Thermogravimetric study on the Co-combustion characteristics of oily sludge with plant biomass

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Thermal utilization of oily sludge has attracted increased attention. This work studies the combustion characteristics of oily sludge, and its interaction with biomass as well as the reaction heat during the drying and combustion processes of oily sludge. All the experiments were performed by thermogravimetry. Results showed that the co-combustion process of oily sludge with biomass could be divided into three stages with the increase of the temperature. Different interactions between components of oily sludge and biomass existed at different temperatures. At low temperatures (280–390 °C) the interaction between these two fuels slowed down the combustion process of the mixtures, however, it accelerated the combustion process at high temperatures (390–620 °C). The caloric requirement curves of oily sludge with various water contents were obtained by DSC (differential scanning calorimetry) curves. The reaction heat of oily sludge or biomass combustion was also presented. The results obtained can improve the understanding of oily sludge during the combustion process and consequently can be used to develop more efficient sludge combustion equipment.

1. Introduction

It is of great interest to gain energy from solid waste in order to reduce its landfilling and decrease the consumption of fossil fuels. Oily sludge is a by-product of oil refineries and considered as a solid waste. In the petroleum industry, oily sludge is generated during crude oil exploitation and processing activities [1]. Oily sludge is primarily composed of large quantities of water, various petroleum hydrocarbons, heavy metals and solid particles [2]. Due to the increasing of oilfields worldwide, the production of oily sludge is growing. In China, approximately 3 million tons of oily sludge is produced annually in petrochemical industry [3]. Since it can be used as an energy resource, its thermal utilization has attracted increasing attention. Oily sludge without proper pre-treatment not only endangers the environment, but also wastes available resources. Many conventional methods were used to treat the oily sludge, such as farmland application and sludge disposal in landfills and oceans. However, because they can either cause serious pollution accidents or need high treatment costs, they are not environmentally friendly or cost-effective. Development green processing measures for oily sludge is essential for sustainability and environmental protection. Various thermal processes, e.g., pyrolysis, gasification, combustion, melting or vitrification, have been proposed for treating oily sludge, since they can destroy the sludge organic fractions and convert the sludge inorganic fractions into stable ash or slag; the thermal processes are considered as the promising methods for the use of oily sludge [4]. Using these thermal processes, oily sludge can either be advantageously reused, or harmlessly disposed at a landfill. To meet the increasingly stringent standards, it is necessary to study the stored energy in sludge and minimize environmental impacts [5].

Combustion is a set of complex exothermic chemical reaction, where the fuel oxidation will not only release the heat but also produce new chemical species including different gas, liquid and solid products [6]. The yield and composition of the products are affected by a range of combustion parameters (i.e. the fuel type, particle size, reaction system, reaction temperature, reaction time and heating rate). The combustion of oily sludge can reduce the space needed for its disposal, decomposing harmful poisonous substance and recovering energy. Due to the high moisture content,
dewatering and drying are the prerequisites for an efficient combustion processing of oily sludge [7]. Based on its components, combustion of oily sludge is regarded as a potential way for solid waste utilization. Some researchers [8–10] reported that the combustion of sewage sludge with other fuels could be significantly improved and better than using sewage sludge as a single fuel. The co-combustion of sewage sludge mixed with different fuels, such as biomass and coal, not only promotes the combustion characteristic of sludge but also improves the environmental impact and creates additional economic value. Biomass has a higher combustion rate than coal, resulting in faster expansion of ignition flame. Compared to coal combustion, biomass combustion has a number of other advantages such as its low ash content, and low S and N content that results in low NOx and SO2 emissions as well as its neutrality on CO2 emissions during its life cycle [11]. Based on the advantages, biomass is regarded as a potential feedstock for thermal conversion.

The literature is rich in resources regarding the disposal of oily sludge; however, the research about the combustion characteristics of oily sludge has been rarely reported. The co-combustion of oily sludge with biomass and their interaction have not been also extensively studied. Therefore, further research is necessary to study the combustion characteristics and interaction between oily sludge and biomass. Moreover, He et al. investigated the heat of pyrolysis and combustion of biomass over the integral of differential scanning calorimetry (DSC) curves [12]. To our knowledge, this research method is not used in the drying and combustion processes of oily sludge. The methods of calculating reaction heat for the drying and combustion processes of oily sludge by DSC curves have been not reported. Reaction heat of oily sludge combustion will be discussed in this work.

