A Preliminary Investigation of Filter Mud Dewatering and Its Combustive Properties

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Abstract. In order to evaluate the possibility of making use of the filter mud from sugar mills as a boiler fuel, experiment on dewatering the mud by filter press with expression at elevated temperature was carried out and the combustive properties of the dewatered mud were investigated. The proximate and calorific values of the dewatered mud were determined, and the combustive properties were analyzed through thermogravimetric analysis. The results shown that the filter mud could be dewatered to the moisture content around 50% when expressed under the pressure between 1.6~2.5MPa at 95℃. The contents of moisture, ashes, volatile matters and fixed carbon on air dried basis were about 9.4%, 19.8%, 58.9%, 12.0% respectively and the calorific value of dewatered mud was around 8450kJ/kg, indicating that it is an appropriate fuel. The thermogravimetric analysis showed that the pyrolysis and combustion of volatiles took place between 200~370℃, where the weight loss accounted for over 50%. The fixed carbon was combusted between 370~550℃. The temperatures at ignition, peak burning rate and burn out were 280℃, 340℃ and 550℃ respectively. The ignition, burnout and comprehensive combustion indexes were 3.8×10⁻⁵ mg·min⁻¹·℃⁻², 7.2×10⁻³ min⁻¹, 1.8×10⁻⁸ mg²·min⁻²·℃⁻³ respectively.

1. Introduction
Filter mud is one of the three major by-products of sugar mills, accounting for 3-4% of the cane processed. It is a large volume waste to be disposed by the sugar mills. As a conventional treatment, most of the mud is made back to the field as a harmless disposal. But recently it becomes more and more unwelcomed by farmers in China. The primary reason for this is that it is hardly effective as an agricultural fertilizer. It generates intense heat (65℃), foul odor and takes long time for natural decomposition[1], furthermore, having water content around 70-80%, it is inconvenient for transportation and application.

On the other hand, there is always a shortfall of fuel for boilers for sugar factories that produce white sugar with double processes (first to produce raw sugar and then to refine it) during the off crop season. For those factories, during the grinding season, the bagasse from the cane is used to burn the boilers, but for the off grinding refinery, surplus bagasse is insufficient as the fuel, purchasing make-up burning fuel constitutes significant cost.

Filter mud of sugar mills is a complex product containing crude wax (5~14%), fiber (15-30%), crude protein (5~15%), sugar (5~15%), SiO (4~10%), CaO (1~4%), PO (1~3%), MgO(0.5~1.5%) and total ash (9~10%)[2]. There may be a possibility that the mud could become a biofuel after it is
dewatered. Dewatering of sludge with membrane filter press has gained application as advanced technology\textsuperscript{3}. The filter mud is the solid residue obtained in the sugarcane juice clarification process, which is possible to be further dewatered by filter press and expression at elevated temperature.

The objective of the current work is to carry out a primary trial on dewatering the filter mud with filter press and expression at elevated temperature, and then investigate its combustive properties, thus to evaluate its possibility as boiler fuel.

2. Experimental

2.1. Material
Filter mud was collected from COFCO Chongzuo Sugar Company Limited. The mud was stored in the fridge at -20\textdegree C before performing the experiment.

2.2. Dewatering
The dewatering was carried out in an experimental rig shown in Fig 1 including mainly a slurry tank with heater, a screw pump, and a filter press with expression. The mud was thawed and mixed with hot water of 4 times the weight of mud, the obtained mud slurry was then heated to 95\textdegree C in the slurry tank. The heated slurry was pumped and filtered in the filter press under the pressures of 0.5MPa. When then filtration ended, the expression pushed by a hydraulic device was carried out under different pressure.

2.3. Analysis of combustive properties
The proximate on air-dried basis for dewatered mud was analyzed according to the China standard for coal analysis, GB/T 212-2008, and the net calorific value on received basis was analyzed according to the China standard, GB/T 213-2008.

