Incineration Properties and Kinetic Studies of Sludge from Old Newsprint Fiber Line

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The basic incineration properties of flotation deinking sludge and two kinds of flocculating sludge from old newsprint fiber line were studied. Coats-Redfern was used for incineration process analysis, and based on the kinetic parameters of the three types of studied sludge at a heating rate of 10 °C·min^{-1}, the reaction orders were confirmed in the organic incineration region. The activation energy of the three sludge types were 38.78, 44.59, and 48.11 kJ·mol^{-1}, and their frequency factors were 2.58 E^{+07} min^{-1}, 3.19 E^{+06} min^{-1}, and 1.50 E^{+06} min^{-1}, respectively. Organics incineration in the flocculation sludge was more difficult than the incineration of the deinking sludge; as the flocculate dosage increased, the incineration difficulty increased. However, the amount of flocculating sludge generated from the flocculation treatment of the deinking white water accounted for less than 10% of the flotation deinking sludge, and their elemental content and calorific value related to combustion were also similar to each other; therefore, it could be predicted that the flocculating sludge would not greatly impact the sludge treatment system. Therefore, it could be incorporated into existing sludge incineration systems for reduction and thermal energy recovery.

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INTRODUCTION

Recovered paper is currently the most important raw material in the pulp and paper industry, which accounted for greater than 72% of the total pulp production in China in 2020 (China Paper Association 2021). However, a large amount of deinking sludge is generated during the deinked pulp manufacturing process. (Liu *et al.* 2015). Many studies have focused on the treatment and comprehensive utilization of oil-based ink sludge using deinking sludge to prepare activated carbon adsorption material that can effectively remove organic pollutants in wastewater and absorb heavy metals in soil (Paz-Ferreiro *et al.* 2017). Ouadi *et al.* (2013) used a medium-speed pyrolysis reactor to continuously pyrolysize dry deinking sludge particles to produce liquid fuel. Amare *et al.* (2019) used anaerobic digestion to pretreat deinking sludge, which not only reduced the organic matter content in the deinking sludge, but also obtained methane gas and recovered energy (Fan *et al.* 2018; Abdelghany *et al.* 2021). Elloumi *et al.* (2018) used deinking sludge as a filler or enhancer high-density polyethylene to prepare bio-composites, which can improve the mechanical properties such as the tensile strength and Young's modulus (Su *et al.* 2020; Wang *et al.* 2022). Vannucchi *et al.* (2021) used deinking sludge for soil modification, mixed with municipal green solid waste compost, pumice and zeolite as nutrients to support tree growth. Most of their research subjects were focused on floating ink section sludge, and a considerable portion of them were oil-based ink.

Kinetic studies are important for reaction mechanisms (Mahmoud and Mahmoud 2021a, Mahmoud *et al.* 2021b). Water-based ink, which has a low price, good printing suitability, and is non-toxic and environmentally friendly, is widely used in newsprint, magazine paper, and office paper printing. However, the factors in the recycling process of water-based ink printing paper, *e.g.*, small ink particles, the lack of hydrophobicity of the ink particles and the re-deposition of water-based ink on the fiber, leads to deinking difficulties (Dorris and Nguyen 1995; Chen 2007a,b; Zhang *et al.* 2009; Su *et al.* 2019). The deinking problem of water-based ink print paper was basically solved through numerous studies (Chabot *et al.* 1995; Ben *et al.* 2000). However, another problem arises when the white water that contained water-based ink is reused in paper mills to save water, *i.e.*, the accumulation of ink particles in the white water affects the whiteness of the paper.

The authors achieved good results in the treatment of water-based ink wastewater *via* flocculation technology, and the treated white water met the requirements for reuse (Su *et al.* 2014). In order to further improve the research results, the physicochemical property and incineration characteristics of the flocculation sludge from the white water containing water-based ink were studied and compared with de-inking sludge. In addition, the safety of the inorganic residues was further evaluated to provide theoretical support for the reuse of white water containing water-based ink and the treatment of its sludge.