This work studies the combustion behavior of oily sludge and the co-combustion characteristics of oily sludge with biomass. Moreover, the calorific value was determined by DSC curves during sludge drying and combustion processes. The experimental results provide reference data for the use of new heat sources, such as boiler flue gas and metallurgy flue gas, as drying medium in order to commercialize drying and combustion of oily sludge.

2. Materials and methods

2.1. Sample preparation

Oily sludge used in this work was obtained from Sinopec Luoyang Company in Luoyang city Henan Province, China. Sinopec Luoyang Company applies the two-stage aerobic biological treatment to treat wastewater. Oily sludge is produced by biological treatment of wastewater. After the biological treatment the flocculation is employed to obtain oily sludge. Total discharge of wastewater includes industrial and living wastewater. Large amounts of aerobic bacteria are used to treat wastewater. In addition, living wastewater contains a large amount of proteins and fats. After the second-stage sedimentation pool, the compositions of oily sludge mainly contain the products of microbial residues decomposition. These products contain proteins, fats and a small quantity of untreated oils. Wood was taken as the representative material of plant biomass. The wood material was obtained from a wood factory around Xi’an in Shaanxi Province, Western China. The release of the volatile components of oily sludge started at a relatively high temperature and it generally occurred in the temperature range of 200–650°C [13]. Initial oily sludge was dried in the oven at 105°C until its mass did not change [14]. The oily sludge obtained by this process refers to dried oily sludge in this work. After milling and sieving into particles of 50–200 μm in diameter, the dried oily sludge and wood were dried in the oven at 105°C for 24 h. The ultimate and proximate analyses of two different samples are shown in Table 1, while Table 2 presents ash composition of the two tested materials in weight. Oily sludge samples with different water contents (20%, 40%, 60% and 90% for initial oily sludge) were obtained under different drying time at 105°C from a constant temperature oven. Dried oily sludge and wood were mixed together, with wood weight percentages of 20%, 40% and 60%.

2.2. Combustion experiments

Thermogravimetric analysis was performed in a STA-409PC thermal analyzer (NETZSCH, German), and its temperature ranges from 30°C to 900°C, with the heating rates of 20°C/min and weight precision of 0.001 mg. Prior to thermogravimetric analysis, the thermogravimetry baseline was adopted to reduce measurement errors after sample insertion. It was corrected by subtraction of predetermined baselines determined under identical conditions except for the absence of a sample. In the thermal analyzer system, each sample was heated in a micro-furnace enclosed by a cooling jacket, and water was used as the cooling agent. The sample temperature was measured with a type S (Pt-Rh/Pt) thermocouple set under the crucible (Al2O3). Sample (10 ± 0.01 mg) was loaded into an Al2O3 crucible for each run. In the experiments, combustion of the samples was carried out over a temperature range of 30–900°C, with a heating rate of 20°C/min, and the total carrier gas (20/80 in O2/N2) flow were constant as 100 mL/min. Tempera-

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Table 1
Ultimate and proximate analysis of two different samples.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Ultimate analysis/wt.%</th>
<th>Proximate analysis/wt.%</th>
<th>LHV/MJ kg⁻¹¹</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Cₚ</td>
<td>Hₚ</td>
<td>Oₚ</td>
</tr>
<tr>
<td>Oily sludge</td>
<td>40.81</td>
<td>4.60</td>
<td>20.32</td>
</tr>
<tr>
<td>Wood</td>
<td>44.75</td>
<td>4.98</td>
<td>39.85</td>
</tr>
</tbody>
</table>

* Moisture as air dried basis.
* Volatile matter as air dried basis.
* Fixed carbon as air dried basis.
* Ash as air dried basis.
* Low heating value.

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Table 2
Ash composition of the two tested materials.

<table>
<thead>
<tr>
<th>Ash samples</th>
<th>Ash composition/wt.%</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>SiO₂</td>
</tr>
<tr>
<td>Oily sludge</td>
<td>4.29</td>
</tr>
<tr>
<td>Wood</td>
<td>23.91</td>
</tr>
</tbody>
</table>
tures were controlled by linear increase to obtain the corresponding thermogravimetric (TG), differential thermogravimetric (DTG) and differential scanning calorimetry (DSC) combustion curves.

