Optimization of Continuous Solid-State Distillation Process for Cost-Effective Bioethanol Production

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Abstract: To improve the efficiency of bioethanol production, an advanced process was required to extract ethanol from solid-state fermented feedstock. With regard to the characteristics of no fluidity of solid biomass, a continuous solid-state distillation (CSSD) column was designed with a proprietary rotary baffle structure and discharging system. To optimize the operation condition, fermented sweet sorghum bagasse was prepared as feedstock for a batch distillation experiment. The whole distillation time was divided into heating and extracting period which was influenced by loading height and steam flow rate simultaneously. A total of 16 experiments at four loading height and four steam flow rate levels were conducted, respectively. Referring to packing, rectifying column, mass, and heat transfer models of the solid-state distillation heating process were established on the basis of analyzing the size distribution of sweet sorghum bagasse. The specific heat capacity and thermal conductivity value of fermented sweet sorghum bagasse were tested and served to calculate the ethanol yielding point and concentration distribution in the packing. The extracting process is described as the ethanol desorption from porous media absorbent and the pseudo-first-order desorption dynamic model was verified by an experiment. Benefit (profit/time) was applied as objective function and solved by successive quadratic programming. The optimal solution of 398 mm loading height and 8.47 m³/h steam flow rate were obtained to guide a 4 m in diameter column design. One heating and two extracting trays with 400 mm effective height were stacked up in an industrial CSSD column. The steam mass flow rate of 0.5 t/h was determined in each tray and further optimized to half the amount on the third tray based on desorption equation.

Keywords: solid-state distillation; bioethanol production; sweet sorghum bagasse; heating time; extracting time; design factors optimization; industrial scale-up

1. Introduction

Nowadays, countries all over the world are focusing on renewable and sustainable energy in the face of the energy shortage and climate change [1]. Bioethanol is one of the promising alternatives to fossil fuel [2]. Liquid fermentation from juice of sugar or starchy crops is widely used in bioethanol production. However, the method suffers from the drawbacks of high energy consumption, high sugar loss, and producing wastewater [3]. Solid-state fermentation (SSF) has the advantages of no juice squeezing energy, limited sugar loss, and no additional water input, but has low efficiency due to its batch operation mode. In feedstock selection, sweet sorghum is considered as the most suitable crop for SSF [4–8]. Sweet sorghum is a type of sugar crop, harvested after 3 months, with highly efficient photosynthesis. It also holds considerable versatility, such as absorbing heavy metal from contaminated land or remediating arable land while simultaneously producing cost-effective bioethanol [9–11]. However, the spongy and medullary structure causes much higher
energy consumption of sweet sorghum than other crops such as sugarcane, and higher sugar loss in squeezing residue [12,13].

Against the disadvantages of batch fermentation, Li et al. [14] developed advanced solid-state fermentation (ASSF) technology to produce bioethanol with sweet sorghum stalks, changing the SSF process from batch-wise to continuous by using a rotary drum bioreactor. More recently, there has been systematic research on SSF and put it into industrial scale [15,16], a demonstration unit with two bioreactors of 55 m in length and 3.6 m in diameter has been successfully operating in Shandong Province, China [17].

With the gradual industrial application of ASSF, extracting ethanol from solid fermented product has attracted more attention. Zhou et al. [18] suggested planar thermal source as a heat source on fermented crushed straw material to separate ethanol in a laboratory scale study. However, because there was no free water in the SSF system, the planar heating method could consume more energy. The most efficient and commonly used method to extract ethanol from fermented biomass was direct steam heating and stripping, which originated from traditional Chinese liquor distillation [19]. To determine the influence factors of liquor distillation, Li et al. [20] carried out experiments on an industrial-scale distiller. It was found that the steam flow rate and bed height were the most important factors influencing the liquor yield and quality. The optimal steam flow rate and bed height were 1.60 kg/min and 1250 mm, respectively, which were determined through chemical and sensory analysis. The difference between fuel ethanol and liquor production was that fuel ethanol production focused on ethanol yielding rather than liquor flavor ingredients [21]. More attention could be paid to distillation efficiency and the recovery rate of ethanol. In addition, the particles used in bioethanol production were fiber-like and irregular, which were different from relatively regular grains in liquor distillation. Li et al. [22] conducted an experiment on a batch solid-state distillation (SSD) column, extracting ethanol from fermented sweet sorghum bagasse. The optimal steam flow rate and loading height was 8–10 kg/h and 700–1000 mm, respectively. However, this work suffered from inadequate research on the interaction between loading height and steam flow rate, since the results were obtained from a single-factor experiment. Further studies are needed to better understand the complex association between both factors and mass and heat transfer model in SSD.

Batch solid-state distillation holds the disadvantage of high energy consumption, low automation level, high labor intensity, and discontinuous procedure. Moreover, the output ethanol concentration in vapor of batch distillation is not stable, so it will not be directly imported into rectification unit without condensation, thereby increase the cost of bioethanol. Li et al. [23] designed a type of continuous solid-state distillation (CSSD) column used in ethanol extraction. With regard to the characteristic of poor fluidity of solid biomass, a proprietary rotary baffle structure and discharging system was suggested. Biomass was motivated by built-in machinery to form a similar mass and heat transfer process by stripping sections of packing rectifying column, whereas the design factors of CSSD column were mainly obtained from the experience of liquor distillation. Studies on CSSD column based on experimental data to enhance bioethanol production efficiency are required.

The objective of the present study was to explore a technology for ethanol extraction from fermented solid-state biomass with low energy consumption, large production capacity, and high output ethanol concentration. In this work, a cross-sectional study was undertaken to determine the combined influence of loading height and steam flow rate on ethanol extraction in SSD. By analyzing experimental data, the optimal results were obtained. CSSD column design factors were calculated and determined, conforming to the optimal results. These design factors could be the theoretical framework when the CSSD column further develops in large-scale industrial application.
2. Materials and Methods

2.1. Materials

2.1.1. Raw Material

The sweet sorghum, a cultivar named “LiaoTian No.1”, was harvested in Tongliao, Inner Mongolia Province, China, in October 2018. The growing period is 122 days. The sweet sorghum stalks were refrigerated in a laboratory. Before fermentation, the stalks with leaves were smashed by a pulverizer. The average total sugar content of the sweet sorghum bagasse was 13.01% (w/w), and the moisture content was 71.49% (w/w).

2.1.2. Particle Size Distribution of Material

The particle size distribution analysis of sweet sorghum bagasse could provide a basis for mass and heat transfer in the distillation process. The shape and size of particles could be acquired by analyzing high-definition photographs of monolayer bagasse against a black background with the particle recognition function of Image J software.