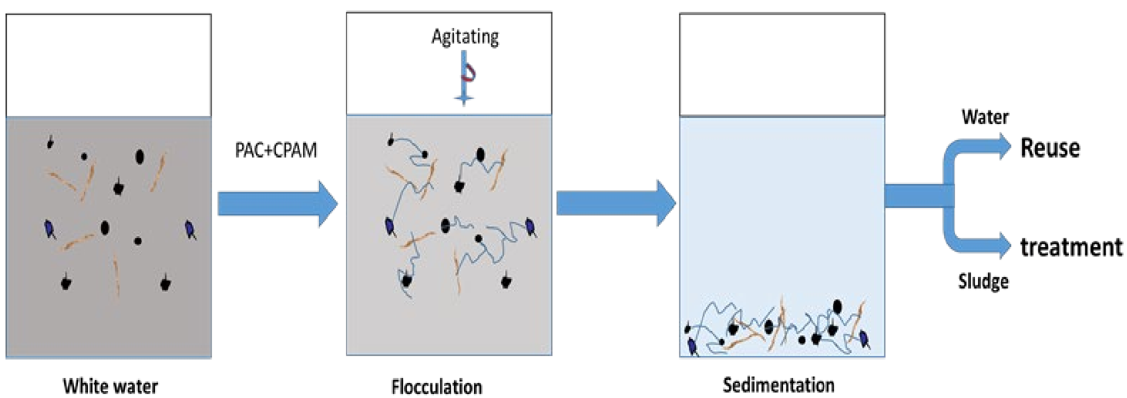

Fig. 1. Schematic diagram of white water treatment process

EXPERIMENTAL

Materials

The deinking sludge was taken from a paper mill using flexographic printed old newsprint (ONP) as the raw material. The two flocculation sludge samples with different flocculation dosages were obtained under the optimized conditions during the experiments of the water-based ink-containing white water treatment *via* flocculation technology (Fig. 1). The sludge samples were dried at 50 °C for 24 h, crushed, and stored a self-sealed bag.

Methods

Analytical equipment

The equipment used in this study included the following: a Vario EL cube Elementar elemental analyzer (Frankfurt, Germany); a TGA/DCS I STARe METTLER thermogravimetric analyzer (Zurich, Switzerland); a FP6410 flame photometry (Shanghai Lepad Scientific Instruments Co., Ltd, Shanghai, China); a ZDHW-5000 calorimeter (Hebi Huadian Analytical Instrument Co. Ltd. Hebi, China); an ICP-MS (Thermo Fisher Scientific Inc, Waltham, MA); and an EDXRF (Thermo Fisher Scientific Inc, Waltham, MA).

Analytical methods

The C, H, N, and S contents of the sample were determined *via* an elemental analyzer under the following conditions. The temperature of the oxidation furnace and reduction furnace were 1150 and 850 °C, respectively. The pressure of He and O_2 were 0.12 to 0.125 MPa and 0.22 MPa, respectively; the content of the other elements were determined *via* ICP-MS. The fiber content was determined by referring to Zhang (2011).

Thermal gravimetric analysis

Thermal gravimetric analysis (TGA) was carried out under atmosphere conditions in the furnace. The sludge was heated at a rate of 10 °C/min from 50 to 1000 °C. The relationships between the physicochemical properties of the sample and the temperature were measured. The sample weight was less than 5 mg to avoid the influences of heat transfer, secondary gas-solid reaction, and mass diffusion factor.

Based on the relevant data of TGA, the weight loss rate of the sludge at time *t* can be calculated according to Eq. 1,

$$\propto = \frac{W_0 - W_t}{W_0 - W_\infty} \tag{1}$$

where W_0 is the initial weight of the sludge (g), W_t is the weight of the sludge at time *t* (g), and W_∞ is the final weight of the sludge (g) (Coats and Redfern 1964; Zhang 1996; Wu *et al*. 2007).

The rate of disappearance of sludge can be expressed by Eq. 2,

$$\frac{d\propto}{dt} = kf(\propto) = k(1-\propto)^n \tag{2}$$

where α is the fraction of sludge decomposed at time *t*; *n* is the order of the reaction, and *k* is the rate constant given by the expression of action.

When the Arrhenius equation, as shown in Eq. 3,

$$k=Ae^{-E/RT} \tag{3}$$

was combined with Eq. 2, it yielded Eq. 4,

$$\frac{d\propto}{dt} = Ae^{-E/RT}f(\propto) = Ae^{-E/RT}(1-\propto)^n \tag{4}$$

where *R* is the gas constant; *A* is the frequency factor, *E* is activation energy of the reaction, and *T* is the absolute temperature.

