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Degradation of Leachate Biochemical Effluents Using Heat Activated Persulfacte Processes: Parameters Optimization and Removal Characteristics of Contaminants

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ABSTRACT

Using heat to activate persulfacte for the generation of sulfate radicals (SO_4^{-}) was proposed for the treatment of leachate biochemical effluents. The effects of different variables on the oxidation efficiency, including oxidant dosage, temperature, pH value and reaction time, were investigated. The experimental results showed that the contaminants in the leachate biochemical effluents can be efficiently removed using the heat/$Na_2S_2O_8$ system. Using the single factor variable method, the optimal conditions included 4 g/L oxidant dosage, 60°C reaction temperature, pH 4 and 12 h reaction time, resulting in the COD and colour removal efficiency of 63% and 100%, respectively. The analyses of three-dimensional excitation-emission matrix fluorescence spectrum (3DEEMFS) and ultraviolet-visible spectra (UV-vis), both illuminated that the macromolecular HA in the wastewater samples were degraded into smaller molecular fulvic acids (FA) via heat/$Na_2S_2O_8$ system. All these encouraged results demonstrated that the persulfacte activation used heat is a promising and efficient technology for the treatment of leachate biochemical effluents.

INTRODUCTION

Increasing volume of municipal solid waste (MSW) can be ascribed to the drastic increase in urbanization and the growth of living standard, and the predicted amount of solid waste in China alone would be 220 million tons (Zhao et al. 2016). Incineration is an efficient technology for MSW disposal, which can reduce waste and achieve the purpose of power generation by reclamation of wastes and incineration. However, for the lack of knowledge in accurately sorting the household refuse, it is a big challenge for the further development of waste incineration in China (Fu et al. 2015). Nowadays, sanitary landfill is still the most prevalent method for the disposal of MSW in China (Han et al. 2016). However, sanitary landfill would bring a serious pollution to water, namely landfill leachate. It is generated from the combination of the decomposition of organic wastes and the rainfall which flowed and percolated through the waste material layers in municipal landfills (Wang et al. 2016, Yang et al. 2015).

The composition of landfill leachate is complicated, which contains a large amount of organic pollutants (such as humic acids, HA and fulvic acids, FA), ammonia nitrogen (NH_4^+-N), heavy metals and some other inorganic salts (Martins et al. 2017, Yang et al. 2015, Zhang et al. 2017). The characteristics of leachate are varied due to the impacts of landfill age, depth and the local climate or season (Moody & Townsend 2016, Oloibiri et al. 2015). Among these, the age of landfill may be the most influencing factors. The leachate can be classified into young (typically < 5yrs) or mature (typically > 10yrs) leachates according to the landfill age (Amor et al. 2015). Usually, the young leachate, which is more biodegradable and can act as carbon source for microorganisms to biological degradation processes, is characterized by a higher concentration of organic matters, and a higher value of 5-day biochemical oxygen to chemical oxygen demand (BOD_5/COD is from 0.4-0.6) (Wu et al. 2016, Zhang et al. 2017). While the BOD_5/COD of mature leachate is lower to less than 0.2, the biodegradability of it is so awful that it is unsuitable to biological treatment. If discharged to natural water bodies directly, instead of properly collecting and safely treating, it will be a great threat to the surface and ground water due to the contaminants contained in the leachate.

Traditionally, biological processes, classified as aerobic, anaerobic and anoxic, are cost effective technologies