3. Results and discussion

3.1. Comparison of the combustion curves for oily sludge and wood

Investigation on the combustion of oily sludge and biomass by thermogravimetric analysis contributes to the prediction of the thermal processing of oily sludge and biomass, and establishes the optimum operational conditions for its better utilization of oily sludge and biomass. Fig. 1 shows the TG and DTG curves for dried oily sludge and wood combustion at a heating rate of 20 °C/min.

As indicated in Table 1, the characteristics of higher volatile matter and lower ash content of wood improve burning qualities compared to oily sludge. Also, the lower sulphur content and nitrogen content of wood are favorable to combustion conditions. The ignition and burnout temperatures are important combustion characteristic parameters for a fuel. The ignition temperature at which the DTG curve shows a sudden decrease is determined in Fig. 1b [15]. The ignition temperatures of oily sludge and wood were 243 °C and 307 °C, respectively. It was found that the ignition temperature of oily sludge was lower about 64 °C than that of wood although the volatile matter of wood was higher than that of oily sludge. Obvious differences between oily sludge and wood are caused by the composition, structure, properties and reactions. Wood mainly consisted of cellulose, hemicellulose and lignin. The three compositions had high heat-stability and were hard to break down. For oily sludge, the main groups were proteins, fats and oils [16,17]. These components were simple and easy to be broken down during the heating process. Therefore, the ignition temperature of oily sludge was lower compared to that of wood. The burnout temperature was obtained when the loss value on the TG curve reached 98% [18]. The burnout temperatures of oily sludge and wood were 525 °C and 502 °C, respectively. The ash of oily sludge was higher about 20.56% than that of wood as shown in Table 1. The ash of oily sludge contained metal salts shown in Table 2 and heavy metals, and these compositions involved complex reactions at high temperature. The wood ash included some alkali metals like potassium (K) and sodium (Na) which released easily at high temperature [19]. So, the burnout temperature of oily sludge was higher 23 °C than that of wood.

As shown in Fig. 1b, with the increase of temperature, the combustion process of dried oily sludge could be divided into three stages: the evaporation of moisture (<170 °C), the release and combustion of the volatile matter (170–390 °C) and burning of the fixed carbon and the residues (390–550 °C), respectively. The three reaction stages were determined based on the localization of the peak temperature points in Fig. 1b. The separation of oil from oily sludge occurs mainly from 200 to 650 °C [20,21]. Some oil have high boiling point and are difficult to evaporate under normal atmospheric temperature. From the thermogravimetric curves of oily sludge in Fig. 1a and 1b, it was concluded that the studied separation of oil from oily sludge studied mainly occurs between 170 and 550 °C. The first stage was caused by the effect of thermal loads, corresponding to the evaporation of moisture in oily sludge. The second stage which belonged to low temperature stage was described by a mass loss of about 34.27%, caused by the release and combustion of volatile matter and organic compounds. The lipa, proteins and saccharides in oily sludge were decomposed in the order of precedence in the second and third stages [22]. For these three components, the decomposition temperature ranged from low and high were 200–300 °C, 300–390 °C and more than 390 °C [22]. The third stage was high temperature stage of the sludge combustion. In this stage, the mass loss was about 38.91%, which was higher than the content of fixed carbon in oily sludge as shown in Table 1. It suggested that high temperature stage was not only the burnout of fixed carbon, but also attributed to the decomposition of organic residues and inorganic matters [23].