A linear heating rate, *e.g.*, °·min^{-1}, can be shown by Eq. 5,

$$\beta = \frac{dT}{dt} \tag{5}$$

Combining Eq. 4 and Eq. 5 yielded Eq. 6,

$$\frac{d\propto}{dT} = \frac{A}{\beta} e^{-E/RT} f(\propto) = \frac{A}{\beta} e^{-E/RT} (1- \propto)^n \tag{6}$$

After rearranging, integrating, and taking ln, Eq. 6, became Eq. 7.

$$\ln\left[\frac{[1-(1- \propto)^{1-n}]}{T^2(1-n)}\right] = \ln\left[\frac{AR}{\beta E}\left\{1 - \frac{2RT}{E}\right\}\right] - \frac{E}{RT} \tag{7}$$

where n ≠ 1 and Eq. 8,

$$ln\left[\frac{[-\ln(1-\propto)]}{T^2}\right] = ln\left[\frac{AR}{\beta E}\left\{1 - \frac{2RT}{E}\right\}\right] - \frac{E}{RT} \tag{8}$$

where n = 1.

Thus a plot of where either n ≠ 1, with $\ln\left[\frac{[1-(1- \propto)^{1-n}]}{T^2(1-n)}\right]$ plotted against $\frac{1}{T}$ or where n = 1, with $ln\left[\frac{[- ln(1-\propto)]}{T^2}\right]$ plotted against $\frac{1}{T}$ should result in a straight of slope $-\frac{E}{R}$ for the correct value of n, since it may be shown that for most values of *E* and for the temperature range over which reactions generally occur. The expression $ln\left[\frac{AR}{\beta E}\left\{1 - \frac{2RT}{E}\right\}\right]$ is sensibly constant, for $\frac{E}{RT} > 1, \left\{1 - \frac{2RT}{E}\right\} \approx 1$ with the slope $-\frac{E}{R}$ and the intercept $ln\left[\frac{AR}{\beta E}\right]$ of the curve. The frequency factor *A* and activation energy *E* can be calculated.

RESULTS AND DISCUSSION

Physicochemical Properties Analysis of the Sludge

The deinking sludge was usually processed in an incineration fluidized bed to eliminate the organic matter from the sludge and recover heat energy in the paper mill. Therefore, the parameters related to combustion, for the flocculation sludge with different flocculent dosages under optimized conditions, were compared with that of the floating deinking sludge from the paper mill in order to investigate the impact of flocculation sludge on the existing sludge treatment system. The results are shown in Table 1.

Table 1. Sludge Parameters Related to Combustion

Sludge Types	Sludge Source	Amount of Sludge Generated ($kg \cdot m^{-3}$)	Fiber Content (%)	Ash (%)	C (%)	H (%)	N (%)	S (%)	Heating Value ($MJ \cdot kg^{-1}$)
Floating deink sludge	Sampled from mill	-	12.21	47.90	24.48	2.71	0.30	0.42	6.30
Flocculation Sludge	Flocculant dosage: 400 mg/L	1.12	10.86	46.16	23.06	3.53	0.38	0.50	6.98
Flocculation Sludge	Flocculant dosage: 600 mg/L	1.52	10.40	47.19	19.68	3.34	0.52	0.76	5.76

Table 1 summarizes the amount of sludge generated under optimized conditions, the fiber content, as well as their parameters related to combustion, including the ash, heat value, and elements contents (C, H, N, and S). When the flocculation dosages were 400 and 600 mg/L, the amount of flocculation sludge generated in the treatment of the ink-containing whitewater were 1.12 and 1.52 kg/m^3, respectively. The average amount of water to be treated per 1 ton of deink pulp produced was approximately 10 m^3; thus, the total amounts of flocculation sludge generated were 11.2 and 15.2 kg, respectively, which accounted for less than 10% of the floating deink sludge, which would not greatly impact the exit sludge treatment system. The fiber content of the flocculation sludge was slightly lower than the flotation sludge. In addition, the amount of flocculent dosage had some influence on the sludge composition (as shown in Table 2), where the fiber, C, and H content, as well as the heat value decreased as the flocculation dosage increased. However, their heat values were similar to the floating deink sludge, so it could be predicted that it is feasible to treat flocculation sludge in the existing sludge incineration system.

