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Research Article

Properties of furfural residue and its drying characteristics in single-shaft paddle heat exchangers

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Abstract

Furfural residue is a detrimental waste generated in furfural production processes. Combustion is an efficient way to dispose furfural residue and drying is a necessary unit operation prior to combustion. In this paper, the properties of furfural residue, including particle size distribution, proximate analysis and pyrolysis process curves were presented. The flow and drying characteristics of furfural residue in a lab-scale single-shaft paddle heat exchanger were documented. The measurements show that the furfural residue contains high moisture (54.26%) and its as received basis lower heating value is 7301 kJ/kg. The high moisture content in furfural residue makes ignition and efficient combustion very difficult. It is feasible to use single-shaft paddle heat exchanger to dry furfural residue to moisture content of 15.94% which is advantageous for further combustion due to low moisture content. No agglutination and blockage were found. This preliminary experimental research provides reference for furfural residue drying unit design.

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1. Introduction

Furfural ($C_5H_4O_2$) is a kind of organic compound which is used in many fields, such as chemical engineering, pharmaceutical engineering and so on [1]. It is commonly produced from agricultural and forestry byproducts, such as corn cob and sugar cane bagasse. At present, China is the largest producer of furfural [1]. In furfural production processes, furfural residue with high moisture content is generated which is detrimental to environment. Many methods have been proposed to dispose furfural residue [2-3]. Among them, high temperature combustion is an important treatment way which has relatively short workflow [4-6]. The combustion device design is closely relevant to furfural residue's properties, including physical features (e.g., agglutination degree, particle size) and proximate analysis results. Direct combustion of materials with high moisture content will cause difficulty in ignition, low combustion temperature, low combustion efficiency, large exhaust gas heat loss and large exhaust treatment cost [7], so removing moisture in materials (i.e., drying) prior to combustion is always necessary for furfural residue disposal system optimization.

Meng [8] discussed the application of spin flash drying technique in drying furfural residue in which hot air (200 °C) directly contacts the wet furfural residue and removes its moisture to obtain dry product with moisture content less than 6%. Gao et al. [9] did mechanism research of furfural residue drying process in which the effects of particle size,

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drying temperature, heating rate and initial load were documented and a few drying models were evaluated. Paddle heat exchanger (or paddle dryer) is a kind of indirect heating equipment with high energy efficiency and low exhaust gas treatment cost that has been used in various sludge and fine-particle material drying processes [10-14]. Different from normal fluid-to-fluid heat exchangers with static heat transfer surfaces that cannot convey solid materials, paddle heat exchanger has rotating heat transfer surfaces. Hollow paddles welded on a hollow shaft have a designed inclined angle. Under operating conditions, when the shaft rotates, the material flows from the inlet end to the outlet end driven by the inclined paddles and at the same time it is heated by the hot shaft-paddle surfaces to reduce its moisture content. The success or not of using paddle heat exchangers strongly depends on the properties of materials to be dried. For instance, two-shaft paddle heat exchangers can dry agglutinating paste materials (e.g., sewage sludge) and non-agglutinating particle materials, but single-shaft paddle heat exchangers are mainly proper for the later. Meanwhile, when the particle size is larger than a critical value, the material flow may be blocked due to small gap between the rotating surfaces and the static surfaces of the shell of the equipment [13]. Up to know, although great progress has been made in modelling drying processes [15-16], it is difficult to quantitatively predict from theory whether a wet material can be successfully dried by paddle heat exchangers or not. Experiment is still the main way of validation.

In this paper we document the basic properties (especially those drying unit design related) of furfural residue and measure the drying characteristics in a lab-scale single-shaft paddle heat exchanger which is simpler than its two-shaft counterpart. The aim is to evaluate the drying feasibility and provide reference for conceptual design of application systems.