for the removal of biodegradable compounds (Wang et al. 2014). However, biological processes alone are not enough for the treatment of leachate with complex components, especially non-biodegradable compounds, hence the effluents need to be further treated. Advanced oxidation processes (AOPs) are particularly applicable in respect to the mature leachate treatment, with which a wide range of compounds from mature landfill leachate can be effectively degraded (Silva et al. 2017). Given the advantages of the AOPs that no massive hazardous sludge was produced, they were deemed to be "environmental-friendly" technologies. Nowadays, AOPs have been widely applied in the field of treatment of wastewater containing different organic compounds that are non-biodegradable and/or toxic to microorganisms (Mahvi et al. 2012). The typical AOPs such as ozonation (O_3) (Cortez et al. 2010), photocatalysis (TiO_2/UV) (Hassan et al. 2016, Jia et al. 2011), Fenton (H_2O_2/Fe^{2+}) (Deng & Englehardt 2006, Hui et al. 2009), and photolysis H_2O_2 (UV/H_2O_2) (Chelmeayala et al. 2010, Vilhunen et al. 2010), whose main mechanisms are all based on the strong oxidation performance of hydroxyl radicals ($\cdot OH$), have drawn a broad interests of researchers. The organic matters in the effluent can achieve its purpose of oxidative decomposition via $\cdot OH$. This free radical can initiate a series of reactions and subsequently oxidize or even mineralize the pollutants into CO_2 and H_2O (Hou et al. 2016).

Recently, owing to the low cost but high oxidation efficiency of sulfate radical ($SO_4^-\cdot$), the $SO_4^-\cdot$ oxidation as a promising method has absorbed increasing attention of researchers in the treatment of recalcitrant wastewater. Moreover, the $SO_4^-\cdot$, whose redox potential is about 2.5~3.1 V (related to the pH of solutions) compared to that of $\cdot OH$ (1.8~2.7 V), has a longer half-life and oxidation capacity to ensure adequate contact time with contaminants and further degrade them via oxidation (Shukla et al. 2011, Yang et al. 2010). Generally, the persulfacte is the main source of $SO_4^-\cdot$ in the laboratory or practical engineering. Similar to H_2O_2, there is also a -O-O- bond in the structure of persulfacte, which can be initiated into a free radical with high oxidation capacity.

Normally, methods, such as heat, UV light, granular activated carbon (GAC), and transition metal ions, are used to initiate $SO_4^-\cdot$ from persulfacte (Liu et al. 2012, Shih et al. 2012, Yang et al. 2010, Yang et al. 2011). Ahmed (Ahmed et al. 2012) evaluated a sulfate radical based technology (Co(II)/persulfacte system) for the treatment of diclofenac and sulfamethoxazole in the pharmacy wastewater. The promising results indicated that the $SO_4^-\cdot$ oxidation had an efficient effect on the removal of these particular pharmaceuticals over $\cdot OH$ oxidation for its higher selectivity. Although the oxidation of $SO_4^-\cdot$ has been employed largely as an alternative AOPs in the treatment of industrial or pharmacy wastewater, it is rarely reported in landfill leachate treatment.

In this study, heat was evaluated to activate persulfate for the generation of $SO_4^-\cdot$ (eq. 1) to treat landfill leachate biochemical effluent.

$$S_2O_8^{2-} + h\nu \rightarrow 2\ SO_4^-\cdot \qquad \qquad ...(1)$$

Experimental parameters were optimized and determined. In addition, the characteristics and chemical composition of wastewater samples were analyzed using three-dimensional excitation-emission matrix fluorescence spectrum (3DEEMFS) and ultraviolet-visible spectra (UV-vis).

MATERIALS AND METHODS

Landfill leachate: The leachate biochemical effluents employed in this study were taken from the Maiyuan sanitary landfill, which is located in Nanchang, a city enjoyed with the prestige of the "hero city" in South China. The landfill which totally covered 10^7 ha, was established in 1997, and the capacity of it was designed to about 18,000,000 m^3. The daily processing capacity in municipal solid waste of this landfill has reached more than 2000 t nowadays. During each sampling, 60 L of landfill leachate was collected in the polyethylene bottles pre-treated by acid, and then transported to the laboratory and stored in a refrigerator at 4°C. The landfill leachate was brown with a pungent smell. Typical characteristics of the landfill leachate are given in Table 1.

Chemicals: Sodium persulfate ($Na_2S_2O_8$, 98% purity) was obtained from the Sinopharm Chemical Reagent Co., Ltd., China. Sodium hydroxide (NaOH, 99% purity) was purchased from Jiangxi Shilong Industrial Co., Ltd., China. Dilute hydrochloric acid (HCl, 31% purity) was bought from Jiangxi Ganzhou Guosheng Railway Industry Co., Ltd., China. All reagents used were analytical grade and distilled water was applied for preparing all aqueous solutions.

