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# Composition and Accumulation of Fouling during Leachate Reverse Osmosis Membrane Treatment in both a Typical Incineration Plant and a Landfill

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**Abstract:** Reverse osmosis (RO) membrane process has been extensively used for leachate treatment both in incineration plants and landfills. However, operation and maintenance costs caused by membrane pollution limit the wide application of RO membrane. In order to further understand membrane fouling, this study analyzes the influent, effluent, concentrated liquid pollutants and membrane fouling of typical waste incineration plants and landfills in Chongqing, a city in the west of China based on the comparison of three-dimensional excitation and emission matrix fluorescence (3DEEM) and nanometer particle size. In addition, this paper discusses the change law of chemical oxygen demand (COD) and main pollutants in membrane fouling in the disc tube reverse osmosis (DTRO) system in a single operation cycle, analyzes the main components of membrane fouling, and draws the accumulation of RO membrane pollutants, in order to provide a reference for understanding the composition, formation and control of membrane fouling.

**Key words:** reverse osmosis (RO) process; leachate treatment; properties of membrane fouling; fouling formation; incineration plant; landfill

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## 0 Introduction

Reverse osmosis (RO) membrane technology, superior in high efficiency and good treatment effect, is a common advanced treatment technology for landfill leachate. However, membrane fouling will appear on the RO membrane with the operation of the treatment system, leading to membrane blockage, increased operation pressure, reduced water production rate, and other problems<sup>[1]</sup>. The adsorbed pollutants on the membrane surface and in the membrane pores are the main reason for the reduction of membrane flux and the deterioration of treatment effect. The pollutants of RO membrane caused by leachate are organic matter mainly composed of humic acid, fulvic acid and protein-like substances, as well as inorganic matter and colloids such as calcium and magnesium<sup>[2-4]</sup>. The properties of humic acid and fulvic acid in the leachate are similar, and their main bodies are aromatic compounds. The molecular weight of fulvic acid is relatively small, generally below 1 000, while that of humic acid is relatively large, generally between 1 000 and 10 000. Since pollutants are difficult to remove, membrane fouling is often irreversible. The system no longer works when its operating pressure reaches the established value. In order to keep it running, the RO membrane needs to be cleaned and replaced, thus increasing operating costs and limiting the promotion of RO membrane technology.

Incineration and landfill are the mainstream ways of treating municipal solid waste (MSW) in China<sup>[5]</sup>. The waste entering incineration plant has a short fermentation and oxidation time, and the leachate produced has a high moisture content and contains a large amount of biodegradable organic matter (such as volatile fatty acids). Therefore, the leachate of incineration plant has high chemical oxygen demand (COD) concentration and good biodegradability. Besides, it generally takes a long time to landfill and biodegrade wastes (generally more than 3 years). The leachate from landfill contains a large amount of humus high in humification degree and difficult to degrade (such as fulvic acid and humic acid), so it contains a low-concentration COD but demonstrates poor biodegradability. The pollution of RO membrane is related to the type, nature and concentration of influent pollutants<sup>[6]</sup>. The fouling process and mechanism of RO membrane may be different due to the different water quality of the two leachate.

At present, RO membrane technology is widely used in leachate treatment in incineration plant and landfill. In order to solve the problem arising from RO membrane fouling during leachate treatment, many researches have been conducted<sup>[7,8]</sup>. However, most of the current studies only focus on the cleaning of RO membrane<sup>[9-11]</sup>, while there are relatively few studies on the composition analysis of membrane scale and its cumulative process. And there is no clear understanding of the types and quantities of pollutants, and there is no report on the comparison of RO membrane scaling mechanism between incinerator and landfill leachate treatment.

In this study, on the basis of three-dimensional excitation and emission matrix fluorescence (3DEEM) and nano-particle size comparative analysis of influent, effluent, concentrated liquid pollutants and membrane fouling from the disc tube reverse osmosis (DTRO) in

typical incineration plants and landfill in Chongqing, a city in the west of China, the main components of membrane fouling were identified. Then, by discussing the change law of COD and main pollutants in the inner membrane pollution of DTRO system during a single operation cycle, the accumulation process of RO membrane pollutants was obtained, which provided a reference for understanding the composition, formation and control of membrane pollution.