2. Experimental Methods

2.1. Property Test

The raw furfural residue sample is shown in Fig. 1 which was collected from a cane sugar plant (Changling Sugar, Guangxi, China) in which furfural was produced from sugar cane bagasse. From the appearance, furfural residue is a mixture of dark brown non-continuous wet particles. The particle size distribution was measured by a series of standard sieves (mesh number 10, 24, 30, 60, 100, 170, 200, 250). By measuring the mass in a specified particle size range, the corresponding mass fraction was obtained. The uncertainty of the corresponding mass weighing is 10 mg [17]. The obtained maximum absolute uncertainty of the mass fraction measurements is 0.00063%. Proximate analysis was performed according to Chinese National Standard GB/T 28731-2012 [17-20]. The uncertainty of the mass measurements in proximate analysis is 0.1 mg. The obtained maximum absolute uncertainty of the measurements of the moisture content (M_{ar}), ash content (A_{ar}), volatile matter content (V_{ar}) and fixed carbon content (FC_{ar}) is 0.06%. The uncertainty of the lower heating value (LHV) measurement is 50 J/g. In order to avoid local pyrolysis due to high temperature, the highest drying temperature should be constrained in design. Pyrolysis process was recorded by HCT-1 TG Analyzer (Beijing Hengjiu Co.). In the pyrolysis experiment, nitrogen with flow rate 50 mL/min was used to provide inert atmosphere. The sample mass was 5.12 mg and the temperature rising rate was set as 10 °C /min. For HCT-1 TG Analyzer, the uncertainties of temperature and mass measurements are 0.1 °C and 0.1 µg respectively [17].

2.2. Drying test

Fig. 2 shows the experimental setup. The specifications of the single-shaft paddle heat exchanger (i.e., dryer) are: heat transfer area 1 m², inclined angle of paddles 4 °, number of paddle pairs 14 and paddle pitch 70 mm. Heat transfer oil was heated in the boiler and

flowed through the hollow shaft and paddles. Furfural residue flowed on the outside of the shaft and paddles and meanwhile the moist content was reduced along the flow path. The oil flow rate was adjusted based on the data of a vortex flow meter (DN32, VF-LUGB24-652, Shanghai Weiliu Co.) and the oil temperatures at the inlet and outlet were measured by thermal resistors (Pt100). In real applications, saturated steam is commonly used as the working fluid. In the present experiment, we adjusted the oil flow rate to make its temperature change along the flow path as little as possible.



Fig. 1. Raw sugar cane bagasse furfural residue.

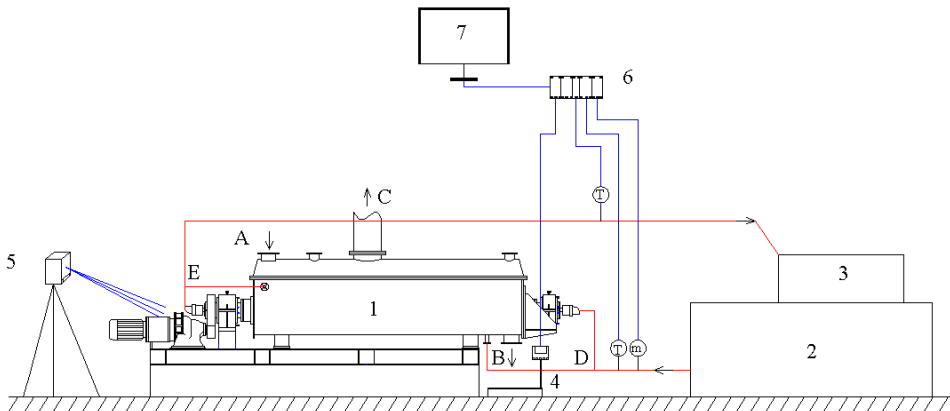


Fig. 2. Experimental setup [17].