2.1.3. Solid-State Fermentation

Saccharomyces cerevisiae TSH-1 frozen with glycerol was used in this study [24]. Yeast cells were pre-cultured in YPD medium (10 g/L yeast extract, 20 g/L glucose, and 20 g/L peptone), incubated at 30 °C with an agitation rate of 200 rpm for 12 h. Then yeast was inoculated into 10 times secondary fermentation medium, incubated at the same temperature with 250 rpm for 6 h. After being washed with sterilized water, the cultivation of yeast was finished when OD_{600} = 10 was measured by spectrophotometer.

The sweet sorghum bagasse and yeast were mixed in a ratio of 10 to 1, and then loaded into a continuous solid fermentation reactor. The reactor was a 50 L rotary drum, driven by motor and gear and equipped with an automatic temperature-control system. After continuous solid-state fermentation for 20 h at 30 °C, the SSB was unloaded and frozen in a refrigerator as experimental materials for this research. The average ethanol content of fermented sweet sorghum bagasse (FSSB) was 6.0 ± 0.2%, analyzed by high performance liquid chromatography (HPLC). The moisture content was 73.4 ± 0.4%.

2.1.4. Measurement of Specific Heat Capacity and Thermal Conductivity

Thermodynamic parameters are an important factor for heat transfer calculation. In this study, a planar thermal source was used to determine the specific heat capacity of FSSB. The FSSB was put on a thermal source with a heat flow rate of 5 W for 30 s. Then the thermocouple average temperature and temperature rise of FSSB was recorded after heating. The specific heat capacity was calculated by the formulation:

$$ C_p = \frac{W \times t}{m \Delta T}, $$

where $W$ is the thermal source power (W), $t$ is the heating time (s), $m$ is the mass of FSSB (g), and $\Delta T$ is the temperature rise (K) recorded by thermocouple.

The thermal conductivity of FSSB was measured by thermal conductivity. Both tests were conducted at intervals of 20 K from 293 K to 373 K. Each test was repeated 3 times on average in the above-temperature range.

2.2. Experiment Methods

2.2.1. Experiment Apparatus

Since CSSD process is the expansion of SSD in time dimension, the design parameters of CSSD column can be determined by batch SSD experiments. Figure 1 illustrates the experiment flow path of...
SSD and experiment apparatus. The distillation column was made of stainless steel, with an inner diameter of 400 mm. The column assembled a conical head on the top and a steam distributor on the bottom. The distillation column and the steam chamber were separated by an 80-mesh screen sintered plate to prevent solid particles from falling into the steam chamber. Column height could be adjusted from 100 to 1200 mm by 100 mm, 150 mm, and 200 mm shell sections. Different combinations of shell sections were selected to adapt to different loading heights. PT100 thermometers were set at the top, bottom, and each section of the column. The sensors were connected to monitor the display and record the temperature changes during the distillation process.

A steam generator (9 kW) provided steam with a rated capacity of 12 kg/h and maximum pressure of 0.7 Mpa. The steam inlet of the column was equipped with a flow regulator, rotor steam flowmeter, and pressure gauge to measure and control the steam flow rate and pressure. A pressure gauge was mounted on the top of the column to monitor the pressure drop when the steam passed through the packing bed. The column outlet on the conical head was connected to the condenser, which was a shell and tube heat exchanger with a heat transferring area of 1 m². A sample connection was located at the condenser outlet. A collection tank was placed on an electronic balance with data storage function to record the collective mass per minute.

2.2.2. Experiment Procedure

The experiment was conducted by the following procedures. Firstly, assemble the distillation column to an intended height and load FSSB into the column until it is completely filled. Smooth the top surface with a scraper. Ensure that no additional stress is applied to FSSB during loading. The natural bulk density of FSSB was measured simultaneously during the loading process. It equals the mass of loading material divided by the volume of the column. The average value of 16 times measurement was 233.7 kg/m³. Secondly, assemble the conical head on the surface and check the sealing performance. Then, connect vapor the outlet to the condenser, keeping the vent open until the first stream of vapor comes out of the column. Switch on the steam generator until the outlet pressure is stable. Regulate the steam flow rate to the intended value and saturation state at atmospheric pressure. Start the timer when the steam inlet regulator is turned on. The steam distributes through the screen sintered plate and initiates interaction with FSSB. Close the vent when the vapor flows out and turn on the condenser cooling water at the same time. The condensate was collected in a flask and accumulated mass was recorded. Distillate concentration was tested by refractometer on sample connection per minute. When the alcohol degree is less than 1% (v/v), turn off the steam valve and stop the timer. The accumulated distillate is then stored for further accurate analysis by HPLC.
2.2.3. Experiment Arrangement

The SSD period is mainly influenced by the interaction of loading height and steam flow rate while steam temperature and ethanol contents of FSSB are stable. It appears that larger steam flow and lower loading height could result in shorter distillation time, but this causes lower production efficiency and more energy consumption at the same time. To determine the optimum production condition of CSSD, distillation time at different steam flow rates and loading height should be investigated. A total of 16 experiments were designed at loading height levels of 250 mm, 400 mm, 550 mm, and 700 mm, and steam flow rate levels of 7.2 m$^3$/h, 10.4 m$^3$/h, 13.6 m$^3$/h, and 16.8 m$^3$/h, respectively, for CSSD simulation.

2.3. Analytical Methods

2.3.1. Moisture Content Analysis

The moisture content of FSSB was determined by measuring the weight loss before and after the sample was dried in the oven at 378 ± 2 K for 5 h.

2.3.2. Ethanol Concentration Analysis

Ethanol in FSSB and vinasse was extracted by a homogenizing method [25] after mixing well, then 20.0 g of materials and 180.0 g of sterilized water were accurately weighed by balance and blended in a homogenizer for 5 min. The cell wall of sweet sorghum was broken while blending. The supernatant was separated from the mixture by an ultrasonic scrubber, then centrifuged at 10,000 rpm for 5 min. It was diluted to a certain amount, and filtered with 0.45 nm membrane and sent to HPLC for analysis. The distillate was diluted and filtered with the same method. Bio-Rad Aminex HPX-87H ion-exchange column and a differential refractive index detector for RID-20A (Shimadzu, Kyoto, Japan) was used in HPLC. The column temperature was 40 °C, and 0.05 mol/L sulfuric acid was prepared as the mobile phase. The flow rate was 0.6 mL/min. The ethanol content was tested by the external standard method.

2.3.3. Other Chemical Components Analysis

HPLC was used to analyze the contents of sugars and organic acids in FSSB simultaneously with the ethanol sample. The chemical composition, except ethanol, in the distillate was tested with high-resolution gas chromatography by China national research institute of food and fermentation industry (CNIF).