## 1 Materials and Methods

### 1.1 Experimental Materials

The samples used in this experiment were taken from a typical incineration plant and a landfill in Chongqing, namely Fengsheng Incineration plant and Changshengqiao landfill leachate treatment system. The specific process flow and sampling point are shown in Fig. 1. The RO membrane component was cleaned once when the water inflow was 1 200 m<sup>3</sup> and 1 000 m<sup>3</sup>, respectively.

The influent, effluent and concentrated liquid of the DTRO system of the incineration plant were 100, 300, 500, 700, 900 and 1 100 m<sup>3</sup>. The influent, effluent and concentrated liquid produced by the first-stage RO system in the landfill were 100, 300, 500, 700 and 900 m<sup>3</sup>.

Dow RO membranes obtained in the treatment process after one cycle of DTRO system operation and before reverse cleaning were used for incineration plant and landfill.

The instruments are listed in Table 1.

### 1.2 Determination of RO Membrane Fouling Composition

3DEEM characterizes the fluorescence peaks of organic matter in leachate with different emission and ex-

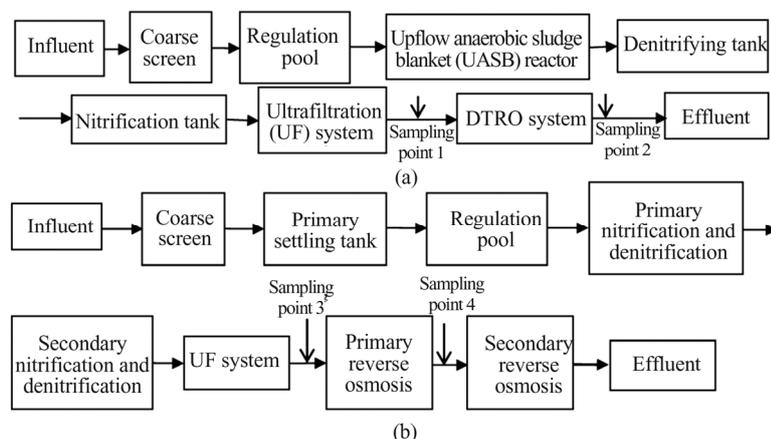


Fig. 1 Leachate treatment process and sampling points in incinerator (a) and landfill (b)

citation wavelengths, and provides complete spectral information, thus revealing the source and component structure of different organic pollutants<sup>[12-14]</sup>. However, as the fluorescence peaks are usually signals generated by multiple superimposed fluorescence groups, some of them may be unrecognizable or inaccurate. Fluorescence regional integration (FRI), another fluorescence spectrum analysis method, can identify and characterize the overlapping objects of fluorescence spectra in multi-

component systems, and overcome the above deficiencies to a certain extent<sup>[15]</sup>. Some studies<sup>[16-19]</sup> divide 3DEEM spectrum into five regions, as shown in Table 2. The fluorescence signal of region III is mainly generated by substances like fulvic acid in ultraviolet region, while region V is related to organics with higher molecular weight and aromatization degree such as fulvic acid, humic acid and polycyclic aromatic hydrocarbon in visible region.

**Table 1 Information and use of laboratory equipment**

Index	Instrument and type	Method	Note
3DEEM	3DEEM analyzer Hitachi F-7000	—	The scanning speed is 2 400 nm/min. The excitation wavelength $\lambda_{ex}$ was 200-600 nm, the emission wavelength $\lambda_{em}$ was 200-600 nm, and the step size was 5 nm. SigmaPlot software was used for data analysis
Grain diameter	Nano-particle analyzer Zetasizer Nano ZS90	—	Particle size measurement range from 2 to 3 000 nm, measurement angle of 90°
COD <sub>Cr</sub>	COD spectrophotometer HACH DR/1010 COD digestion device HACH DRB 200	Potassium dichromate method	The determination of COD <sub>Cr</sub> was divided into two ranges of 0-150 mg/L and 0-1 500 mg/L according to the concentration

**Table 2 Zoning of 3DEEM**

Region	Organic type	$\lambda_{ex}$ / nm	$\lambda_{em}$ / nm
I	Tyrosine protein	220-250	260-330
II	Tryptophan protein	220-250	330-380
III	Fulvic acids	220-250	380-550
IV	Soluble microbial metabolites	250-380	260-380
V	Humic acid humus	250-500	380-550