Experimental procedures: Heat-activated experiments were carried out in a water bath oscillator (model SHA-C, China) at room temperature (25°C). Briefly, 100 mL of leachate biochemical effluent was taken out and placed in a 250 mL conical flask. Then the initial pH of leachate was adjusted using the pre-prepared solutions of 25% NaOH and 10% HCl. Afterwards, sodium persulfate as an oxidant, appropriate amounts of it were dosed into the leachate. The mixed

Table 1: Characteristics of landfill leachate in the experiment.

Parameter	Values
pH	6-7
COD	1000-1400 mg/L
Ammonia nitrogen	300-400 mg/L
Colour	600-700 times

solutions were placed in the water bath oscillator at different temperature for oscillation at the speed 160 r/min. After oscillation for hours, the supernatant were collected for centrifugation at the speed 3000 r/min for 15 min. Without any agitation and settled for a certain time, the supernatant was withdrawn after centrifugation and was used for chemical analysis. The pH values were measured by a pH meter (model PHS-3C, China). Chemical oxygen demand (COD) was gauged by microwave-assisted closed digestion apparatus (model WXJ-III, China). Dilution factor method was selected to determine the colour of wastewater samples. Each experiment was conducted in duplicate, and the results reported in this paper were the arithmetic average of the two tests.

Analytical Methods

3DEEMFS: The leachate samples before and after treatment were diluted 10 times, and subsequently filtered with 0.45 mm filters (millipore) before being analyzed. A certain amount of wastewater samples was taken to wash quartz dish in 4~5 times prior to the steps of adding wastewater samples to be measured. The quartz dish with distilled water was set as a blank control group in the experiment. 3DEEMFS of wastewater samples before and after treatment were determined using a fluorescence spectrophotometer (Hitachi F-2000, Japan), of which the excitation source was a 150 W xenon lamp. A scan speed of 400 nm × min^{-1} was used with a slit width opening of 10 nm. The previous filtered wastewater samples were scanned in a 1 cm quartz cell at excitation wavelengths from 250 nm to 500 nm at 5.0 nm increments by varying the emission wavelengths from 250 nm to 600 nm at 5.0 nm increments. The data of 3DEEMFS analyses were handled using a windows-based software Origin 8.6. The colour images plotted according to the data collected from automatically analysed by the apparatus. The characteristics and chemical composition of wastewater samples before and after treatment were represented significantly on the images (Zhu et al. 2012).

UV-vis: The ultraviolet spectrophotometer (model UV-2450, China) was used to record the UV-vis absorption spectra of wastewater samples. Adjusting the pH to 7, the wastewater samples were all scanned under the scanning interval at 1 cm over the wavelength range of 190 nm-500 nm at the moderate scanning speed.

RESULTS AND DISCUSSION

Oxidation Performances of Landfill Leachate Biochemical Effluent Using $SO_4^-\cdot$ Oxidation Under Variable Factors

Oxidant dosage: It is oxidant dosage that is the most crucial factor for the removal of persistent contaminants in the landfill leachate biochemical effluent. Since the $Na_2S_2O_8$ is the direct source of $SO_4^-\cdot$, the oxidation performance is significantly enhanced with the increase of $Na_2S_2O_8$ dosage. The effect of $Na_2S_2O_8$ dosage on the efficiencies of COD and colour removal was evaluated under pH 3 and 12 h reaction time at room temperature. The removal curve of COD and colour is shown in Fig. 1.

Observing the removal curve of COD and colour, the oxidation performance had a slight rise with the increasing oxidant dosage in the experimental range. When the dosage was 1 g/L, the removal rates of COD and colour were 5% and 45%, respectively. It was obvious that when using $SO_4^-\cdot$ initiated from heat/$Na_2S_2O_8$ system for the removal of COD and colour, the effect of colour removal was superior to that of the COD. The reason was that the coloured substances of contaminants in the wastewater samples were reacted with $SO_4^-\cdot$ in priority. Continuing to increase the oxidant dosage to 4 g/L, the removal rate curve of them had a slow rise, and the removal rates of COD and colour reached 15% and 58% respectively. Although the removal rates of them would continue to increase when more oxidants were added, the oxidation effects would not change by a wide margin. Furthermore, taking the costs of reagents into consideration in the practical application, it is unwise to employ large amounts of oxidant dosage for the oxidation experiments. Thus, in case of fluctuation of water quality, when the COD of effluents was 1000-1800 mg/L, 4 g/L oxidant dosage was selected as an optimal dosage.