Incineration Characteristics of the Flocculation Sludge in Air Atmosphere

To investigate the combustion performance of the three types of sludge, the relationship between the physicochemical properties of the samples and the temperature was measured *via* TGA with a heating rate of 10 °C/min as the temperature was increased from 50 to 1000 °C. Figures 2, 3, and 4 present the incineration curves of the sludges. The weight loss parameters of incineration for each of the samples are summarized in Table 2.

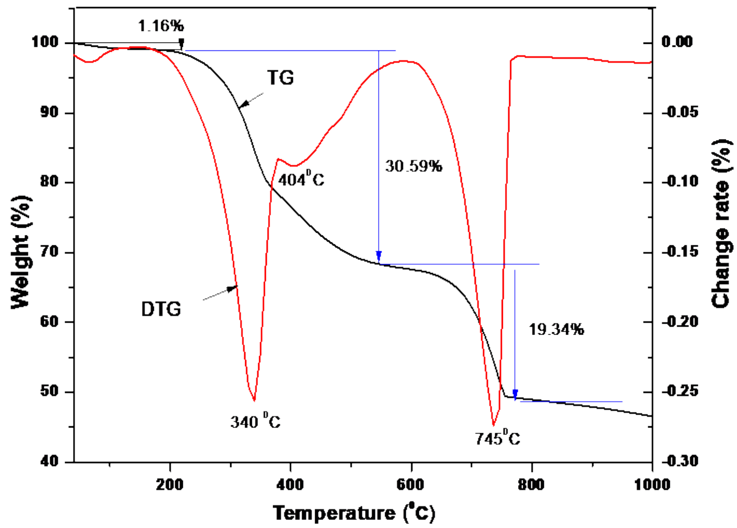

Fig. 2. Incineration curve of the floating deink sludge

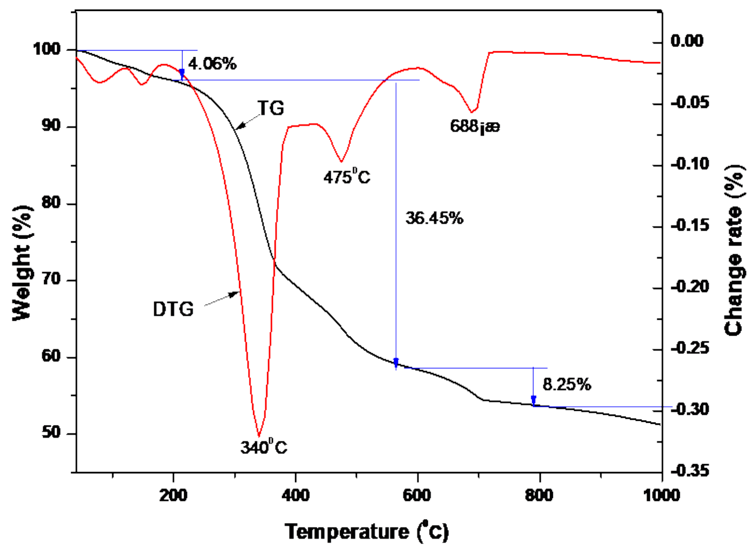

Fig. 3. Incineration curve of the flocculation sludge (Flocculent dosage: 400 mg/L)

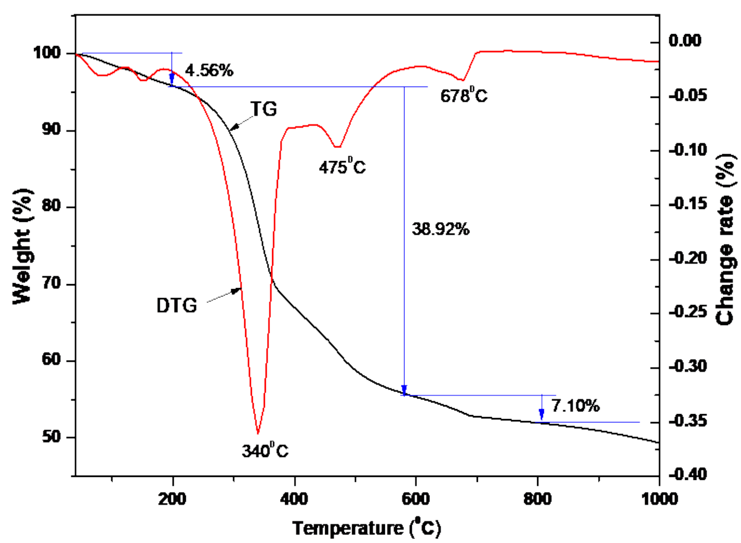

Fig. 4. Incineration curve of the flocculation sludge (Flocculant dosage: 600 mg/L)