On the basis of unit fluorescence spectrum, each region divided by 3DEEM was calculated quantitatively<sup>[20]</sup> by FRI. The discrete integral formula is expressed as:

$$f_i = \sum_{ex} \sum_{em} I(\lambda_{ex} / \lambda_{em}) \Delta\lambda_{ex} \Delta\lambda_{em} \quad (1)$$

where  $f_i$  is integral value,  $\Delta\lambda_{ex}$  excitation wavelength interval (take 5 nm),  $\Delta\lambda_{em}$  emission wavelength interval (take 5 nm), and  $I(\lambda_{ex} \lambda_{em})$  fluorescence intensity (FI) at each point.

### 1.3 Determination of RO Membrane Fouling Formation Process

The indicators in line with the Pollution Control Standard of Domestic Waste Landfill (GBL6889-2008) were used for monitoring in this experiment. The high content of COD in landfill leachate represents a large amount of organic matter and other pollutants in the leachate, and is a potential contaminant of RO membrane,

which is clearly required by the discharge standard. Therefore, COD concentration is one of the important detection indexes for RO membrane pollution process analysis.

#### 1.3.1 Conservation of materials

According to the conservation of material, the content of membrane fouling can be calculated by that of inlet water, outlet water and concentrated liquid.

$$n_{TI} = \sum_{i=1}^k C_{i(IN)} V_{i(IN)} \quad (2)$$

$$n_{TE} = \sum_{i=1}^k C_{i(EF)} V_{i(EF)} \quad (3)$$

$$n_{TC} = \sum_{i=1}^k C_{i(CL)} V_{i(CL)} (1 - \eta) \quad (4)$$

$$n_{TD} = C_{i(DI)} V_{i(DI)} \times d \times c \quad (5)$$

where  $C$ ,  $V$ ,  $n$ ,  $d$ , and  $c$  represent the concentration, volume, amount of substance, number of membranes and number of membrane columns, respectively. “TI” and “IN” represent the total influent and influent, “TE” and “EF” represent the total effluent and effluent, “TC” and “CL” represent the total concentrated liquid and concentrated liquid, “TD” and “DI” represent the total dirt and dirt. The coefficient  $\lambda$  represents the ratio of material output to input of the DTRO system:

$$\lambda = (n_{TE} + n_{TC} + n_{TD}) / n_{TI} \times 100\% \quad (6)$$

It is assumed that the water quality of 200 m<sup>3</sup> leachate near each water sample remained unchanged, and the water production rate  $\eta$  of DTRO system in incineration plant and landfill was 75%. At the end of DTRO operation, three RO films with fouling were taken, and the fouling was dissolved in 100 mL ultra-pure water, respectively. COD of the fouling solution was then measured, and the average value was taken. The coefficient  $\lambda$  corresponding to COD was obtained from formulas (2) to (6) to calculate the conservation of materials.

### 1.3.2 Linear relationship between FI and fulvic acid

According to Shi's study<sup>[21]</sup>, the concentration of organic compounds can be calculated by the FI of each component of leachate. In addition, Liu and Wu<sup>[22]</sup> indicated that the concentration of fulvic acid in water samples can be accurately reflected by FI analysis of 3DEEM spectrum. In order to quantitatively analyze the total amount of fulvic acid in leachate, this study set a voltage to be 700 V and used FI under  $\lambda_{ex} / \lambda_{em} = 320 \text{ nm} / 400 \text{ nm}$  as 3DEEM line of fulvic acid<sup>[23]</sup>. The content of fulvic acid in water sample was calculated by measuring peak height. According to the standard curve of fulvic acid and the conservation of materials, the content of fulvic acid in membrane scale was calculated, and the amount of scale formation and the total amount of accumulated scale formation of leachate influent per 200 m<sup>3</sup> in the DTRO system of incineration plant and landfill was further obtained.