1- paddle heat exchanger; 2- boiler; 3- oil tank; 4- electronic scale; 5- photo tachometer; 6- data acquisition unit; 7- computer; A- material inlet; B- material outlet; C- exhaust gas outlet; D- oil inlet; E- oil outlet; m- flow meter; T- temperature sensor

The flow rate of furfural residue at the outlet (m_o) was obtained via real-time continuous weighing with the sampling period of 1 s by an electronic balance (TCS-150) with 0.01 kg accuracy. A computer with data acquisition unit recorded the outlet furfural residue mass continuously and the outlet mass flow rate was then calculated based on the mass vs. time data. The inlet flow rate (m_i) was deduced according to dry matter mass conservation.

$$m_i = \frac{1-x_o}{1-x_i} m_o. \quad (1)$$

In Eq. (1), x_i and x_o are inlet and outlet moisture contents respectively and were tested based on Chinese National Standard GB/T 28731-2012 [18]. Besides, in experiments, the rotating speed of the shaft, which can be adjusted by motor inverter in the range 0~50 rpm, was acquisitioned by an infrared photo tachometer (XSM/C).

3. Results and Discussion

3.1. Properties

The particle size distribution of raw (wet) furfural residue with moisture content of 54.26% is shown in Fig. 3. The data in Fig. 3 are correlated as

$$y = 2.67909x^2 - 16.38295x + 30.03408 \quad (dm = 0-0.09 \text{ mm}) \quad (2a)$$

$$y = -0.35185x^5 + 9.66385x^4 - 105.86887x^3 + 578.81593x^2 - 1582.94284x + 1745.53418 \quad (dm = 0.09-1.7 \text{ mm}) \quad (2b)$$

where

$$x = \ln(1000dm) \quad (3a)$$

$$y = \ln(1000r/\Delta d) \quad (3b)$$

In Eqs. (2a), (2b) and (3a), dm is the average particle diameter in a specified range, e.g., $dm = 1.2 \text{ mm}$ in the range 0.7-1.7 mm. In Eq. (3b), r and Δd represent the mass fraction in a specified range and the range length respectively, e.g., $\Delta d = 1 \text{ mm}$ in the range 0.7-1.7 mm.

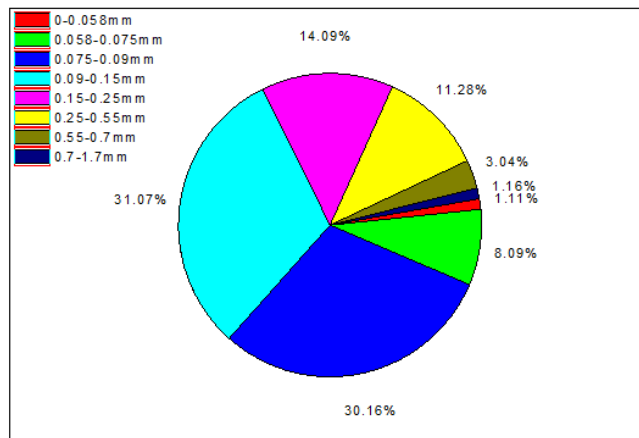


Fig. 3. Particle size distribution of raw furfural residue (moisture content: 54.26%)

According to Fig. 3, the size of most particles (95.8%) is less than 0.55 mm and the average size is 0.174 mm. Fine particles mean that blockage may not happen in single-shaft paddle

heat exchangers. This prediction was validated in flow and drying experiments. Besides, this measurement also provides evidence for selecting proper combustion mode. In the experiment performed by Gao et al. [9], the range of particle sizes is 0.5~10 mm which is very different from that of the sample in this paper although the two samples have similar moisture content. The corresponding bulk density of the present sample is 400 kg/m³ [17] with the uncertainty less than 1 kg/m³ which is also different from that in Ref. [9]. In fact, the present sample is cane sugar bagasse furfural residue but the sample in Ref. [9] is not. This comparison shows that the physical properties (like particle size and bulk density) significantly depend on how the furfural residues are produced. Agglutination is a critical physical problem in paddle heat exchanger applications. By direct observation, we noticed that the furfural residue particles are dispersed, meaning that agglutination may not matter. This was finally checked via drying experiment too.