Temperature: Temperature is another important factor for the oxidation. In general, the higher the temperature, the better the oxidation performance is. The reason is that an appropriate rise of temperature will accelerate the migration rate of free radicals and contaminants. However, when the migration rate reaches its maximum at a certain temperature, the removal rates of contaminants would not increase any more with temperature rise. The results in this section were all obtained at initial pH 4, 4 g/L $Na_2S_2O_8$ dosage and 12 h reaction time. The effect of different temperatures on the COD and colour removal is shown in Fig. 2.

As shown in Fig. 2, when the $Na_2S_2O_8$ dosage was 4 g/L at room temperature, the removal rates of COD and colour were only 20% and 55%, respectively. Keeping the $Na_2S_2O_8$ dosage constant, when the temperature rose to 45°C, the removal rates of them increased by 14% and 35%, respectively. Comparing with the results, 60°C was the optimum oxidation temperature in this experiment, at which the removal rates of COD and colour both reached their maximum, 63% and 100% respectively. Observing the COD removal curve, its removal trend tended to be a slight decreasing when the temperature was over 60°C. The reason was that the content of $SO_4^-\cdot$ which generated from the broken -
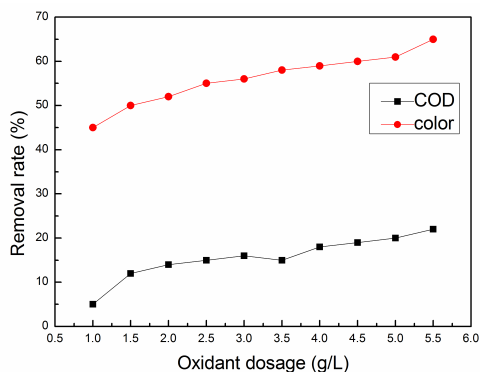
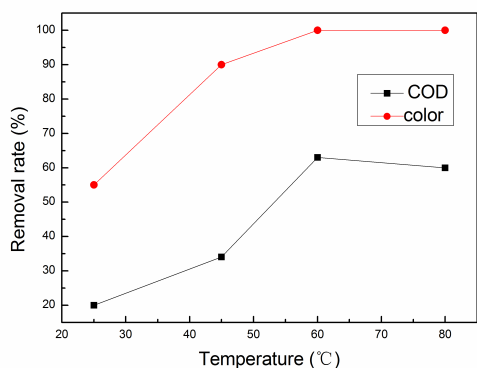Fig. 1: The oxidation efficiency at different oxidant dosages.

Fig. 3: The oxidation efficiency at different pH values.

Fig. 2: The oxidation efficiency at different temperatures.

Fig. 4: The oxidation efficiency at different reaction times.

O-O- bond in the $S_2O_8^{2-}$ at a lower temperature was not adequate, resulting in the unfavourable oxidation performance. However, at a higher temperature, excessive $SO_4^-\cdot$ would be initiated by heat, which caused the decrease of COD removal rate, potentially due to the scavenging reactions between the generated sulfate radicals themselves. Moreover, one thing needed to be a complement explanation that the heat/$Na_2S_2O_8$ system had a significant efficiency for the colour removal. Under the conditions at initial pH 4, 2 g/L $Na_2S_2O_8$ dosage, the effluent has been completely transparent after oxidation for 12 h at 60°C, at which the colour removal was up to 100%.

pH: Based on the technology of $SO_4^-\cdot$ oxidation for the contaminants degradation in the landfill leachate biochemical effluent, pH value is a significant parameter. Fig. 3 shows the effect of different pH on the removal of COD and colour.