As Fig. 1 suggests, the combustion process of the wood was also divided into three stages. Compared with oily sludge, the TG and DTG curves of wood had much higher mass loss and mass loss rate respectively. The first stage was the evaporation of water from 30–130 °C. The second stage occurred at temperatures ranging from 130–400 °C, corresponding to the release of volatile matter. A great quantity of gas was produced at this stage with high temperatures and a long vapour residence period. This process improved the formation of higher percentages of liquids rich in organic molecules and significant char yields. During the release process, the inorganic mineral elements in Table 2 affected the degradation mechanisms of organic molecules existed in the wood and the chemical compositions of the volatile matter. The three main components of wood were cellulose, hemicelluloses and lignin. These components were broken down in the order of precedence in the second and third stages. The mass loss of lignin normally displayed a much wider decomposition temperature range of 160–627 °C, however, hemicelluloses degraded at 160–240 °C, and cellulose with a wider spectrum of inorganic elements occurred at the higher temperature range of 240–360 °C [24]. Accordingly, there was an obvious weight loss rate peak of the volatile matter on the DTG curve, and its value was 25.42%/min at 334.5 °C. At last, the third stage at 400–520 °C was the combustion and burnout of fixed carbon and residues. As the release came to completion, oxygen could diffuse to the highly reactive char product. At the same time, the oxidation rate rapidly increased and reached a maximum rate as a result of the exothermic combustion. The maximum weight loss rate on the DTG curve was 11.20%/min at 440.1 °C. The TG curve flatted out gradually after the third stage ended.

3.2. Co-combustion of oily sludge and biomass

The co-combustion characteristic of dried oily sludge and wood mixtures has been studied. Fig. 2 shows the TG and DTG curves at a heating rate of 20 °C/min for the combustion of dried oily sludge, wood and their mixtures in different proportions.

As shown by curves in Fig. 2, it can be seen that each TG curve for the mixtures varied between that of the individual materials, showing obvious contributions of sludge and wood to these profiles. The DTG curves of the mixtures showed two obvious peaks corresponding to the yield and combustion of the volatile matter and the burning of the fixed carbon and the residues in the mixtures, respectively. In the stage of the yield and combustion of the volatile, DTG curves for the mixture varied between those of the individual materials. But in the stage of the burning of the fixed carbon and residues, DTG curves for the 60%sludge/40%wood and 40%sludge/60%wood mixtures were beyond the DTG curve range of the individual materials. This indicated that adding a certain amount of wood increased the combustion intensity of mixtures. As oily sludge mass percentage increased, the combustion of the 80%sludge/20%wood mixtures was more similar to that of dried oily sludge.

Table 3 shows the combustion characteristic parameters of samples with different mass percentages of oily sludge. In Table 3, $T_i$ and $T_f$ are the ignition temperature and the burnout temperature for the samples, respectively. $T_1$, $T_2$ and $T_3$ are the temperatures according to the first peak, the second peak and the third peak, respectively. $DTG_{max}$ is the maximum mass loss rate and $M_0$ is the residual mass. Combustibility index was marked as $S$, and its value is obtained based on the literature [25].
As shown in Table 3, the ignition temperature of the mixtures increased with the increase of wood mass percentage. When the wood mass percentage was at 60%, the burnout temperature and \( M_m \) reached the minima of 514 °C and 14.56% respectively. Also, the maximum weight loss rate reached the maximum of 13.31%/min, which was 3.10%/min higher than that of dried oily sludge. Accordingly, the \( S \) value reached the maximum under the experimental conditions examined. The bigger the \( S \) value was, the better the combustion quality was [25]. Based on this fact, it is concluded that adding biomass to oily sludge can promote the combustion of oily sludge. The mixed ratios discussed above provide a valuable reference for the efficient utilization of oily sludge.

3.3. The interaction between oily sludge and wood

In order to illustrate the co-combustion mechanism of oily sludge and wood, the interaction of these two materials was studied. The interaction between fuels is a concern in many co-combustion applications. To investigate whether there is an interaction between oily sludge and wood, the theoretical TG/DTG curves of the mixtures are calculated by the average weight of the individuals [26].

\[
Y_{\text{mixture}} = X_{\text{sludge}} Y_{\text{sludge}} + X_{\text{wood}} Y_{\text{wood}}
\]

where \( X_{\text{sludge}} \) and \( X_{\text{wood}} \) are the percentage of oily sludge and wood in the mixture, respectively, and \( Y_{\text{sludge}} \) and \( Y_{\text{wood}} \) are the mass loss or mass loss rate of oily sludge and wood respectively.

For the different mixtures, the experimental TG and DTG curves and the calculated TG and DTG curves are shown presented in Fig. 3. As shown in Fig. 3a, the calculated TG curves were in agreement with the experimental TG curves at the initial and the end stage. However, the experimental TG curves lagged behind the calculated TG curves for each mixture when the temperature was higher than 330 °C, especially at 465 °C for the 60%sludge/40%wood mixture, the deviation value was up to 7.2%. Based on this result above, it indicated that some interaction may occur between oily sludge and wood during the co-combustion process.