Thermogravimetric experiments were conducted with the ZRY-2P TGA equipment produced by Shanghai Precision Science Instrument Co., Ltd. The dewater filter mud was dried in a box dryer at 105 \textdegree C for 2 hours until constant weight was obtained. Approximately 10 \pm 0.5 mg of the sample was used in alumina crucibles in each thermogravimetric analysis. The temperature is set from 0 to 910\textdegree C at the heating rate of 10\textdegree C min\(^{-1}\) under the air atmosphere. The sampling temperature is set as 40\textdegree C. The samples selected randomly in the same batch were repeated for three times in an experiment to confirm the repeatability and authenticity of the generated data, and the resultant errors were within \pm 2%.

Instruments used included Muffle furnace (Shenzhen Zhongda electric furnace manufacturer, China), PARR 6300 automatic oxygen bomb calorimeter (PARR USA), ZRY-2P thermogravimetric analyzer (Shanghai Precision Science Instrument Co., Ltd., China), Box dryer (Wujiang Dade electric oven manufacturing Co. Ltd. China), BSA224S analytical balance (SARTORIUS Germany), etc.

3. Result and Discussion

3.1. Dewatering
The moisture contents of the mud dewatering under different expression pressures are shown in table 1.

| Expression pressure /MPa | 0.8  | 1.6  | 2.5  |
|--------------------------|------|------|------|
| Moisture content /%      | 58.2 | 53.7 | 48.8 |

It is shown that under the experimental conditions, the filter mud could be dewatered to the moisture content around 50\%, the average original moisture content was 75\%. The dewatered filter mud is shown in Fig. 2.
3.2. Dewatering

The proximate and calorific values of the dewatered filter mud were determined as table 2. In the table, $M_{ar}$ is the moisture content on received basis, $M_{ad}$, $A_{ad}$, $V_{ad}$, $FC_{ad}$ are contents of moisture, ash, volatile matters and fixed carbon on air dried basis respectively. $Q_{ar,net}$ is the net calorific value on received basis.

| Expression pressure, /MPa | $M_{ar}$ /% | $M_{ad}$ /% | $A_{ad}$ /% | $V_{ad}$ /% | $FC_{ad}$ /% | $Q_{ar,net}$ /kJ·kg⁻¹ |
|---------------------------|-------------|-------------|-------------|-------------|-------------|-------------------------|
| 0.8                       | 58.2        | 9.82        | 19.06       | 59.99       | 11.13       | 7037.6                  |
| 1.6                       | 53.7        | 9.50        | 19.72       | 58.91       | 11.87       | 7949.9                  |
| 2.5                       | 48.8        | 9.31        | 19.86       | 58.85       | 11.98       | 8943.3                  |

The test results show that the dewatered filter mud has high content of volatile matters. If the filter mud is dewatered to the moisture content of 50%, the contents of moisture, ashes, volatile matters and fixed carbon on air dried basis were 9.4%, 19.8%, 58.9%, 12.0% respectively. The calorific value of bagasse having 50% moisture content is estimated to be about 7540 kJ/kg⁰¹, the dewatered mud is shown to have even higher calorific value, indicating that it could be an appropriate fuel.

3.3. Thermogravimetric analyses

3.3.1. Thermogravimetric curve analyses. As shown in Fig.3, the weight loss versus temperature curve of the dewatered filter mud during heat treatment up to 900°C in the air atmosphere at a 10°C /min of heating rate is provided. Based on the process of combustion, the curve can be divided into four phases: the heating and drying, the emission and combustion of volatiles, the combustion of fixed carbon, and the formation of ashes. The phase of heating and drying took place between 40~200°C, there was a slight and gradual weight loss in this phase. The pyrolysis and combustion of volatiles took place between 200~370°C, the weight loss was significant, accounting for over 50% of the overall weight loss. The fixed carbon was mainly combusted between 370~550°C, during which the carbon was burning into carbon dioxide and a thermal heat of 32750J per gram carbon was released. As the temperature increased from 550°C to 900°C during the experiment, no significant weight loss was observed and the residual ashes was formed.