## 2 Results and Discussion

### 2.1 Component Analysis of Membrane Fouling

As can be seen from Fig. 2, seven fluorescence characteristic peaks appeared in the water sample during the experiment, which were visible fulvic acid fluorescence peak (peak A), UV fulvic acid fluorescence peak (peak B), aromatic protein (peak C), highly excited tryptophan protein (peak D), highly excited tyrosine protein (peak E), low-excitation tyrosine protein (peak F), and

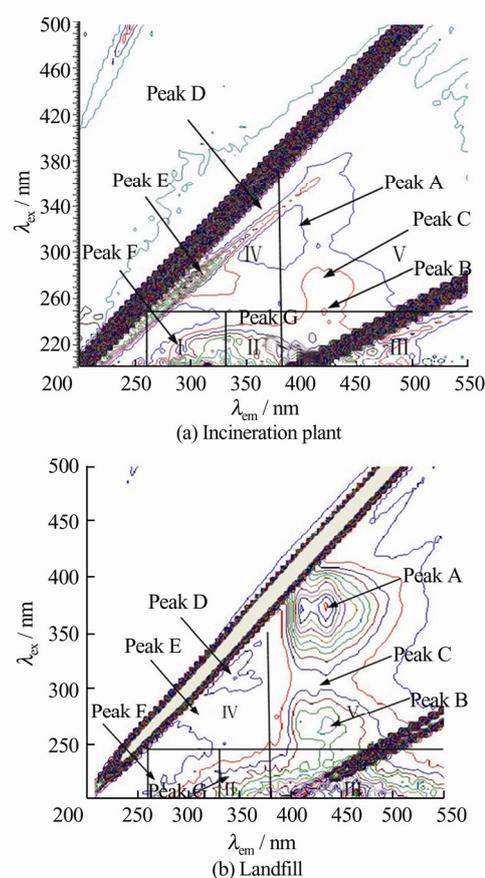


Fig. 2 3DEEM of RO membrane fouling

low-excitation tryptophan protein (peak G). The 3DEEM isolines of RO fouling solution in the incineration plant and landfill were sparsely distributed, indicating the relatively small content of organic matter in fouling. Combined with Fig. 3, it can be seen that no matter in incineration plant or landfill, the proportion of organic matter in zones III and V was the largest, which was 51.52% and 33.25% for incineration plant, and 34.13% and 55.26% for landfill, respectively. The main pollutants in zones III and V included UV fulvic acid and

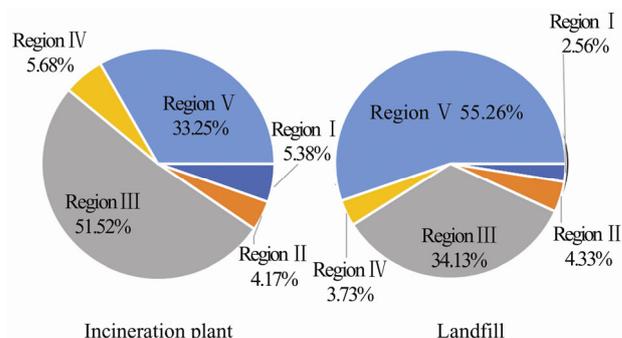


Fig. 3 Proportion of RO membrane fouling in organic matter integral value of each region on 3DEEM

visible fulvic acid respectively. Therefore, the main pollutants in RO membrane fouling could be UV fulvic acid and visible fulvic acid, which is consistent with the research results obtained by Han<sup>[24]</sup>. UV fulvic acid of incineration plant and UV fulvic acid of landfill accounted for more than half of their respective organic pollutants.

According to Table 3, the peak value of peak B (75.9) was significantly higher than that of peak A (51.8), indicating that the UV fulvic acid with simple structure and small molecular weight possibly blocked the pores on the membrane surface and thus these pores gradually accumulated and deposited on the membrane surface, resulting in poor membrane permeability and membrane pollution. This is consistent with Meng's research<sup>[25]</sup> statement. Peaks D, F and G were prominent, and the

fluorescence peaks were higher than peaks A and B, indicating that the membrane fouling respectively contained more protein substances and more high-excitation tryptophan with low-excitation tyrosine and low-excitation tryptophan. The fluorescence peak of peak E was as high as 213.2, which is clearly seen on the spectrum, indicating that the highly excited tyrosine protein may also be one of the main contaminants of the membrane. According to the study by Li *et al*<sup>[26]</sup>, protein-like substances have an important effect on membrane fouling. The higher the concentration of protein-like substances, the greater the possibility of membrane fouling. It contains more protein and humic acid substances. Membrane fouling is more likely to occur, which is consistent with the above research conclusions.