3. Results and Discussion

3.1. Experiments Results

3.1.1. Particle Size Distribution

According to the average data of five groups of samples analyzed by Image J software, it was found that the amount of fibrous particles with dimension less than 1 × 10 mm (diameter × length) was the largest. The second largest amount was the particle size smaller than 1.5 × 15 mm. As shown in Figure 2b, the two types of particles accounted for 93.48% of the bagasse, and the rest of the sizes were less distributed. Therefore, in this research, the material can be simplified to a mixture composed of the particles of 1 × 10 mm (67.49%) and 1.5 × 15 mm (25.99%) in the mass and heat transfer model.
Figure 2. Particles of sweet sorghum bagasse: (a) Photograph of sweet sorghum bagasse; (b) Shape and particle size distribution.

3.1.2. Specific Heat Capacity and Thermal Conductivity

The test results of specific heat capacity and thermal conductivity in the temperature range from 293 K to 373 K are listed in Table 1. The specific heat capacity of absolutely dry sweet sorghum stalk can be estimated by subtracting the proportion of water and ethanol, which is less than the literature data of intact corn stalks [26] due to air being mixed between the smashed particles, which is consistent with the actual distillation process. Figure 3 shows the relationship between temperature and specific heat capacity or thermal conductivity. The curves were fitted with a 3 order polynomial and regressed with Equations of (2) and (3). The $R^2$ was 0.9957 and 0.9971, respectively.

\[
C_p = 3.9383 - 0.0046712 T + 8.5714 \times 10^{-6} T^2,
\]

\[
\lambda = -1.1020 + 0.0076702 T - 1.0179 \times 10^{-5} T^2.
\]

Figure 3. Relationship between temperature and specific heat capacity or thermal conductivity.
Table 1. Specific heat capacity and thermal conductivity in different temperature of FSSB.

| Temperature (K) | Mean Specific Heat Capacity (kJ/kg·K) | Thermal Conductivity w/(m·K) |
|----------------|--------------------------------------|----------------------------|
| 293            | 3.306                                | 0.272                      |
| 313            | 3.318                                | 0.304                      |
| 333            | 3.332                                | 0.325                      |
| 353            | 3.360                                | 0.337                      |
| 373            | 3.389                                | 0.345                      |

3.1.3. Chemical Components in FSSB and Distillate

The sugar and organic acid content in FSSB was tested by HPLC with an external standard method in the sample. The theoretical yield for conversion of glucose to ethanol was 0.511 (g/g). The total residual sugar in FSSB is 12.6 g/L and an ethanol conversion efficiency of 90.25% was obtained at SSF condition, which was higher than 87.33% of liquid fermentation [27]. The content of organic acid is significantly lower than that in the classic fermentation process, especially the acetic acid content, which was only 40 mg/L, and was normally above 100 mg/L in liquid fermentation. The result indicates that the saccharomyces cerevisiae TSH-1 and SSF technology were more suitable for bio-ethanol production.

Since the boiling point of organic acid is higher than the distillation temperature, there was no organic acid volatilized in the distillate. The harmful substances of methanol in the distillate was only 44 mg/L, which was far less than China’s national standard for edible alcohol of 150 mg/L [28]. The flavor volatile of acetaldehyde was 469.5 mg/L, which could match the content of commercial Fen-flavor liquor. However, the aroma components such as ethyl acetate were only 95 mg/L, significantly less than 800–1700 mg/L in commercial liquor. This was mainly caused by the difference of fermentation in the raw materials and the function of saccharomyces cerevisiae. The esters production was also related to fermentation time. A short fermentation period was also the reason for esters insufficiency. However, the distillate can be used as a base liquor for food purposes, with a higher economic value added and evaluated by CNIF.

3.1.4. Distillation Experiments Results

Distillation time was divided into heating time ($t_H$) and extracting time ($t_E$) by yielding point. The initial yielding ethanol concentration ($E_Y$) was determined by the first minute sample analyzed by HPLC. The ethanol recovery ($R_c$) was calculated by dividing the ethanol amount in the distillate by ethanol amount ($E_c$) in the FSSB. The region where the temperature equals the saturated vapor temperature at yielding point was defined as lean concentration region. The height of lean concentration region ($H_L$) was recorded by thermometer at different locations of the column. The pseudo-first-order kinetic rate ($k_1$) was calculated based on the data of ethanol amount per minute tested by the refractometer and balance. The lean concentration region and pseudo-first-order kinetic rate will be discussed in the following sections.

3.2. Solid-State Distillation Process

3.2.1. Mass Transfer Model

Distillation is a mass transfer process in which the volatility difference of each component is utilized to separate the liquid mixture. When the liquid or liquid–solid mixture was heated to boiling point, more volatile components were contained in the vapor than in the mixture and non-volatile components were retained in the liquid phase, so that the mixture can be partly or completely separated.

Solid-state distillation is also a process that utilizes the volatility difference of each component to realize the separation of different components. The difference between SSD and liquid distillation is that the volatile components are normally adsorbed in the porous fiber-like medium in the SSD process. There is no free water in the medium, therefore, the steam is both the heat carrier and desorption fluid in SSD.
In this study, the ethanol absorbed in FSSB was desorbed and concentrated by continuous upward steam. The SSD column can be seen as a special packed column. FSSB were not only the material containing ethanol components, but also the packing with a large specific surface area. Therefore, the packing bed can be divided into innumerable thin layers perpendicular to the steam flow. It is assumed that the physical properties of the thin layer are uniform. The steam contacted with the bottom FSSB layers at first, and vaporized ethanol flowed through the gas film to the surface of the fibrous particles, then was stripped by steam and flowed into the upper layer. Ethanol condensed at the upper low temperature layer to increase the ethanol concentration. The continuous steam stream vaporized ethanol and condensed upward again. The ethanol concentration of vapor in column free space achieves the vapor–liquid equilibrium with the surface FSSB layer. The condensate could dilute the ethanol concentration in FSSB to some extent. However, considering that the ratio of heating vapor to FSSB is normally less than 0.1, and it occupies the non-free water space only, the excess condensate will fall back to the steam chamber after heating. Therefore, the influence of heating vapor dilution was not considered in this study. After several processes of vaporization, condensation, and concentration, the ethanol content reaches a maximum at the surface layer. Then it discharges from the outlet and is condensed in a heat exchanger. As the steam continuously flows in, the ethanol in the column gradually is separated from FSSB until the concentration at the outlet is close to zero. When the liquor yields, the process of SSD turns into unsteady state due to there being no raw material supplement in the batch process. The ethanol concentration in distillate gradually decreased as the ethanol content reduced in the column.