Observing the curves of COD and colour removal, the removal rates of COD and colour, both increased at the initial stage and then subsequently decreased. In addition, comparing the two removal curves, it can be seen that the pH had a more significant effect on the COD removal than that of colour. The change rangeability of COD removal
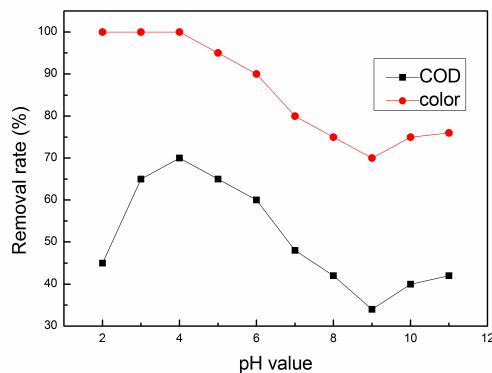
reached 35%, which indicated that pH value played a remarkable role in the generation of $SO_4^-\cdot$ from using heat to activate $Na_2S_2O_8$. The COD removal was only 45% at pH 2, indicating that lower pH would have an effect on the degradation of persistent contaminants in leachate. The reason can be ascribed to two aspects: one was that when the wastewater was under acid condition, the massive H^+ in it would lead to the activity loss of activated persulfate by the Fe^{2+} in the existence form of $[Fe(H_2O)_6]^{3+}$, $[Fe(H_2O)_5]^{2+}$ and $[Fe(H_2O)_6]^{2+}$; another one was that excessive $SO_4^-\cdot$ were activated from $S_2O_8^{2-}$ at a lower pH, and the scavenging reactions took place between the generated sulfate radicals themselves and further resulting in the great reduction of persulfate utilization (Liang et al. 2008). When the pH increased to 4, the COD removal reached 70%. However, the COD removal tended to decrease when the pH was over 4, of which the maximum decreasing amplitude was 36% than that at pH 9 (34%). In contrast, the colour removal was as high as 100% at the pH range of 2-4. But the colour removal also performed notable decrease with pH sostenuto increasing, which was the same as the COD removal did. From the results concluded above, the oxidation system can also deteriorate at a higher pH. The probable reasons were described as follows: First, hydroxy radical (·OH)
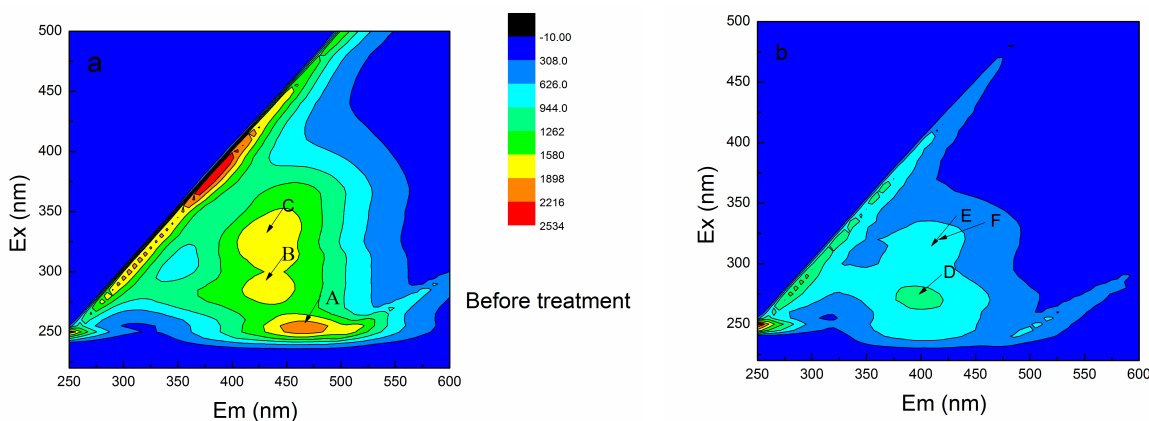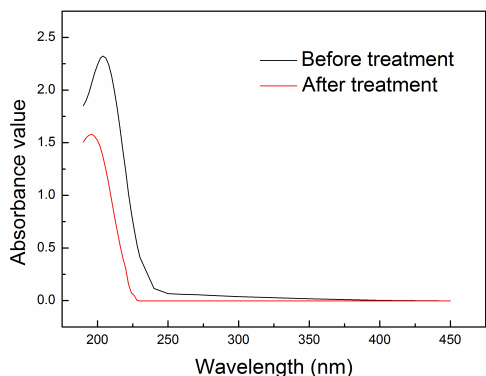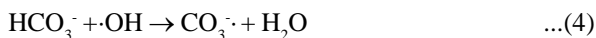
Fig. 5: 3DEEMFS of samples before and after treatment.