**Table 3 Peak position and intensity of fluorescent substances in RO membrane fouling**

Peak	Fluorescent component	Incineration plant		Landfill	
		$\lambda_{ex} / \lambda_{em}$ (nm/nm)	Peak intensity	$\lambda_{ex} / \lambda_{em}$ (nm/nm)	Peak intensity
A	Visible fulvic acid	360 /410	51.8	375/435	615.5
B	UV fulvic acid	225/410	75.9	265/435	323.8
C	Aromatic protein	280/395	29.2	300/420	127.7
D	Highly excited tryptophan protein	300/335	96.5	315/350	54.6
E	Highly excited tyrosine protein	275/300	213.2	280/310	92.2
F	Low-excitation tyrosine protein	215/290	85.4	215/275	74.2
G	Low-excitation tryptophan protein	225/365	96.2	220/335	205.5

As can be concluded from the landfill data, both peak A and peak B had a strong response. The peak A (651.5) was significantly higher than the peak B, possibly due to the fact that the accumulated UV fulvic acid resulted in film pollution, and that the molecular weight was relatively large. Visible fulvic acid was retained on the RO membrane. Peaks D, E and F were significantly lower than peaks A and B, indicating that the lower content of high-excitation tryptophan, high-excitation tyrosine and low-excitation tyrosine in the dirt. The peak value of peak G was as high as 205.5, and there was a strong response, indicating that low-excitation tryptophan-like proteins may be one of the main pollutants of RO membranes.

According to Zhang *et al*<sup>[27]</sup>, tyrosine-based proteins and tryptophan-based proteins are membrane contaminants that are difficult to remove. Therefore, combined with the above analysis, the organic matter difficult to remove in the RO membrane treatment system of incineration plants and landfills may be highly excited tyrosine protein and low-excitation tryptophan protein, respectively.

According to the study by Zhang *et al*<sup>[27]</sup>, FRI can reflect the relative content of organic matter represented by each region. Cui *et al*<sup>[28]</sup> showed that  $r(B/A)$ , the ratio of fluorescence intensity between peak B and peak A, is an index related to the maturity and structure of organic matter, and can be used to represent the degree of humification of dissolved organic matter (DOM) in leachate. The higher the  $r(B/A)$  value, the lower the degree of humification of leachate. The  $r(G/E)$  value indicates the composition and degradation of proteins in leachate. The higher the  $r(G/E)$  value, the more complex the proteins in leachate, the worse the biodegradability. The  $r(G/B)$  value represents the proportion of protein substances in leachate. The higher the  $r(G/B)$  value, the larger the proportion of protein in leachate.

As can be seen from Table 4, DOM humification in RO membrane fouling in landfill was greater than that in incineration plant, and protein composition was more complex than that in incineration plant. However, the proportion of DOM protein in RO membrane fouling in

landfill was not as high as that in incinerator, which indicated that in the process of RO, proteins in the membrane fouling of the incineration plant continued to accumulate, becoming the main pollutant of membrane fouling, which is consistent with the analysis result of fluorescence integral.

**Table 4 FI ratio of DOM in RO membrane fouling**

Item	Incineration plant	Landfill
$r(B/A)$	1.47	0.53
$r(G/E)$	0.45	2.23
$r(G/B)$	1.27	0.63

According to Baun *et al*<sup>[29]</sup> and Lou *et al*<sup>[30]</sup>, the pollutants in leachate can be divided into three parts according to particle size by membrane technology, namely, soluble substances and fine colloid (<450 nm), coarse colloid (450-1 200 nm), and suspended matter (>1 200 nm). The organic matter in leachate can be divided into

alcohols, organic acids and fulvic acid of small molecular weight, as well as fulvic acid and protein high molecular weight<sup>[31-33]</sup>.