3.2.2. Ethanol Yielding Point

In the SSD process, the concentration of ethanol increases with the vapor rise. When the height of the packing bed is infinite, the theoretical ethanol yielding point could reach the azeotropic point of ethanol and water, which is \( y^* = 89.4\% \) in molarity. Whereas the actual maximum yielding point is much lower than this value. The reason can be revealed by operation line method.

It was still assumed that the packing bed of column could be divided into infinite thin layers. FSSB thin layer holding one mole liquid can be seen as a mass transfer unit. \( x_f \) is the ethanol molarity of FSSB. The liquid in layers with solid material was heated to a boiling point and required mole saturated steam. \( y_{n-1} \) is the ethanol molarity of condensed steam and \( x_n \) is the ethanol molarity after vapor–liquid is mixed. The material balance was calculated as follows:

\[
(1 + v)x_n = vy_{n-1} + 1 \times x_f, \tag{4}
\]

The equation of operation line obtained by transform Equation (4) to

\[
y_{n-1} = \frac{1 + v}{v}x_n + \frac{1}{v}x_f, \tag{5}
\]

The condensing heat of the rising steam vaporizes ethanol and water, and also raised the temperature of the solid phase to the boiling point of the liquid. Therefore, the amount of heating steam \( v \) can be calculated as follows:

\[
v = \frac{1}{w_{W}^{m-1} + w_{E}^{m-1}} \int_{T_0}^{T_B} C_{p,FSSB} dT + \left( w_{W}^{m} Q_{W} + w_{E}^{m} Q_{E} \right) \times \frac{M_W}{(1 - x_f)M_W + x_f M_E} \tag{6}
\]

The ambient temperature \( T_0 \) is 25 °C, and the inner pressure of column remains at atmosphere pressure due to the vent being connected with air when heating. The boiling point of ethanol solution at morality concentration of FSSB \( T_B \) is 93.0 °C. \( \omega \) is the mass fraction of ethanol or water. The corresponding ethanol mass fraction in equilibrium vapor phase \( w_{E}^{m-1} = 0.42 \). The specific heat capacity can be calculated according to Equation (2). Including the ethanol and water vaporization...
heat value of $T_B$ which is $Q_W = 2275.1 \text{ kJ/kg}$ and $Q_E = 808.8 \text{ kJ/kg}$, respectively, the heating steam capacity can be calculated as the result of $v = 1.374$.

When the ethanol concentration in FSSB is constant, the intersection point between the operation line starts from $x_f$ and the vapor–liquid equilibrium curve is the highest molarity that can be achieved by SSD, as shown in Figure 4.

![Figure 4. The number of theoretical plate determination by graphic method.](image)

When heating vapor flow rate $v = 1.374$ and the initial ethanol molarity $x_f = 0.031$, the steps were drawn on the figure by a method similar to the graphical calculation of theoretical rectifying plates. The number of theoretical plates required $N$ was 3. The corresponding gas phase molarity is about $y_n = 0.53$ (Mass concentration $m_{wn} = 0.74$), which was regarded as the highest ethanol yielding point.

The theoretical packing height can be determined by the height equivalent to theoretical plate (HETP) method. For fiber-like packing, HETP has no empirical data and formula for reference, but can be estimated according to the thermal conductivity. While the steam passed through the theoretical plate, the FSSB was heated by the heat of steam condensation. It was considered that before the lowest theoretical plate boiling, the steam would not transfer upward. So the theoretical plate temperatures of upper and lower surface are $T_0$ and $T_B$, respectively, while mass transferring on the theoretical plate. According to the definition of thermal conductivity, the theoretical plate height can be calculated as:

$$HETP = \frac{E}{\int_{T_0}^{T_B} \lambda dT} = \frac{V\rho_v \left[ Q_W + 0.5m_{wn} \left( Q_E - Q_W \right) \right]}{3600 \int_{T_0}^{T_B} \lambda dT}$$ \hspace{1cm} (7)

where $E$ is the steam heat flow rate which gradually decreases in the process of steam rising along with mass transfer. The average heat flow rate of theoretical packing could be approximately treated as half the sum of the highest and lowest layer. As the steam flow rate $V = 10.4 \text{ m}^3/\text{h}$, the saturated steam density $\rho_v = 0.597 \text{ kg/m}^3$, then the calculated value of HETP was 0.147 m, and height of the theoretical packing was $N \times HETP = 0.441$ m.

While the height of FSSB exceeds the theoretical packing height, the concentration of ethanol could not increase with steam rising due to the limitation of the operation line. In other words, a certain height of high ethanol concentration region exists above the theoretical packing height.

During the heating period, the front of high concentration region moves upward continuously. When it reaches the surface, the vapor–liquid equilibrium is achieved between the free space at the top of the column and the liquid phase of the FSSB topmost layer. During the movement of the high concentration front, the concentration of the lower layer gradually approaches zero as the ethanol
content increases in the upper layer, forming a lean concentration region. A transition region exists between the lean and high concentration regions.

It can be calculated that when the steam flow rate \( V = 10.4 \text{ m}^3/\text{h} \), the theoretical packing height was 441 mm. When the loading height is higher than theoretical packing height, a high concentration region would appear, and the initial yielding ethanol concentration could reach the theoretical value. While the loading height is less than 441 mm, the yielding ethanol concentration could decrease.

In order to obtain the ethanol concentration data of different heights at yielding point, the thermometers were set at every 100 mm up from the bottom of the column, as shown in Figure 5a. As the liquor yielding from the column, all FSSB below the top layer reached the bubbling point. The measuring temperature of each layer could speculate the ethanol concentration accordingly.

![Figure 5. Ethanol concentration distribution at yielding point (a) photograph of thermometer location; (b) Ethanol concentration along 250 mm loading height \( (V = 10.4 \text{ m}^3/\text{h}) \) at yielding point; (c) Ethanol concentration along 400 mm loading height \( (V = 10.4 \text{ m}^3/\text{h}) \) at yielding point; (d) Ethanol concentration along 550 mm loading height \( (V = 10.4 \text{ m}^3/\text{h}) \) at yielding point; (e) Ethanol concentration along 700 mm loading height \( (V = 10.4 \text{ m}^3/\text{h}) \) at yielding point.](image)

Figure 5b–e show the concentration distribution at different loading heights at ethanol yielding point when the steam flow rate \( V = 10.4 \text{ m}^3/\text{h} \). Further analysis reveals that the height of the theoretical packing layer is closely aligned with the calculated results. The height of the lean concentration region...
is proportional to the heating time, indicating that under a certain amount of steam, the rising speed of the lean concentration front is constant. When the loading height was lower than theoretical packing height, a concentration gradient was formed above the lean concentration region. Assuming that the gradient is constant, the mass concentration of the liquid phase in the surface layer could be calculated based on the conservation of ethanol mass, as follows:

\[ m_{yn} = 2m_f \frac{H}{(1 - H_L)} \]  