Fig. 6: The absorbance in different wavelengths.

can be initiated through the reactions between $SO_4^-\cdot$ and OH^- at alkalescence (eq. 2).

$$SO_4^-\cdot + OH^- \rightarrow SO_4^{2-} + \cdot OH \qquad ...(2)$$

However, the oxidation-reduction potential of $\cdot OH$ was lower than that of $SO_4^-\cdot$ at higher pH, which resulted in the oxidation ability to decline when using $\cdot OH$ for the oxidation of organic compounds (Wang et al. 2017). Second, higher alkalinity of wastewater caused by the increasing pH value, would accelerate side reactions of $\cdot OH$ with CO_3^{2-} and HCO_3^- (eqs. 3 and 4), and further reduce the oxidation efficiency.

$$CO_3^{2-} + \cdot OH \rightarrow CO_3^-\cdot + OH^- \qquad ...(3)$$

$$HCO_3^- + \cdot OH \rightarrow CO_3^-\cdot + H_2O \qquad ...(4)$$

In summary, higher pH value was unsuitable for the removal of contaminants in leachate biochemical effluent. Moreover, the similar conclusion had been drawn out by Shen (Li et al. 2009) in his study of using heat to activate persulfates for the degradation of dichlorophenones.

Reaction time: The reaction time of the experiment on the removal of COD and colour was controlled in the method of single factor variable. Namely, adjusting the initial pH of the wastewater sample at 4, the results of this section were carried out when the $Na_2S_2O_8$ dosage was 4 g/L at 60°C. The effect of reaction time on the removal of COD and colour is shown in Fig. 4

It can be seen from the COD and colour removal curves that the removal of both tended to rise quickly at the initial stage and then subsequently tended to be stable with the reaction time prolonging. When the reaction time was 2 h, the COD and colour removals were 45% and 60%, respectively. Prolonging the reaction time to 12 h, the COD removal reached 63%, while the colour achieved complete removal. However, the removal rates of COD and colour would not almost change any more when the reaction time was over 12 h, indicating that the reaction of $SO_4^-\cdot$ oxidation for the organic degradation in effluents was basically finished. Two reasons can be used to elucidate the trend of COD and colour removal curves. First, large amounts of $SO_4^-\cdot$ would generate with the adding of $Na_2S_2O_8$ at the beginning of the reaction, which made the COD and colour removals quick. After a long time reaction (12 h), the concentration of $SO_4^-\cdot$ in the solutions was at a low level due to the consumption by the previous organics, indicating the removals of COD and colour would not markedly elevate any more. Second, owing to the complex components of landfill leachate, the dissolved organic matters (DOM) can be classified into two kinds: easy degradation and non-degradation. The removals of COD and colour declined obviously due to the oxidation of massive easy-degradation organics at the beginning of the reaction. Nevertheless, the ratio of non-degradation organics dominated in the solution at the later period of the reaction. Consequently, the increment speed of COD and colour removals were decreased, resulting in the removal rates of COD and colour not rising any more.

Table 2: Fluorescence spectra parameters of wastewater samples.