As can be seen from Table 5, the particle sizes of pollutants in RO membrane fouling were mostly in the range of 450-1 200 nm, and the proportions of the incineration plant and landfill were 71.2% and 89.5%, respectively. This area was dominated by fulvic acid with small molecular weight. The above analysis results show that fulvic acid is the main pollutant in membrane fouling. Therefore, the main pollutant of membrane fouling is small-molecule fulvic acid with particle size ranging from 450 nm to 1 200 nm. According to Zhang's research<sup>[34]</sup>, the fulvic acid with small molecular weight is mainly distributed in the gel body, making it difficult to penetrate the membrane, thus resulting in the continuous concentration of concentrated liquid and the increase of particle size. In addition, according to Wang *et al*<sup>[35]</sup>, fulvic acid with small molecular weight is the main cause of RO membrane pollution.

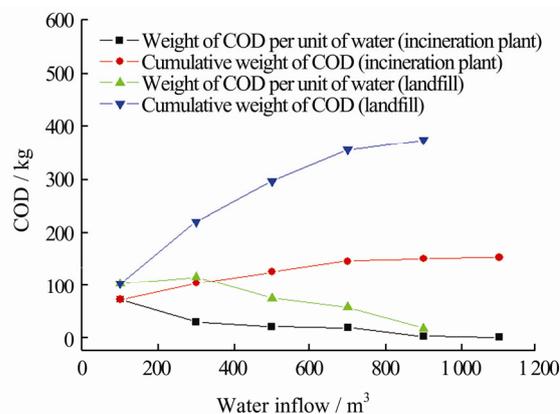
**Table 5 Distribution of particle size of membrane fouling**

Source	Pollutant size/nm			Particle size distribution/%		
	Average	Range	Peak	<450 nm	450-1 200 nm	>1 200 nm
Incineration plant	609.0	141.8-1 281.0	692.4	25.9	71.2	2.9
Landfill	745.2	91.3-1 281.0	913.6	5.0	89.5	5.5

## 2.2 Accumulation Rule of Membrane Fouling and Fulvic Acid

The COD coefficient  $\lambda$  of the incineration plant and the landfill DTRO system was calculated to be close to 100%, so the COD in the membrane fouling can be calculated by material conservation. Combined with Fig. 4 and Fig. 5, it can be seen that the scaling law of RO membrane in incineration plant and landfill was similar. With the operation of DTRO system, the amount of COD and fulvic acid in membrane fouling per unit of water inflow exhibited a downward trend, indicating the large amount of scale per unit in the first half of the operation cycle (the first half of the water inflow stage) possibly because of the smooth membrane surface in the first half of operation and the high likelihood of pollutants to adsorb and deposit on the membrane surface<sup>[36]</sup>. In the later stage of operation, the membrane surface was rough, and the amount of pollutants and pressure increases, which made it quite difficult for these pollutants to adhere to

the membrane surface. In the same operation cycle, COD and fulvic acid per unit of influent water in the fouling of RO membrane in landfill were significantly higher than those in incinerator, indicating that the RO membrane in landfill was easier to scale than that in incinerator.



**Fig. 4 COD of membrane fouling in one operation cycle of DTRO system**

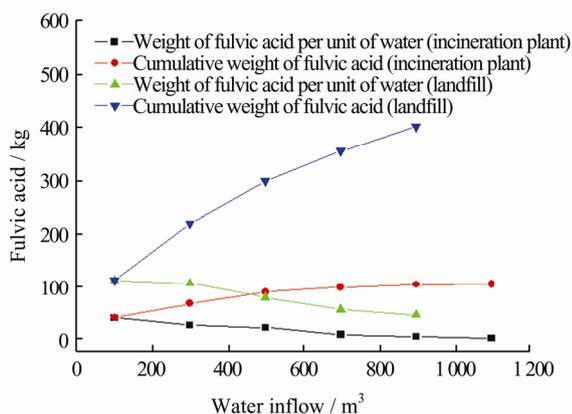


Fig. 5 Change of fulvic acid in membrane fouling during one operation cycle of DTRO system

### 3 Conclusion

The main pollutants in RO membrane fouling of the incineration plant and landfill are UV fulvic acid and visible fulvic acid with particle size ranging from 450 nm to 1 200 nm, and the pollutants that are difficult to remove include highly excited tyrosine protein and low-excitation tryptophan protein, respectively.

The membrane pollution of RO membrane in incineration plant and landfill mainly occurs in the first half of system operation. With the operation of DTRO system, the amount of COD and fulvic acid in membrane fouling per unit influent shows a downward trend. In the same operation period, the RO membrane in landfill is more likely to scale than that in incineration plant.

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