Table 2. Weight Loss Characteristic Parameters of Different Sludge Incineration Zones (Heating Rate: 10 °C·min^{-1})

Sludge Types	T_1 (°C)	T_2 (°C)	T_3 (°C)	W_1 (%)	W_2 (%)	W_3 (%)	da_1/dT (%·°C^{-1})	da_2/dT (%·°C^{-1})	da_3/dT (%·°C^{-1})	M_∞ (%)
Floating deink sludge	340	404	745	84.65	75.10	64.74	-0.26	-0.09	-0.27	46.61
Flocculation Sludge (400 mg·L^{-1})	340	475	688	79.54	63.85	55.47	-0.32	-0.10	0.06	51.24
Flocculation Sludge (600 mg·L^{-1})	340	475	678	78.03	60.81	53.23	-0.36	0.10	0.03	49.42
Note: T_1, T_2, and T_3 are the peak temperatures; W_1, W_2, and W_3 are the residue weights at different peak temperatures; da_1/dt, da_2/dt, and da_3/dt are the maximum incineration rate of the samples; and M_∞ is the final residue weight of the sample										

As shown in Figs. 2, 3, and 4, the TG curves of the two flocculation sludges with different flocculent dosages in the air atmosphere were similar, while they were obviously different from the floating deink sludge. According to the characteristics of the DTG curve, the weight loss of the samples could be divided into 3 regions. The first region was from 50 to 200 °C. The DTG curves show several small fluctuations as part of water, and some low molecular weight substances, *e.g.*, some low molecular weight volatile acids were separated from the sludge samples. The weight losses of the three samples in this area were 1.16%, 4.06%, and 4.56%, respectively. The second region was from 200 to 400 °C, and there were large peaks on the DTG curve. The peak temperature of the maximum weight loss was at 340 °C, which was primarily from the volatile substances thermal cracked from the cellulose, hemicellulose, lignin, and connection materials of the ink in the sludge samples. The incineration of cellulose, a straight glycan polymer of glucose with a certain crystal structure, primarily occurs between 300 °C and 400 °C; while hemicellulose, composed of different monosaccharaides, are more readily decomposed compared to cellulose; its incineration primarily occurs between 200 °C and 300 °C; for lignins, an aromatic polymer with an amorphous mesh structure, they have a wide incineration decomposition temperature range, starting at 200 °C until the end of its incineration (Raveendran *et al.* 1996; Hu *et al.* 2007; Carrier *et al.* 2011). The third region was from 400 to 700 °C. There were two small peaks at 475 and 680 °C in the DTG curves of the two flocculating sludges; their total weight loss rate reached approximately 48.76% and 50.58%, respectively. However, there was no further decomposition when the temperature continued to increase greater than 700 °C. However, there was also a maximum peak at 745 °C in the DTG curve of the floating deink sludge with a weight loss rate of approximately 19.34%, its total weight loss rate was approximately 51.09%. The different incineration characteristics of the two types of sludge were primarily due to their different components, the same thermal decomposition peak at a low temperature was from their similar fiber content, while the weight loss of the flotation deinking sludge at 745 °C was primarily from carbon as pigment in the ink.

Kinetic Analysis

The Coats-Redfern method was used to analyze the combustion performance of the different samples by analyzing the weight loss data (Zhang 1996; Wu *et al*. 2007). The kinetic characteristics of the incineration region of the sludge organics were the primary concern, and the effect of the water content, ash, and metal salt melt on the kinetics needed to be eliminated. As shown in Fig. 5, ln $\left[\frac{[1-(1-\propto)^{1-n}]}{T^2(1-n)}\right]$ plotted against $\frac{1}{T}$ was well fitted when n = 2, in the selected region from 200 to 552 °C. The activation energy E and frequency factor A of the combustion process were calculated, as summarized in Table 3.