Peaks	E_x/E_m	Intensity	Composition
A	255/465	2154	HA
B	285/430	1684	visible FA and HA
C	330/430	1832	visible FA
D	275/400	1034	FA
E	315/410	765	visible FA and HA
F	320/415	767	visible FA

Spectra Analyses of Wastewater Samples

3DEEMFS of wastewater samples: 3DEEMFS was used to illuminate the dissolved organic matter (DOM, containing protein-like or humic substance-like) according to the 3D-matrix fluorescent fingerprints. The 3DEEMFS of wastewater samples before and after treatment are shown in Fig. 5. In order to avoid the internal filtration effect, the water samples were diluted 10 times, but the fluorescence signals were still very strong in many areas. Each 3DEEMFS was corresponding to special substances contained in the samples, such as HA (E_x/E_m=250~390 nm/370~480 nm), visible FA and HA (E_x/E_m=280~370 nm/380 nm~460 nm), visible fulvic acid (E_x/E_m=310~360 nm/370~450 nm), and fulvic acid (E_x/E_m=200~350 nm/350~460 nm). All the fluorescence spectra parameters of wastewater samples before and after the treatment of heat/$Na_2S_2O_8$ system, including peak location, fluorescence intensity and composition, are listed in Table 2.

On the basis of the analyses above, a large amount of HA was oxidized into FA with smaller molecular weight and simpler structure via $SO_4^-\cdot$ oxidation. Furthermore, the fluorescence intensity of FA degraded almost 55% after treatment. The result was in accordance with the removal efficiency of COD.

UV-vis of wastewater samples: To be well known, the UV-vis spectrum is one of the efficient means, which was widely used to study the components and structures of the fractions. The higher the absorbance in the ultraviolet region, the more complex structure and the higher aromaticity of the wastewater samples would be.

As shown in Fig. 6, the absorbance of leachate biochemical effluent only appeared in the ultraviolet spectrum range at 200-400 nm, while there was almost no absorbance in the visible region. The result indicated that the leachate biochemical effluent was characterized with unsaturated conjugated bond. In addition, after the oxidation of heat/$Na_2S_2O_8$ system, the absorbance of the treated wastewater sample was significantly lower than that of leachate biochemical effluent at the same wavelength. The declined trend

of the absorbance represented that the complex structure and aromaticity of molecules in wastewater samples were oxidized via $SO_4^-\cdot$ oxidation into smaller molecular weight substances or even mineralized into CO_2 and H_2O_2.

The absorbance values at 254 nm (E_{254}) and 280 nm (E_{280}) were used as indicators for the hydrophobicity and aromaticity of structure. The E_{254} and E_{280} values of treated effluents were both 0, while the values of leachate biochemical effluents were 0.067 and 0.056, respectively. This result indicated that the macromolecular organic matters with complex structure can be degraded via heat/$Na_2S_2O_8$ system. The kind of substituent group and degree of substitution can be reflected by E_{253}/E_{203} ratio. Generally, the higher E_{253}/E_{203} ratio means they are rich in a carbonyl group, a hydroxyl group, and a carboxyl group in the substituent, while the lower ratio means that the substituent group on the aromatic ring is mainly the aliphatic chain. After the treatment of heat/$Na_2S_2O_8$ system, the value of E_{253}/E_{203} degraded from 0.124 to a negative number. The result greatly indicated that the kind of substituent group and the degree of humification in the wastewater samples were both decreased. According to the previous literature, the E_{250}/E_{365} value was negatively related to the humification degree of water. The appearance of an interesting phenomenon that the E_{250}/E_{365} value after treatment was higher than that of leachate biochemical effluent, can be ascribed to the FA oxidized from macromolecular HA.

CONCLUSIONS

In this study, the heat activated persulfacte oxidation was proved to be an efficient method for the treatment of landfill leachate biochemical effluents from a sanitary landfill located in Nanchang, China. Controlling the other factors constant, the single variables on the removal efficiency of COD and colour were investigated separately. Taking all factors into comprehensive analysis, the optimal conditions included 4 g/L oxidant dosage, 60°C reaction temperature, pH and 12 h reaction time, which resulted in the COD and colour removal efficiency of 63% and 100%, respectively.

According to the analyses of 3DEEMFS and UV-vis, the

degree of molecular complexity and aromaticity decreased significantly in water samples. Moreover, a significant reduction of organic pollutants illuminated that the HA had been degraded into fulvic acid with smaller molecular weight.

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