**Fig. 1.** TG (a) and DTG (b) curves of dried oily sludge and wood.

**Table 3**

Combustion characteristic parameters of the mixtures.

<table>
<thead>
<tr>
<th>Samples</th>
<th>( T_1 / \degree \text{C} )</th>
<th>( T_2 / \degree \text{C} )</th>
<th>( T_3 / \degree \text{C} )</th>
<th>( T_4 / \degree \text{C} )</th>
<th>( T_5 / \degree \text{C} )</th>
<th>( \text{DTG}_{\text{max}} / % \text{min}^{-1} )</th>
<th>( M_m / % )</th>
<th>( S / \text{K}^{-1} \text{min}^{-2} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dried oily sludge</td>
<td>243</td>
<td>279</td>
<td>–</td>
<td>480</td>
<td>525</td>
<td>10.21</td>
<td>21.88</td>
<td>8.596E-08</td>
</tr>
<tr>
<td>80% Sludge/20% wood</td>
<td>244</td>
<td>283</td>
<td>340</td>
<td>454</td>
<td>526</td>
<td>10.14</td>
<td>20.58</td>
<td>8.610E-08</td>
</tr>
<tr>
<td>60% Sludge/40% wood</td>
<td>277</td>
<td>–</td>
<td>337</td>
<td>439</td>
<td>523</td>
<td>11.31</td>
<td>16.95</td>
<td>8.910E-08</td>
</tr>
<tr>
<td>40% Sludge/60% wood</td>
<td>292</td>
<td>–</td>
<td>338</td>
<td>440</td>
<td>514</td>
<td>13.31</td>
<td>14.56</td>
<td>10.331E-08</td>
</tr>
<tr>
<td>Wood</td>
<td>307</td>
<td>–</td>
<td>335</td>
<td>440</td>
<td>502</td>
<td>11.20</td>
<td>8.56</td>
<td>9.030E-08</td>
</tr>
</tbody>
</table>
ter released from wood reacted with oily sludge residues. It was for this reason that the experimental DTG curves lagged behind the calculated DTG curves. In the high temperature range of 390–620 °C, the interaction appeared to accelerate the combustion process of the mixtures. After wood was added to the oily sludge, the more heat was released to promote the thermal decomposition reaction. It improved the decomposition depth of the sludge residues [29]. Moreover, the wood experienced thermal degradation and produced the char at the high stage. The char could play a catalytic role in the oily sludge residue decomposition and increase the reaction rate. In addition, wood contained a certain amount of chlorine and alkali metals shown in Table 2. These matters may react with components of oily sludge to produce compounds with the low point (e.g. NaCl(g), KOH(g), K₂SO₄(g) and HCl(g), etc) [19].

3.4. Effect of water content on combustion process

After applying deductions for water content, thermogravimetric combustion curves of oily sludge with different water contents are presented in Fig. 4, showing different TG, DTG and DSC characteristic curves for oily sludge with different water contents. Changes in water content for oily sludge had a significant effect on combustion process of oily sludge. One of the peculiar characteristics of the combustion of initial oily sludge was the high content of moisture. In this study, the water contents in oily sludge decreased from 90% to 20%, thereby showing different thermogravimetric curves. The TG curves changed markedly. In the release and combustion of the volatile matter stage, the oily sludge with higher water contents (60–90%) had some small peaks as shown in Fig. 4b. This kind of sludge presented a semi-solid (liquid-plastic) form [30]. The more the water contents were, the less solid residues were. Correspondingly, the amounts of organic compounds and volatile matter produced were lower after oily sludge was dried. On the other side, the oily sludge with lower water contents (20–40%) expressed a jelly form and had more solid residues [30]. The amounts of organic compounds and volatile matter produced were, therefore, larger. There were clear peaks on the DTG curves as shown in Fig. 4b. In the burning of the fixed carbon and the residues stage, there were obvious waves on the TG, DTG and DSC curves for sludge with lower water contents. From the analyses presented above, the water content of oily sludge played an important role in oily sludge combustion. It was found that oily sludge with low water content enhanced combustion.
3.5. Reaction heat

