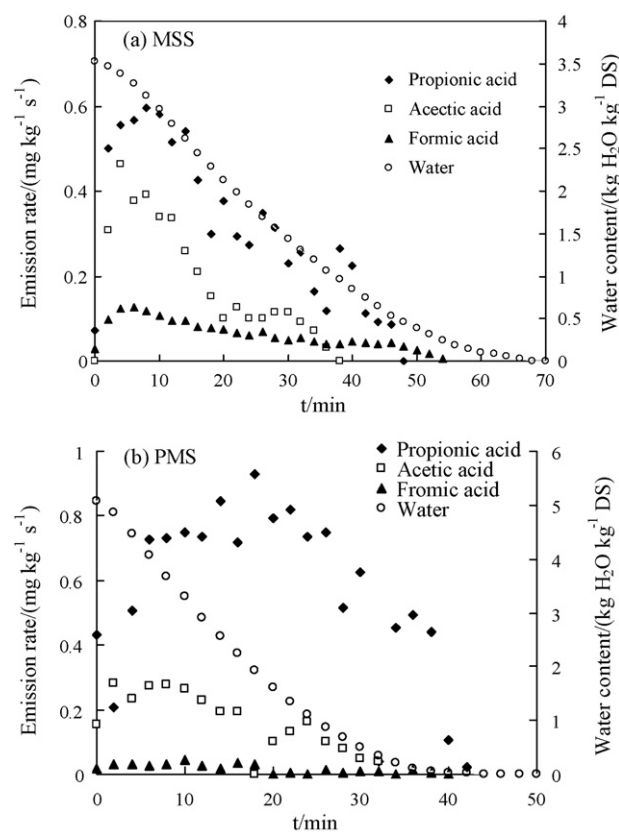


Fig. 6. VFAs emission rates vs. water content during drying of (a) MSS and (b) PMS (drying temperature $T = 160^\circ\text{C}$, air velocity $V_a = 0.108 \text{ m/s}$).

much lower than those of hydrothermal treatment process. In addition, when the water content of the sludge decreases, the hydrolytic action will be weakened, as well as the VFAs emission rates. As shown in Fig. 6, there were no VFAs emission when the MSS and PMS were totally dried.

3.1.4. CO_2 emission

As shown in Fig. 1, the dry air was used as carrier gas in the batch drying test. Therefore, the total CO_2 concentration measured during the drying process included the part emitted from sludge drying and the part existed in the dry air. The CO_2 emission rates shown in Fig. 5 was calculated by subtracting the CO_2 concentration in dry air from the total CO_2 concentration. It was obvious that the CO_2 emission was significant compared with other VCs emissions. There was a sharp increase of the CO_2 emission at the early state of drying processes for both kinds of sludge. After reaching the peak value, the CO_2 emission rate dropped continuously with time. The CO_2 emission rate of the MSS was much higher than that of the PMS, and the peak value of the CO_2 emission of the MSS was more than twice over that of the PMS.

Table 2

Data of continuous drying tests

Sludge type	MSS				PMS			
	1	2	3	4	5	6	7	8
Test no.	1	2	3	4	5	6	7	8
Heating oil temperature ($^\circ\text{C}$)	140	150	160	170	140	150	160	170
Feed rate (kg/h)	15	14.5	13.5	16.8	13.2	12.8	12.4	15.1
Inlet moisture ($\text{kg H}_2\text{O kg}^{-1} \text{ DS}$)	3.69	3.69	3.69	3.69	4.85	4.85	4.85	4.85
Discharge moisture ($\text{kg H}_2\text{O kg}^{-1} \text{ DS}$)	1.91	1.12	0.97	1.01	0.42	0.32	0.31	0.35
Paddle rotation (rpm)	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5
Air flow rate (Nm^3/h)	17.7	15.8	16.4	15.6	18.4	18.2	17.8	17.2