(8)

where \( H_L \) is the height of the lean concentration region (Table 2). The temperature close to 100 °C indicates that the ethanol concentration was close to zero. When \( V = 10.4 \text{ m}^3/\text{h}, H = 400 \text{ mm}, \) it could be seen from Figure 5b that \( H_L \) accounted for about 25% of the total loading height, so the mass concentration of ethanol in the upper surface can be estimated as \( m_{yn} = 21.6\% \) based on Equation (8).

| No. | \( H \) mm | \( V \) m³/h | \( Ec \) % (w/w) | \( t_H \) min | \( t_E \) min | \( R_c \) % (w/w) | \( k_1 \) min⁻¹ | \( H_L \) mm | \( E_Y \) % (v/v) |
|-----|------------|--------------|----------------|---------|---------|---------------|------------|-------|-------------|
| 1   | 250        | 7.2          | 6.1            | 12.5    | 30.9    | 95.8          | 0.102      | 100   | 67.4        |
| 2   | 250        | 10.4         | 6.1            | 9.6     | 22.3    | 96.7          | 0.152      | 75    | 66.8        |
| 3   | 250        | 13.6         | 5.9            | 8.9     | 11.4    | 95.7          | 0.276      | 60    | 61.6        |
| 4   | 250        | 16.8         | 6.0            | 7.7     | 8.9     | 95.7          | 0.341      | 60    | 57.4        |
| 5   | 400        | 7.2          | 6.0            | 17.2    | 34.4    | 96.6          | 0.098      | 135   | 75.4        |
| 6   | 400        | 10.4         | 5.9            | 13.3    | 19.3    | 97.1          | 0.183      | 100   | 67.3        |
| 7   | 400        | 13.6         | 6.2            | 11.2    | 13.6    | 96.8          | 0.253      | 80    | 65.4        |
| 8   | 400        | 16.8         | 6.0            | 10.5    | 9.7     | 97.3          | 0.372      | 80    | 63.0        |
| 9   | 550        | 7.2          | 6.1            | 20.6    | 48.0    | 96.7          | 0.079      | 150   | 77.9        |
| 10  | 550        | 10.4         | 6.1            | 16.1    | 28.8    | 95.7          | 0.109      | 120   | 74.8        |
| 11  | 550        | 13.6         | 5.9            | 12.8    | 15.8    | 96.8          | 0.219      | 100   | 66.7        |
| 12  | 550        | 16.8         | 6.2            | 11.4    | 11.6    | 96.2          | 0.282      | 90    | 62.8        |
| 13  | 700        | 7.2          | 5.8            | 24.7    | 76.8    | 95.8          | 0.041      | 180   | 78.4        |
| 14  | 700        | 10.4         | 6.2            | 19.0    | 46.8    | 97.3          | 0.077      | 140   | 77.1        |
| 15  | 700        | 13.6         | 6.0            | 16.0    | 23.7    | 96.9          | 0.147      | 120   | 76.2        |
| 16  | 700        | 16.8         | 6.2            | 14.4    | 15.9    | 96.0          | 0.202      | 100   | 76.5        |

As the binary system of ethanol–water exists great negative deviation to Raoult’s law, ethanol concentration in the vapor phase calculated by the relative volatility method in the low concentration range would produce large errors. According to the data of vapor–liquid equilibrium in the range of 0~36 wt% [29], the relationship between \( m_{yi} \) and \( m_{xi} \) under fourth-order regression analysis is:

\[ m_{yi} = 0.02314 + 9.1408m_{xi} - 58.227m_{xi}^2 + 178.479m_{xi}^3 - 200.611m_{xi}^4 \]  

(9)

where \( R^2 \) was 0.9946. The calculated alcohol yielding concentration of vapor was 69.5% (v/v) based on the above equation. The error with the experimental result was 3.1%.

When the loading height was higher than the theoretical packing height, the initial ethanol yielding concentration was lower than the calculation, which could be caused by insufficient heating in the high concentration region while ethanol was yielding. The condensed ethanol concentration would increase in the first few minutes.

The conclusion can be drawn from the theoretical analysis and experimental results that the initial ethanol yielding concentration was only related to the loading height and steam flow rate under certain ethanol content in FSSB. The steam flow rate determined the theoretical packing height. When the loading height was higher than theoretical packing height, the yielding ethanol concentration could reach the maximum. The lower steam flow rate and higher loading height might cause the ethanol concentration to have increased liquor outcomes but lower production efficiency.
3.2.3. Ethanol Extracting Kinetics Analysis

The ethanol was discharged from the distillation column and condensed in a cooler, which was equivalent to a theoretical plate. The distilled ethanol concentration \( x_0 \) equals the vapor at the free space, which can be tested by refractometer per minute.

As shown in Figure 6a, the curve of ethanol extracting concentration was relatively flat at the beginning, which was due to the gradual movement of high concentration front, resulting in the ethanol content in the top layer of FSSB changing a little. When the high concentration front reached the top layer, the ethanol extracting concentration began to decrease significantly. When \( x_0 < 1\%\) (v/v), the ethanol content of top FSSB layer was less than 0.1% (v/v). It is considered that most of ethanol in FSSB was extracted because all the layers under the top were the lean concentration region. When distillation continued, the ethanol yielding rate was low, but it also reduced product concentration and increased energy consumption. So the experiment stopped when \( x_0 \) less than 1% (v/v). The analysis on residual ethanol content in vinasse indicates that the ethanol recovery rate at the end of distillation in different experiments range between 95.8 and 97.6%, with an average recovery rate of 96.6% (Table 2).

**Figure 6.** Ethanol concentration change with extracting time; (a) Ethanol extracting concentration in different steam flow rate; (b) Extracting time comparison between FSSB and solution.

FSSB is a fiber-like porous media. According to particle size distribution analysis, the specific surface area of FSSB was up to 1.39 m\(^2\)/g. Ethanol was adsorbed in FSSB particles or between the lapping channels. So the SSD is distinguished from liquid batch distillation. The time required for batch liquid distillation to extract ethanol from saturate 8.1 wt% ethanol aqueous and achieve the recovery rate of 96.6% can be calculated by Equation (10)

\[
M_L \frac{dx}{dt} = -V \rho_y (m_y - m_{y_0})
\]

There is no ethanol in the vapor, \( m_{y_0} = 0 \); when \( T = 0 \), \( m_s = m_f \); where \( m_y \) and \( m_s \) is in accordance with vapor-liquid equilibrium (11), \( M_L \) is the mass of saturated ethanol aqueous solution, the same as the amount of FSSB liquid holding. When \( V = 10.4\) m\(^3\)/h, \( H = 400\) mm, \( m_s = 0.966 m_f \), for example, solving the differential equation obtained result of extracting time was 32.8 min, which is more than the 19.3 min timed in the SSD process, as shown in Figure 6b. The reason for this is that FSSB provided a huge packing surface and a concentration gradient had been formed in the heating period. The upper layer equilibrium had a higher ethanol concentration vapor in SSD, enabling the ethanol extracting process to be more efficient.