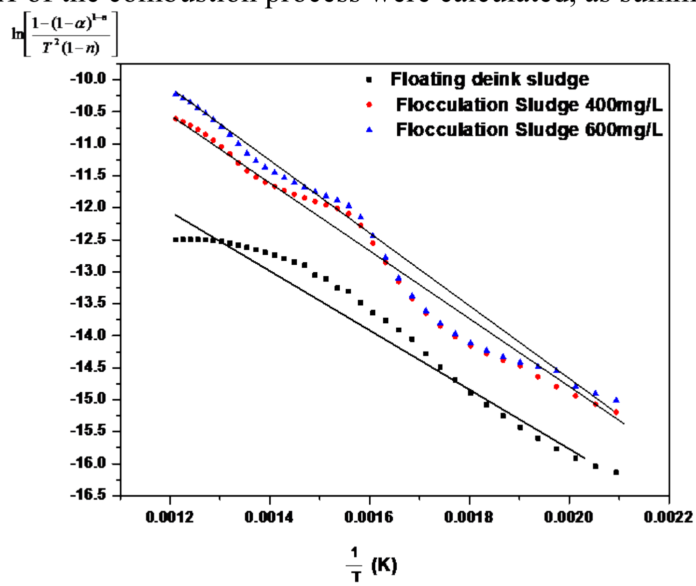

Fig. 5. Linear fitting data for the incineration of different sludges

Table 3. Incineration Kinetic Parameters of Different Samples (Heating Rate: 10 °C·min^{-1})

Sludge Types	Temperature Range (°C)	Fitting Equation	R^2	E (kJ·mol^{-1})	A (min^{-1})
Floating deink sludge	200 to 552	y = -4664.2x – 6.3161	0.9503	38.78	2.58E+07
Flocculation sludge (400 mg·L^{-1})	200 to 552	y = -5363.7x – 4.0848	0.9708	44.59	3.19E+06
Flocculation sludge (400 mg·L^{-1})	200 to 552	y = -5787.0x – 3.2556	0.9712	48.11	1.50E+06

As shown in Fig. 5 and Table 3, the linear relationship was well fitted. Thus, the kinetic equation can be used to describe the incineration reaction process of the organic compounds in the selected region. The activation energy of the flocculation sludge was greater than the activation energy of flotation sludge, and the activation energy increased as the flocculent dosage increased. Activation energy is an inherent property of a material. It is the minimum energy needed to achieve effective collision, the smaller the activation energy, the greater the reactivity, the stronger the reaction capacity, the faster the reaction speed; therefore, it is easier to fire (Zhang 1996). The calculated frequency factor sequence from large to small is as follows: flocculation sludge (600 mg·L^{-1}), was greater than flocculation sludge (400 mg·L^{-1}), which was greater than flotation deinking sludge. Only an activated molecular collision is an effective collision, which causes the reaction, in a chemical reaction. The larger frequency factor indicated that the more effective number of collisions between the activated molecules, the easier of the reaction, the more intension of the reaction degree, and the faster the reaction speed (Raveendran *et al.* 1996; Hu *et al.* 2007). The results of the activation energy and frequency factor showed that the organics in the flotation deinking sludge was much easier to incinerate than the flocculation sludge. In addition, the difficulty of incineration increased as the flocculent dosage increased.

Elements Content of the Flocculation Sludge and its Ash

Whether deinking sludge is a hazardous waste has been controversial in China. Therefore, qualitative analyze of the elements content in the flocculation sludge was performed *via* fluorometric spectrometer followed by a quantitative test *via* ICP-MS and an elemental analyzer. The results are shown in Fig. 6 and Table 4.