Table 1 documents the proximate analysis results of raw (wet) furfural residue where M_{ar} is moisture content, A_{ar} is ash content, V_{ar} is volatile content, FC_{ar} is fixed carbon content and LHV is lower heating value. The moisture content (54.26% for the present sample) is the most important parameter for drying unit design. Note that in real applications, the moisture content of raw furfural residue may not be completely the same because of different furfural production processes, deposit, transportation and inevitable non-uniformity. Generally, the highest moisture content is about 60% [1-2]. We assumed the ambient temperature is 25 °C and the latent heat of water evaporation is 2257 kJ/kg at 0.1 MPa. We considered the sensible heat loss of liquid water heating and the latent heat loss of water evaporation in combustion process. From the LHV with moisture content of 54.26%, the LHV with moisture content of 60% can be estimated, which is about 6062 kJ/kg. This result tells that furfural residue with moisture content less than 60% can be used as fuel. A properly designed drying-combustion integration system can generate net heat to outside users.

Table 1. Proximate analysis (as received basis) of raw furfural residue.

| M_{ar} | A_{ar} | V_{ar} | FC_{ar} | LHV |
|----------|----------|----------|-----------|------------|
| 54.26% | 1.73% | 30.07% | 13.94% | 7301 kJ/kg |

Fig. 4 presents the pyrolysis process curves (i.e., TG and DTG). In the range from initial temperature to about 100 °C, TG drops fast which is usually called drying stage. The following stage ranges from 100 °C to about 270 °C, at the end of which most moisture loses. When the temperature is higher than 270 °C, slight pyrolysis initiates. For materials with moisture, when the hot solid surface temperature approaches pyrolysis temperature, the material temperature is usually much lower than the solid surface temperature due to water evaporation cooling effect. Even though, due to non-uniformity of the material particle size distribution and flow distribution, it is possible to generate local high temperature for the material. So, based on this consideration, it is safe to use oil temperature no higher than 270 °C to make sure that no pyrolysis occurs for any particles in any location in a paddle heat exchanger.