When the whole liquid holding in feedstock reaches a saturated state, the steam will no longer condense during rising. A constant vapor flow is formed in the packing bed and extracted ethanol is continuously extracted from each FFSB layer to increase the concentration. Therefore, the ethanol extracting process is similar to desorption ethanol from the fiber-like adsorbent. The steam is used as
a carrier gas, which provides the gas phase that is unbalanced with the aborted solution. Therefore, the extracting process can be described by desorption kinetics equation.

The concentration of ethanol at the outlet of the condenser is the same as vapor on the top free space of the column, which can be tested by a refractometer. The concentration multiplied by the liquor yielding amount is the desorbed ethanol mass per minute. The equilibrium absorption amount \(m_e\) is considered as the ethanol content per kilogram in FSSB as yielding. The desorbed amount \(m_t\) changed over time and the residual ethanol adsorption amount in FSSB was \(m_e - m_t\). Pseudo first order, second order, and internal diffusion dynamics model were applied to fit \(m_e\) and \(m_e - m_t\). It turned out that the pseudo first order kinetics model was the most suitable to describe the ethanol desorption process from FSSB. The average coefficient index \(R^2\) was 0.976, the closest to 1 in all three models. The pseudo first order equation is as follows:

\[
k_1t_E = \ln m_e - \ln m_t
\] (11)

where \(k_1\) is the pseudo-first-order rate constant (min\(^{-1}\)), and \(t_E\) is the extracting time. The desorption kinetic constant \(k_1\) was inconsistent under different experimental conditions (Table 2). The steam flow rate mainly affects the ethanol diffusion inside the fiber-like medium. Ethanol was not uniformly distributed in FSSB as yielding time, but produced a concentration gradient on loading height, which is relative to the desorption rate. The combined effect of steam flow rate and loading height on the distillation rate can be described by the extracting time.

3.3. Optimization of Solid-State Distillation Process

3.3.1. Total Distillation Time

As discussed in the previous section, the total distillation time can be divided into heating time and extracting time. There is no heat and mass flowing out in the heating process. All the heat of steam condensation is used to heat FSSB. The average temperature after heating is assumed as \(T_m\), the heat balance of the distillation column as adiabatic system is calculated as follows:

\[
Vt_HQ_E = \frac{1}{4} \pi D_0^2 H \rho_{FSSB} \int_{T_0}^{T_a} C_p dT + Vt_H(T_{WB} - T_m)
\] (12)

Only \(T_m\) is related to \(H\) and \(V\) among all parameters, so \(t_H\) can be simplified into the formation of (13):

\[
t_H = a \frac{H}{V} + b
\] (13)

According to the linear fitting of the test data, the relationship between \(t_H\) and \(H/V\) is obtained with \(R^2 = 0.9916\) as follows:

\[
t_H = 0.2041 \frac{H}{V} + 5.2152
\] (14)

Mass and heat transfer have become unsteady in the extracting process. Although the process can be described as first-order desorption kinetic model, the kinetics constants \(k_1\) and desorption mass flow \(m_t\) were also related to \(H\) and \(V\). The correlation between extracting time and \(H, V\) is also established by polynomial fitting based on experiment data, \(R^2\) is 0.9865:

\[
t_E = 66.2278 + 0.01641H - 7.099V + 1.3861 \times 10^{-4}H^2 + 0.3033V^2 - 0.00803HV.
\] (15)

The fitting results were displayed in Figure 7.
where $P$ be calculated by:

$$4010 = 2260, 6270$$

The average recovery rate of ethanol is 96.6%, so $P = 0.966 \times 0.06 \times F$, and the vanisse amount can be calculated by:

$$W = F - P + V t$$

It can be seen from the above discussion that the distillation time is mainly related to the loading height $H$ and steam flow rate $V$. The higher loading height is, the larger product capacity per unit time and the higher the concentration of ethanol output. However, the heating time and extracting time are both prolonged, and the production efficiency is reduced. The larger steam flow rate causes the shorter distillation time. However, when a large amount of steam is condensed in the heating period, the ethanol concentration in FSSB would be reduced to affect the ethanol concentration of product. In addition, the utilities consumption and production cost will be increased.

In order to optimize the production conditions, benefit $B$ (profit per minute) is selected as the optimization objective function. The prices of raw materials, utilities, and products are shown in Table 3. The objective function is defined as follows:

$$\max B = p_1 P + p_3 W - p_2 F - p_4 V \frac{F_0}{60}$$

where $P$ is the ethanol product in distillation process, $W$ is the by-product of vanisse that can be processed into feed, $F$ is the raw materials such as sweet sorghum, yeast, and enzyme, the subsequent industrial cost plus average profit of purifying ~25% to fuel ethanol is 4010 CNY per ton of absolute ethanol.

### Table 3. The price of raw materials, utilities, and products.

| Item                                | Unit         | Price/CNY $^1$ |
|-------------------------------------|--------------|----------------|
| Fuel ethanol $^2$                    | t            | 6270           |
| Sweet sorghum $^2$                  | t            | 324.8          |
| Yeast and Culture medium $^2$       | per ton FSSB | 11.5           |
| Steam ($p_4$)                       | t            | 190            |
| Vinasse ($p_3$)                     | t            | 320.7          |
| Charges from 25 wt% to absolute ethanol $^2,3$ | t            | 4010           |

$^1$ The price is according to cost accounting report of Dongying sweet sorghum ethanol project. $^2 p_1 = 6270 - 4010 = 2260, p_2 = 324.8 + 11.5 = 336.3$. $^3$ The average cost of utilities consumption, equipment, labor, etc.

The average recovery rate of ethanol is 96.6%, so $P = 0.966 \times 0.06 \times F$, and the vanisse amount can be calculated by:

$$W = F - P + V t$$
which assumes the steam condensed in FSSB occurring in heating time only. So objective function was transform into Equation (17).