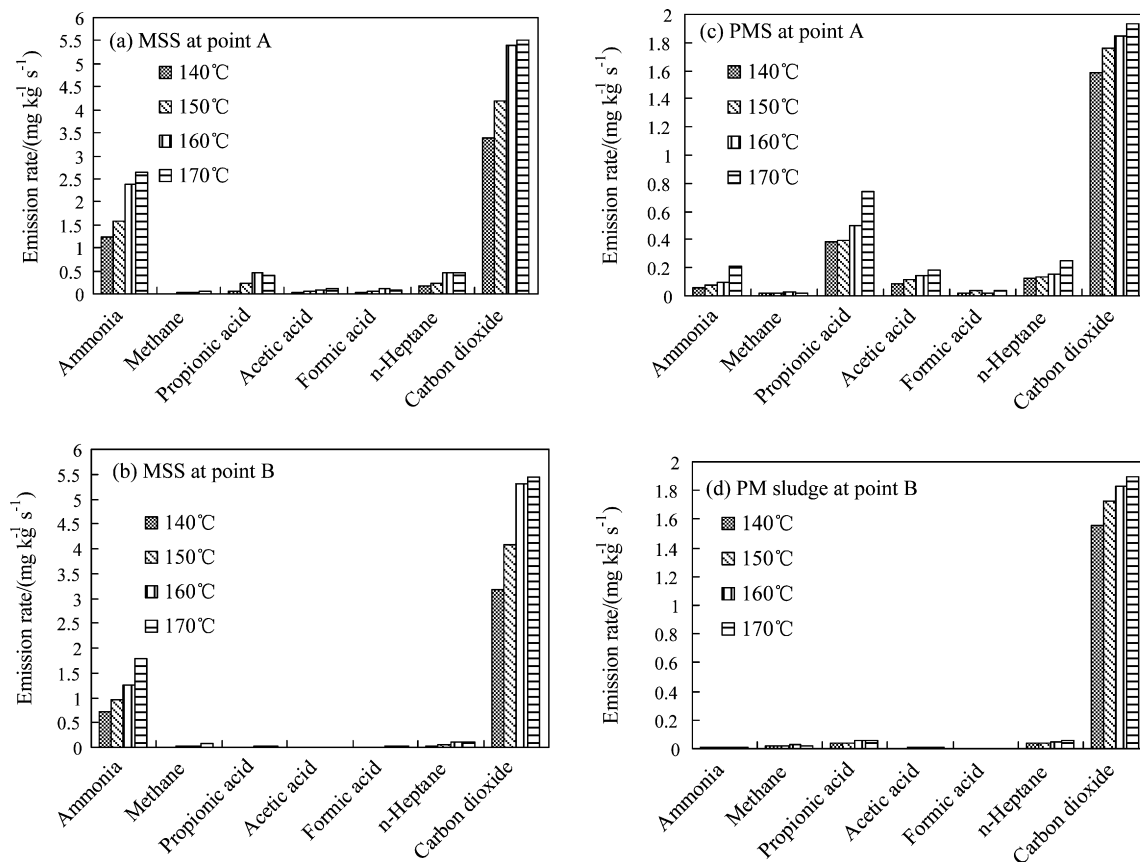


Fig. 7. Effect of heating oil temperature on the emission rates of ammonia, methane, *n*-heptane and VFAs for MSS and PMS at points A and B, respectively.

There were at least two possible ways responsible for the CO₂ emission. One way is that CO₂ was formed from the hydrolysis of amino acid which has been discussed above, and the other way is that it was formed from the decomposition of bicarbonate. The HCO₃⁻ concentrations of both sludge solutions were measured based on DZ/T 0064.49-93 method. In this method, HCO₃⁻, CO₃²⁻ and OH⁻ of aqueous solution are titrated quantitatively by hydrochloric acid through phenolphthalein indicator and methyl orange indicator. The result showed that there were no CO₃²⁻ and OH⁻ in both aqueous sludge solutions. The HCO₃⁻ concentration were 15.4 g kg⁻¹ DS for the MSS and 12.7 g kg⁻¹ DS for the PMS. Therefore, it is reasonable to infer that part of the CO₂ was emitted from decomposition of bicarbonate during the sludge drying process.

3.2. Emission characteristics of VCs during continuous drying process

In the continuous drying test, sludge was transferred continuously from the feeding end of the dryer to the product discharge at the other end as it was being dried, and the retention time of sludge in the dryer was 30 min when the paddle

rotation speed was 2.5 rpm. Table 2 presents the experimental conditions of the test. There were four tests for each MSS and PMS, respectively. The tests began as soon as the temperature in the dryer was kept constant for half an hour, each test lasted at least 60 min. The gas analysis was conducted all through the test, and the ultimate result of the test was the average of it.

Five kinds of VCs, i.e. NH₃, C₇H₁₆, CO₂, VFAs and CH₄ were identified from the tests. Fig. 7(a and c) shows their emission rates at four different drying temperatures. It is obvious that the CO₂ emission rates of the MSS and PMS was markedly higher than other four compounds, and that the higher drying temperature led to the higher emission rate of the VCs. When the drying temperature increased from 140 to 170 °C, the NH₃ emission rate increased by 120%. However, as shown in Fig. 7c, the NH₃ emission rate of the PMS was much lower than that of the MSS, and the CO₂ and VFAs were the main compounds emitted from the PMS drying process. In the case of the PMS, the higher drying temperature also led to higher emission rate of VCs, except the CH₄ and formic acid. It was found that the propionic acid had a marked increase when the drying temperature increased from 160 to 170 °C. It was interesting that the

Table 3
The condensate characteristics of the MSS and PMS

Sludge type	MSS				PMS			
	140	150	160	170	140	150	160	170
Heating oil temperature (°C)	140	150	160	170	140	150	160	170
Condensate temperature (°C)	36.2	42.3	49.7	52.3	41.1	44.2	49.3	52.3
pH	9.62	9.73	9.8	9.71	5.81	5.53	5.41	5.34
COD (mg/L)	650.4	772.2	1079.5	1012.5	497.0	528.0	528.5	724.8
NH ₄ ⁺ (mg/L)	796.8	822.8	898.8	856.1	11.5	17.9	22.7	30.5
Sulfur content (mg/L)	8.4	9.5	10.0	11.1	1.1	1.1	1.5	3.1

CH₄ which was not found in the batch drying test was identified in the continuous drying test. The reason is that the concentration of the CH₄ reached the detectability of the gas analyzer, because of much higher sludge handling capacity in the continuous drying test.