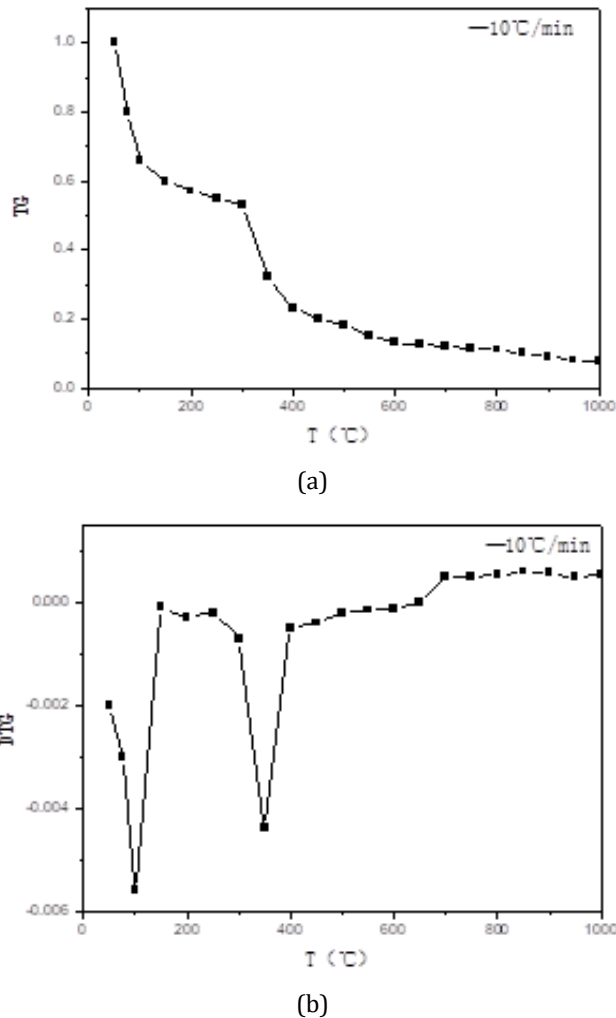


Fig. 4. Pyrolysis process curves of furfural residue (a) TG, (b) DTG

3.2. Drying characteristics

For specified furfural residue, its flow rate m_i at the inlet of the specified paddle heat exchanger in this paper depends on the rotating speed n , whose effect is given in Fig. 5. For the flow test without heating, the maximum relative uncertainty of m_i (or m_o) is less than 0.2% in the specified flow rate range. Approximately, the flow rate increases with the increasing of rotating speed linearly. How to choose n in design is a problem of trade-off. Higher rotating speed increases furfural residue disposal capacity but shortens the residence time which increases the outlet moisture content.

Table 2 documents two cases of drying experiments. In the drying test, the absolute uncertainties of the temperature and moisture content measurements are 0.1 °C and 0.02% respectively. The obtained uncertainty of the inlet flow rate (m_i) based on Eq. (1) is about 0.1 kg/h for both cases. More details of the experiments can be found in Ref. [17]. Both cold-state and drying experimental results do not show furfural residue agglutination

on solid surfaces and flow blockage. For imitating saturated steam heating process, we adjusted the oil temperature difference between inlet and outlet to be very small (less than 3 °C). Case II uses higher average oil temperature (181.45 °C) than Case I (132.0 °C). Although the two cases have different inlet moisture contents, the furfural residue flow rates are the same. The outlet moisture content of Case I is close to the inlet moisture content of Case II. We can approximately seem the outlet of Case I as the inlet of Case II but with different oil temperature. Based on the data in Table 2, we can calculate the removed moisture Δm in each case.

$$\Delta m = \frac{x_i - x_o}{1 - x_o} m_i. \quad (4)$$

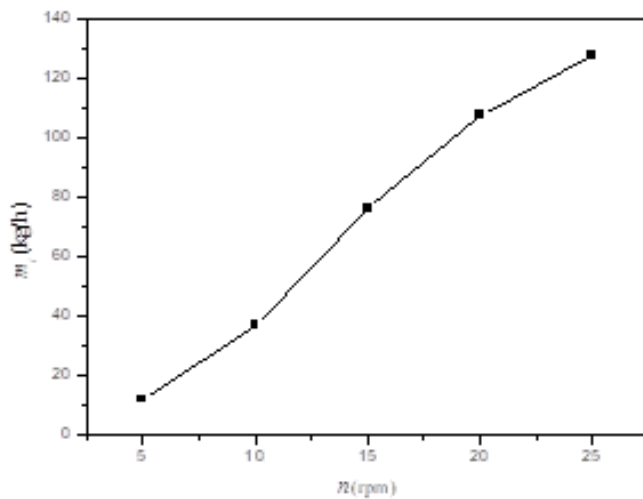


Fig. 5. Flow rate of raw furfural residue (moisture content: 54.26%)

Table 2. Results of drying experiments

| Parameters | Case I | Case II |
|---|--------|---------|
| Rotating speed (rpm) | 10 | 10 |
| Inlet oil temperature (°C) | 133.9 | 182.9 |
| Outlet oil temperature (°C) | 130.1 | 180.0 |
| Inlet furfural residue temperature (°C) | 22.0 | 22.0 |
| Inlet mass flow rate of furfural residue (kg/h) | 37.0 | 37.0 |
| Inlet moisture content of furfural residue (%) | 54.26 | 41.31 |
| Outlet moisture content of furfural residue (%) | 40.64 | 15.94 |

Based on Eq. (4), Δm is 8.49 kg/h for Case I and 11.17 kg/h for Case II. Case II with higher oil temperature removes more moisture than Case I. In the present analysis, assume that the temperature difference between oil and furfural residue (or the oil temperature itself) is more significant and the overall heat transfer coefficient does not change significantly with moisture content in the specified range. It is safe to use 181.45 °C (oil temperature of

Case II) to dry wet furfural residue (with flow rate 37.0 kg/h) from moisture content 54.26% to 15.94% with two 1 m² single paddle heat exchangers in series if we combine Case I and Case II together. The equivalent paddle heat exchanger area is 2 m². For combustion process, less than 20% moisture content is usually acceptable from the viewpoints of technology (i.e., ignition) and economy. Lower outlet moisture content means larger paddle heat exchanger area or more equipment investment. Under such condition, the exhaust gas of drying process will carry dust easily. Trade-off in design is necessary.