The diameter of existing CSSD column is 4 m, which is 100 times larger than the laboratory condition, and loading height is less than 1500 mm under motor power limitation. The maximum steam boiler capacity is 14 t/h, converted to the experimental steam flow rate of 234 m$^3$/h. So the constraint conditions are determined as following:

$$ \begin{align*}
\max B &= \frac{2.91H + 1.67V}{7.14 + 0.0164H - 7.10V + 1.39 \times 10^{-4}H^2 + 0.303V^2 - 0.0803HV + 0.204V^2 - 0.189V} \\
0 < H &\leq 1500, \ 0 < V \leq 234
\end{align*} \quad (18)
$$

The locally optimal solver in MATLAB software was used to resolve the constrained non-linear programming problem by successive quadratic programming (SQP). Initially $(H, V) = (0, 0)$, the results converged to the local minimum at $(H, V) = (398, 8.47)$. Although more local optimal solutions with different initial values were compared under constraint conditions, the first solution lead to the maximum benefit value and was acceptable for practical problems.

Considering the price fluctuation of raw materials and products, we adjusted the price coefficient in the objective function to observe the change of optimization results. It can be seen that when the cost of raw materials fluctuates by 10%, the change range of optimization parameters is within 4%. When the steam price changes, for example, replacing a nature gas boiler with a biomass fuel boiler, the unit price of steam reduces to 144 CNY/t, $H = 409.7$ mm and $V = 9.11$ m$^3$/h are the optimal solutions. It is concluded that the influence of raw material and steam price on the optimization results are limited.

When the solid-state distillation column is designed as a 4 m diameter on site, 400 mm is the most optimal height of the tray as the design basis in engineering.

### 3.4. Continuous Solid-State Distillation

Conventionally, the ethanol in fermented solid-state bagasse were extracted by batch distillation, which suffer from some disadvantages such as low production efficiency, high energy consumption, fluctuating ethanol concentration and labor intensive. Continuous solid-state distillation (CSSD) is a new technology to solve the bottleneck in ethanol production.

The CSSD column, shown in Figure 8, consisted of three trays with the function of preheating, distillation, and discharging from top to bottom. The feedstock was loaded from the top of column by a screw conveyor and was uniformly distributed on the first tray. The motor with a reducer at the bottom drove the blades on shaft rotating, pushing the feedstock in the tray, moving one cycle and dropping into the next tray through an appropriate discharge port near the inlet. After heating and distillation, the vinasse was pushed by baffles to outlets and unloaded. Part of the vinasse could be treated as solid fuel used in a biomass fuel boiler to generate low-pressure steam for distillation, another part as cattle feed. The heating process is similar to the rectification operation, but the difference is that in CSSD there is no descending fluid formed by the reflux condenser from the upper to lower tray. So, the ethanol concentration of each tray gradually decreases during the CSSD process.

The saturated steam passed through the header pipe and entered the distributors under each tray to heat biomass or extract ethanol. The steam counter-current contacted with biomass on trays for heat and mass transferring. FSSB was heated to the boiling point of ethanol in the first tray and then turned into the next tray. Ethanol was stripped on this tray and discharged through the tube with steam. Each tray was equipped with an independent steam distributor to avoid the vapor contacting between the two trays that could reduce the ethanol concentration in the upper tray or the vapor could be condensed again. The vapor in tubes came together into the outlet on the top of column and was further purified in the subsequent separation unit.

CSSD process can be regarded as the expansion of batch distillation process in time dimension. Its mass and heat transfer model is consistent with batch distillation perpendicular to the direction of steam. Therefore, the experiment results can be used to guide the design of a CSSD column.
When the steam flow rate of both extracting trays is the same, where,

\[ n \]

\[ F \] vapor in the process of rise will dilute the whole product. It is considered that lowering the third tray is less than the second, half of it, for example, \( \frac{1}{2} \) steam of the first tray. After the process optimization, the external supply of steam consumption was reduced to 0.75 t/h.

Equation (20):

\[
\begin{align*}
\frac{dm_1}{dt} &= -k_1 m_1 \\
\frac{dm_2}{dt} &= -k_1 m_2
\end{align*}
\]  

where, \( k_1 = 0.132 \) is based on the batch distillation experiment result. According to initial concentration and concentration continuity condition, the change curve of ethanol in the CSSD process can be obtained by solving the differential equations as shown in Figure 9. It can be seen that the ethanol concentration in the second tray drops rapidly from 8.1 wt% to 1.14% when FSSB flows out at the discharge gap. Hence, the low concentration stripping vapor from the third tray mixed with the upper vapor in the process of rise will dilute the whole product. It is considered that lowering the third tray’s steam amount will increase the ethanol concentration in distillate, which can be calculated by differential Equations of (19), namely:

\[
\begin{align*}
\frac{dm_1}{dt} &= -k_1 m_1 \\
\frac{dm_2}{dt} &= -k_1 m_2
\end{align*}
\]  

On the basis of optimization results, the design loading height (H) was 400 mm, and the steam mass flow rate of each layer (V_m) was 0.5 t/h. The corresponding heating time (t_H) is 15.1 min and extracting time (t_Y) is 25.8 min. The extracting time is about 1.7 times the heating time. Considering 5 min unloading time, two extracting trays and one heating tray can be set and rotate under 4 rph CSSD column shaft speed.

In the extracting period, FSSB in the trays rotates under the shaft force and the concentration changes at each point on the tray circumference. However, the heat and mass transfer in the vertical direction is the same as the batch condition. As discussed above, since the extracting process conforms to the pseudo-first-order desorption equation, the variation of ethanol content in the liquid phase of the FSSB in two distillation trays can be expressed by differential Equations of (19), namely:

\[
\begin{align*}
\frac{dm_1}{dt} &= -k_1 m_1 \\
\frac{dm_2}{dt} &= -k_1 m_2
\end{align*}
\]  

where, \( k_1 = 0.132 \) is based on the batch distillation experiment result. According to initial concentration and concentration continuity condition, the change curve of ethanol in the CSSD process can be obtained by solving the differential equations as shown in Figure 9. It can be seen that the ethanol concentration in the second tray drops rapidly from 8.1 wt% to 1.14% when FSSB flows out at the discharge gap. Hence, the low concentration stripping vapor from the third tray mixed with the upper vapor in the process of rise will dilute the whole product. It is considered that lowering the third tray’s steam amount will increase the ethanol concentration in distillate, which can be calculated by Equation (20):

\[
m_Y = \frac{F \times m_0 \times R_C}{V_M \times n + F \times m_0 \times R_C}
\]  

where, \( F \) is the FSSB capacity of column, \( R_C \) is ethanol recovery rate, and \( n \) is the steam flow coefficient. When the steam flow rate of both extracting trays is the same, \( n \) is 2. When the flow rate of the third tray is less than the second, half of it, for example, \( n \) is 1.5. The ethanol recovery rate when \( n = 1.5 \) still exceed 95%, whereas the distillate ethanol concentration can be increased from 21.4 wt% to 26.3 wt%, saving more steam energy consumption and reducing the subsequent purification operation difficulty at the same time. In order to further reduce energy consumption and waste water discharge, the sufficient residue of rectifying column from subsequent process can be reheated as the heating steam of the first tray. After the process optimization, the external supply of steam consumption was reduced to 0.75 t/h.