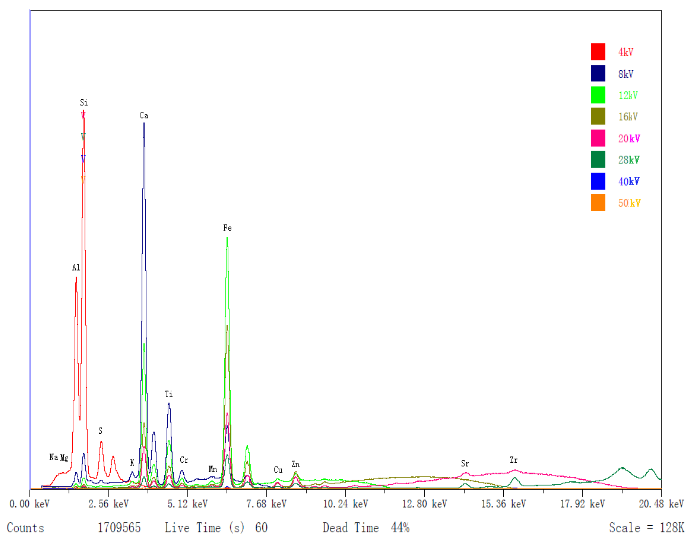

Fig. 6. EDXRF test results of the flocculating sludge

Table 4. Elements Content (mg·g^{-1}) in the Flocculation Sludge and Its Ash

	C	H	N	S	O	Na	Mg	Al	Si
I	230.6	35.3	3.80	5.00	539.8.	1.24	0.61	70.07	73.18
II	2.35	3.12	0.20	7.70	584.8	2.69	1.32	151.8	158.4
III	196.8	33.4	5.20	7.60	548.5	1.68	0.52	84.85	84.71
IV	2.05	3.46	0.30	9.85	542.4	3.56	1.11	179.8	179.5

	K	Ca	Fe	Cu	Mn	Zn	Cr	Ti	Zr	Sr
I	0.13	34.91	4.50	0.14	0.104	0.20	0.04	0.32	0.05	0.05
II	0.29	75.62	9.75	0.30	0.22	0.43	0.08	0.69	0.10	0.11
III	0.13	30.18	5.59	0.052	0.10	0.20	0.05	0.38	0.05	0.05
IV	0.28	63.96	11.85	0.11	0.22	0.42	0.10	0.80	0.11	0.10

Note: I -Flocculation Sludge (400 mg·L^{-1}) ; II - Ash of the Flocculation Sludge (400 mg·L^{-1}); III - Flocculation Sludge (600 mg·L^{-1}); IV - Ash of the Flocculation Sludge (600 mg·L^{-1})

As shown in Fig. 6, the elements Na, Mg, Al, Si, S, K, Ca, Cr, Cu, Fe, Mn, Zn, Ti, Sr, and Zr were detected in the EDXRF. The elements with an atomic weight below 11 are not included due to limitations in the capability of the fluorescence spectrometer. As shown in Table 4, besides the elements C, H, and O in the flocculation sludge, the Ca, Al, and Si contents were also high. The Ca, Al, and Si contents in flocculation sludge (400 mg·L^{-1}), flocculation sludge (600 mg·L^{-1}) and the resulting ash primarily came from filler. The elements C and H primarily came from the fiber in the sludge. The primary elemental content in the ash of the flocculation sludge were Al, Si, Ca, and O, as shown in Table 4. In addition, trace amounts of heavy metals, *e.g.*, Cu, Zn, and Cr, were detected in the sludge ash, although its content was lower than the limit of standard that sludge used in agriculture GB standard 18918 (2002) in acidic soil: the Cu is less than 800 mg·kg^{-1}, the Zn is less than 2000 mg·kg^{-1}, the Cr is less than 600 mg·kg^{-1}; in neutral or alkaline soils: the Cu is less than 1500 mg·kg^{-1}, the Zn is less than 3000 mg·kg^{-1}, and the Cr is less than 1000 mg·kg^{-1}, However, their accumulation in the soil and its long-term impact on the ecological environment need to be further studied.

CONCLUSIONS

1. The amount of sludge generated in the flocculation treatment of deink white water accounted for less than 10% of the flotation sludge, which would not greatly impact the sludge treatment system. The elemental content and calorific value related to combustion were similar to those in the flotation deinking sludge.
2. The incineration kinetic research results showed that although the activation energy of the organics in the flocculation sludge was slightly higher than the activation energy of the flotation sludge; however, they all belong to types that are ready for combustion. The flocculation sludge can be incorporated into the existing sludge incineration system for reduction and thermal energy recovery.
3. Trace amounts of Cu, Zn, and Cr were detected in the ash of the flocculation sludge. However, the content was lower than the Chinese environmental standards limit.

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