Sludge drying equipment can reduce the water content of sludge and improve the dewatering characteristics so that the end products of dewatered sludge can be disposed of with less handling problems and environmental consequences. A design factor of sludge drying equipment is to keep heat transfer constant. A key problem of heat transfer calculation is that the sludge temperature variation is difficult to evaluate with precision. To calculate caloric requirement of sludge with different water contents during drying process is a computationally intractable problem. At present, there are more studies on the influence of some factors such as drying temperature and drying time on sludge drying than there are studies about heat transfer calculation during the sludge drying process [31]. The present study also analyzes the problem of heat transfer, since the heat transfer from external environment is the key factor influencing the sludge drying. Terminal temperature, heating rate and residence time are major characteristics of the heat transfer.

According to the literatures [12], the reaction heat of the sludge drying process under different water contents can be calculated by integrating the DSC curves. Fig. 5 shows that the caloric requirement curves of sludge under different water contents in the dry environment at temperatures ranging from 30° C to 200° C. It can be seen from Fig. 5 that 1218.30 kJ/kg, 807.98 kJ/kg, 220.42 kJ/kg, 208.78 kJ/kg and −18.12 kJ/kg were required when initial oily sludge, oily sludge with 60% water content, oily sludge with 40% water content, oily sludge with 20% water content and dried oily sludge were heated from 30° C to 200° C respectively. This tendency was consistent with the report about thermal energy requirement by Bennamoun et al. [32]. When dry oily sludge was heated to 200° C, some unstable substances in dry oily sludge began to separate and release heat after reacting with O2. It was therefore not difficult to see that the caloric requirement decreased greatly with the decrease of the water content in the oily sludge. Oily sludge was at a preheated stage on the temperature zone of 160–200° C and its caloric requirement were almost constant.

According to the literatures [12], the reaction heat curves of the combustion process of oily sludge and wood combustion can be calculated by integrating the DSC curves in Fig. 6a. The reaction heat curves of dried oily sludge and wood combustion are presented in Fig. 6b. Because of combustion reaction, the values of the reaction heat in Fig. 6 were negative in comparison with that of Fig. 5. It could be seen from Fig. 6 that 5.89 MJ/kg and 5.54 MJ/kg were released when wood and dried oily sludge were heated from 30° C to 900° C respectively. Compared with the lower heating value of...
the samples in Table 1, the net heat emission efficiencies of wood and dried oily sludge were 33.85% and 32.57% respectively. This is due to the contact of the carrier gas and the surface of the sample from the upper micro-furnace, reacting from top to bottom. The carrier gas didn't completely interact with the whole sample. Therefore, the combustion reaction efficiencies of oily sludge and wood were relatively low.

The oily sludge combustion will still play the important guiding part in the future waste disposal. However, the combustion technologies which are proved in practical disposal don't perform well. During practical combustion process, sludge passes through zones with different temperatures where drying, decomposition, combustion and the cooling of the ash take place. It is difficult to calculate precisely reaction heat of various temperature stages. As shown in Fig. 6, reaction heat curves for sludge combustion in different temperature ranges was obtained. When designing oily sludge combustion furnaces, reaction heat curves will enhance the efficiency of the design. The reaction heat curve provides reference data to understand the sludge combustion process and can be used to develop more efficient sludge combustion equipment.

4. Conclusions

The co-combustion characteristics of oily sludge and plant biomass, the caloric requirement of oily sludge drying process as well as the reaction heat of oily sludge or biomass combustion have been studied in this work. Thermogravimetric analysis showed that the combustion process of oily sludge was obviously different from that of biomass. Adding biomass to oily sludge could promote its combustion process of oily sludge. Different interactions between the co-combustion processes of oily sludge and biomass at different temperatures were observed. At 280–390 °C the interaction slowed down the combustion process of the mixtures, but it accelerated the combustion process at 390–620 °C. The drying caloric requirement of oily sludge under different water contents decreased greatly with the decrease of the water content. In different temperature ranges the reaction heat of oily sludge combustion is different from that of biomass. The results obtained provide references to understand and improve the oily sludge drying and combustion process.

References


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