Based on the derivative thermogravity (DTG) curves shown in Fig.4, the largest pyrolysis happened between 250~400°C, the highest extreme point took place at about 330°C, signifying the highest pyrolysis rate of volatiles. Generally, the highest pyrolysis rate of cellulose occur at 360-390°C and that of hemicellulose at 270-300°C, the highest pyrolysis rate should signify that the major
component of filter mud was bagacillo. Another smaller extreme point took place at 450 °C, during period of combustion of carbon, this may be due to the pyrolysis of calcium salts of organic acids, as a large quantity of lime milk is added in the clarification of cane juice.

3.3.2. Determination of combustion characteristic parameters. The thermogravimetric experiment provides the temperature and mass loss varying with time. Fig.5 is the comprehensive thermogravimetric analysis diagram of dewatered filter mud.

From Fig 5 the flowing parameters are obtained:
(1) the ignition temperature, $T_i$, at which a sample starts burning, which is determined by the TG–DTG tangent method\cite{5-8},
(2) the peak temperature ($T_p$) corresponding to the maximum decomposition rate, and
(3) the burn out temperature at the end of the decomposition ($T_f$) corresponding to the point in which the mass loss starts to plateau with increasing time.

These parameters can be derived from TG and DTG curves\cite{9,10}.

The ignition index ($D_i$) and the burnout index ($D_f$) are defined in Eq. (1) and Eq. (2) below, respectively\cite{11,12}:

$$D_i = \frac{DTG_{\text{max}}}{t_p t_i}$$

where DTG$_{\text{max}}$ is the maximum combustion rates, $t_p$ is the corresponding time of DTG$_{\text{max}}$, $t_i$ is the ignition time.

$$D_f = \frac{DTG_{\text{max}}}{\Delta t_{1/2} t_p t_f}$$

where $\Delta t_{1/2}$ is the time range of DTG/DTG$_{\text{max}} = 0.5$, $t_f$ is the burnout time.

The comprehensive combustion index S is defined in Eq. (3).

$$S = \frac{DTG_{\text{max}}DTG_{\text{mean}}}{T_i T_f}$$
where DTG mean is the mean combustion rates. The comprehensive combustion index reflects the ignition, combustion and burnout properties of a sample. Generally speaking, the higher the index, the better the combustion performance of the sample.

Based on the thermogravimetric analysis, the combustion characteristic parameters of the dewatered filter mud are derived as shown in table 3.

### Table 3: Combustion characteristic parameters of filter mud

| Parameters | $T_i$ /°C | $T_p$ /°C | $T_f$ /°C | $D_i$ /mg·min$^{-1}·°C^{-2}$ | $D_f$ /min$^{-1}$ | $S$ /mg$^2·min^{-2}·°C^{-3}$ |
|------------|-----------|-----------|-----------|---------------------------|-----------------|--------------------------|
| Values     | 280       | 340       | 550       | $3.8×10^{-5}$              | $7.2×10^{-3}$   | $1.8×10^{-8}$        |

4. Conclusion

1. The filter mud from sugar mills was experimentally dewatered with a filter press with expression at elevated temperature. The mud was dewatered to the moisture content around 50% under the pressure between 1.6-2.5MPa at 95°C.

2. The proximate analysis and the calorific values determination of the dewatered mud shown that the contents of moisture, ashes, volatile matters and fixed carbon on air dried basis were about 9.4%, 19.8%, 58.9%, 12.0% respectively and the calorific value of dewater mud was around 8450kJ/kg.

3. The thermogravimetric analysis showed that the pyrolysis and combustion of volatiles took place between 200–370°C, where the weight loss accounted for over 50%. The fixed carbon was combusted between 370–550°C. The temperatures at ignition, peak burning rate and burn out were 280°C, 340°C and 550°C respectively. The ignition, burnout and comprehensive combustion indexes were $3.8×10^{-5}$ mg·min$^{-1}·°C^{-2}$, $7.2×10^{-3}$ min$^{-1}$, $1.8×10^{-8}$ mg$^2·min^{-2}·°C^{-3}$ respectively.

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