All the VCs identified above are regarded as hazardous or contaminative chemicals [16]. NH₃ is a toxic and corrosive air pollutant with extremely pungent odor, and is a systemic irritant by inhalation; heptane is a toxic and colorless liquid with gasoline-like odor; VAFs is a common odorous contaminant which has been widely reported [17,18]; CO₂ and CH₄ are green house gases that will induce global warming effects. Besides the main VCs studied above, the reduced organic sulfides, hydrogen sulfide and amines which should be measured by other methods were not studied in this paper. They were also common toxic and odor-causing pollutants, and will be further studied in the future work. Therefore, it is very important to control the emission of VCs during sludge drying process. As shown in Fig. 2, a condenser was equipped to collect moisture and volatile matters from the paddle dryer. Fig. 7(b and d) shows the emission rate of the VCs leaving from the condenser. It was found that more than 90% of VFAs and 70% of *n*-heptane were collected in the condensate. Only 33–47% of the NH₃ from the MSS was collected, because of its high emission rate. In the case of CH₄ and CO₂, however, they were almost not condensed at all. Therefore, further purification of gas is needed before it being released into environment.

The pH values of condensate were measured, and it was found that they were strongly interrelated to the emission rates of VCs. As shown in Table 3, the condensate from the MSS was basic due to high NH₃ emission. Similarly, high VFAs emission resulted in acidic property of condensate from the PMS. However, the effect of drying temperature on the pH of the condensates was insignificant. Table 3 shows that the COD values for the condensates of the MSS was much higher than that of the PMS, which indicated that the condensates of the MSS contained much more chemically oxidizable organics. The higher NH₃ and *n*-heptane emission rates of the MSS are interrelated to its higher COD levels. The NH₄⁺ and aqueous sulfate solutions of the condensates were determined quantitatively by ion chromatography, and the aqueous sulfate solutions which might be from sulfur bearing compounds were oxidized by peroxide addition. The result in Table 3 shows that the NH₄⁺ and sulfur content of the MSS were markedly higher than that of the PMS. The result also confirmed the emissions of sulfur bearing compounds which cannot be measured by FTIR analyzer, but they were at much lower emission level compared with NH₃ emission.

The VCs emission ability of the MSS and PMS were significantly different. Almost every kind of VCs emissions of the MSS measured in the experiments were higher than that of the PMS, especially in NH₃ emission. These discrepancies were caused by the differences in sludge properties. MSSs are a residue resulting from the treatment of wastewater released from various sources including homes, industries, medical facilities, street runoff and businesses, while PMSs are from treatment of wastewater released from paper manufacturing. The major organic loading of MSS is a complex mixture of fats, proteins, carbohydrates, humic material and fatty acids [19]. However, PMS contains mainly cellulose, varying amounts of highly lignified materials, and molecules of anthropogenic origin depending on the paper manufacturing practices used [20]. The organic matters of the MSS and PMS were not measured in this article, but it can be found from the ultimate analysis of the sludges (Table 1) that the higher ammonia emission and sulfur content of the condensate interrelated to the higher nitrogen and sulfur content in the MSS.

4. Conclusion

An online FTIR analytical method has been used for determining the main VCs emissions during the MSS and PMS drying. In the batch drying test which was conducted on a tubular drying furnace, four kinds of volatile matters, i.e. NH₃, C₇H₁₆, CO₂ and VFAs were identified. The results indicated that these four kinds of compounds were formed through different ways, and their emission characteristics were also different. The NH₃ emission rate passes through three stages, i.e., the rising rate stage, the constant rate period and the decreasing rate period. The C₇H₁₆ emission rate continuously increased along with the loss of moisture, and then began to decrease when the sludge was totally dried. In the case of the VFAs, when the water content of the sludge decreases, the hydrolytic reaction will be weakened, as well as the VFAs emission rates.

NH₃, C₇H₁₆, VFAs, CO₂ and CH₄ were identified in the continuous drying test. The moist air collected from the dryer underwent condensing before it was released to the atmosphere. The high NH₃ emission rate led to basic condensate of the MSS, but the condensate of the PMS was acidic owing to relatively high concentration of VFAs dissolved. The higher COD value of the MSS condensate interrelated to the higher ammonium and sulfur content of it. Besides the odorous contaminants, it is also important to note that the green house gases, CO₂ and CH₄, were found in the MSS and PMS drying. In fact, CO₂ is highest of all the five VCs identified. Therefore, further treatment of these gases are needed.

Acknowledgements

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