3.3. Further discussions

In engineering design, drying unit is suggested to be integrated into the drying-combustion integration system. Fig. 6 shows how the paddle heat exchanger is used in the system. Wet furfural residue is dried in the paddle heat exchanger using saturated steam generated by burning dry furfural residue in the boiler. More steam than needed by drying is output to outside users. Because of the indirect drying feature, most of the exhaust gas generated in the paddle heat exchanger is steam. It is possible to recover and reuse the heat stored in the exhaust gas through proper methods like condensing. If so, the energy efficiency of the drying-combustion integration system will be improved. In China, there are a lot of industrial steam boilers with steam pressure no higher than 1 MPa, including some installed in furfural production factories. The saturation temperature of steam at 1 MPa is about 180 °C which is very close to oil temperature used in Case II. So it is convenient and cheaper to use these boilers to burn dry furfural residue and provide saturated steam to dry wet furfural residue. Detailed analysis and optimization on drying-combustion integration system will be performed later.

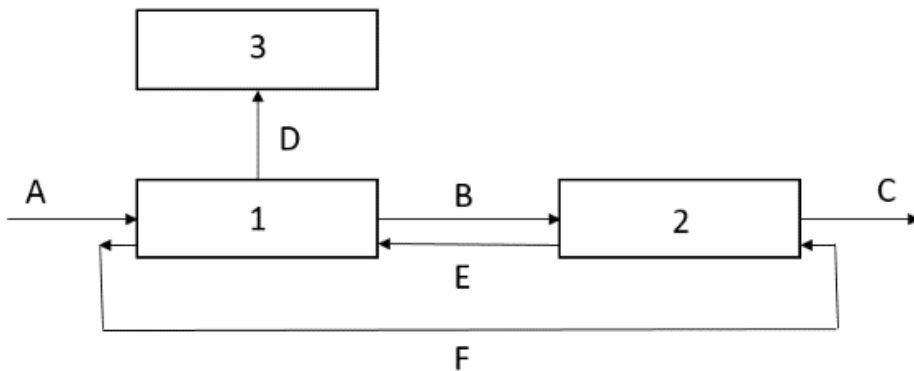


Fig. 6. Furfural residue drying-combustion integration system

1- paddle heat exchanger; 2- boiler; 3- exhaust gas disposal unit; A- wet furfural residue; B- dry furfural residue; C- ash; D- exhaust gas; E- steam; F- condensing water

4. Conclusion

The present work is a preliminary investigation that provides evidence for conceptual design of application systems. The aim is to check the feasibility of using single-shaft paddle heat exchanger to dry wet sugar cane bagasse furfural residue. Measurements of basic properties of furfural residue were performed. The average particle size is 0.174 mm, very different from that in literature. This shows that the particle size significantly depends on how furfural residue is generated. The lower heating value of furfural residue with

moisture content of 54.26% is positive which means that properly designed drying-combustion integration system does not consume net heat in furfural residue disposal processes. Pyrolysis curves were also documented for design need. Furfural residue flow rate and drying characteristics were tested in a lab-scale single-shaft paddle heat exchanger. No agglutination and flow blockage were found. The results confirm that it is feasible to dry wet furfural residue to moisture content of 15.94% with reasonable equipment design and working fluid parameters in the viewpoint of engineering applications. Compared with raw wet furfural residue, the product after drying can be burned more easily and efficiently. This conclusion provides basis for future design of drying-combustion integration system. More research will be performed in the future, e.g., in scale-up performance of furfural residue drying systems based on single-shaft paddle heat exchangers.

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