Figure 8. Structure of continuous solid-state distillation (CSSD) column.
was simulated by the flash evaporation module FLASH2 of ASPEN software. The flash temperature while solid-state fermentation refers to the literature [14]. The energy consumption of liquid distillation process are shown in Tables 4 and 5, respectively. The liquid state fermentation refers to the literature [30], which is the squeezing process. The steam required for juice concentration process was counted in the energy consumption of solid-state fermentation.

When the steam flow rate of both extracting trays is the same, the 2nd tray consumes more steam than the 3rd tray. After the process optimization, the external supply of steam consumption was reduced to 0.75 t/h. The engineering data of CSSD column could be modified as per an industrial scale, based on optimization results appropriately. The design of construction drawings and selection of the steam boiler, motor, and reducer were based on the above factors.

In addition to steam consumption, CSSD also requires electricity to drive the shaft and blades rotating and the operation loading screw conveyor. Driving the rotation of the blades mainly requires overcoming the viscous resistance and inertia force of FSSB and bearing friction force. The calculated shaft power was 8.15 kW and the corresponding motor output power was 10.47 kW. The motor power for conveyer was 5.63 kW, and the steam consumption was 1.23 tons. All energy consumption in this analysis was based on 1 ton crude ethanol of 26.3% mass concentration.

3.5. Economics Analysis

In order to verify the advantages of ASSF and CSSD, we conducted an economic comparison of ethanol production with sorghum stalks using the classic liquid state fermentation and solid-state fermentation.

For classic liquid state fermentation, the electricity consumption in the liquid process mainly comes from the squeezing juice process, while the juice clarification, filtration, and concentration process require steam consumption and produces a large amount of biological wastewater. Approximately 5% sugar loss in squeezing causes more feedstock amounts of liquid state fermentation than solid state fermentation. The material and energy consumption of the solid and liquid bioethanol production process are shown in Tables 4 and 5, respectively. The liquid state fermentation refers to the literature [30], while solid-state fermentation refers to the literature [14]. The energy consumption of liquid distillation was simulated by the flash evaporation module FLASH2 of ASPEN software. The flash temperature was 96.7 °C. The feeding ethanol concentration was 8.9 wt%, and the corresponding sugar content was 20.2%. The steam required for juice concentration process was counted in the energy consumption of the squeezing process.

Figure 9. The decline curve of ethanol concentration with rotating time in different column tray. (a) The same steam flow rate in 2nd and 3rd tray; (b) The 3rd tray steam flow rate is half of the 2nd tray.
Table 4. Materials balance sheet of solid and liquid process based on 1 ton 26.3 wt% crude ethanol.

| Process                  | Input (ton) | Material                | Output (ton) |
|--------------------------|-------------|-------------------------|--------------|
| Solid-state process      |             |                         |              |
| Smash                    | Sweet sorghum | 4.23                   | Bagasse 4.23 |
| Seed culture             | Water       | 0.62                    | seed liquid 0.67 |
| - Barley malt            | 0.04        | -                       | -            |
| - Yeast and Enzymes      | 0.01        | -                       | -            |
| Fermentation             | Bagasse     | 4.23                    | FSSB 4.62    |
| - Seed liquid            | 0.67        | Carbon dioxide 0.28     |
| Distillation             | FSSB        | 4.62                    | Crude ethanol 1 |
| - Vapor                  | 1.23        | Vinasse 4.85            |
| Liquid-state process     |             |                         |              |
| Squeeze                  | Sweet sorghum | 4.54                   | Juice 4.03   |
| - Water                  | 1.75        | Vinasse 1.91            |
| - Lime                   | 0.01        | Wastewater 0.36         |
| Seed culture             | Water and Nutrient salt | 0.05 | Seed liquid 0.06 |
| - Yeast and Enzymes      | 0.01        | -                       | -            |
| Fermentation             | Juice       | 4.03                    | Fermented liquid 3.87 |
| - Seed liquid            | 0.06        | Carbon dioxide 0.22     |
| Distillation             | Fermented liquid | 3.87       | Crude ethanol 1 |
| - -                      | -           | Wastewater 2.87         |

Table 5. Energy input sheet of solid and liquid process based on 1 ton 26.3 wt% crude ethanol.

| Process                  | Energy Input (kWh) |
|--------------------------|--------------------|
| Solid-state process      |                    |
| Smash                    | Electricity 26.71   |
| Seed culture             | Electricity 5.67    |
| - Steam                  | 17.6               |
| Fermentation             | Electricity 20.7    |
| - Steam                  | 15.84              |
| Distillation             | Electricity 771.42  |
| - Steam                  | 857.94             |
| Liquid-state process     |                    |
| Squeeze                  | Electricity 54.64   |
| - Steam                  | 386.34             |
| Seed culture             | Electricity 3.79    |
| - Steam                  | 6.18               |
| Fermentation             | Electricity 35.38   |
| - Steam                  | 420.05             |
| Distillation             | Steam 906.38       |

As is shown in Table 4, the energy input of solid state process is less with the same energy output, while the consumption of high-grade electricity energy is only 73.4% of that of the liquid state process. Moreover, ASSF and CSSD technologies are eco-friendly processes because of the minimum biological wastewater production, which are worth promoting and applying for wastewater treatment cost saving. Last but not least, CSSD outputs more boiled vinasse that can be processed to cattle feed with higher quality and more health.

Economic analysis for producing 1 ton 99.5% fuel ethanol from sweet sorghum stalks by using solid state process is displayed in Table 6. The unit price and operation cost are according to cost accounting report of Dongying sweet sorghum ethanol project with production capacity of 10 kilotons per year. The rectification and dehydration units consumes 2.4 tons steam and 81 kWh electricity energy. The cost of 1 ton of 99.5% ethanol with the use of solid state technology is 3828 CNY, much less than the cost of 5835 CNY with liquid state method [30]. (The exchange rate of RMB to USD as 1 to 7). It could make a profit of 2442 CNY per ton as purchase price of Chinese fuel ethanol. These results show that it is economically feasible to produce ethanol from sweet sorghum stems by using continuous solid state fermentation and distillation technologies.
Table 6. Economic analysis of 1 ton 99.5% fuel ethanol with ASSF and CSSD process.

| Item            | Amount | Unit Price/CNY | Cost/CNY |
|-----------------|--------|----------------|----------|
| Feedstock       | 16 tons| 324.8          | −5196.8  |
| Seed culture    | -      | -              | −184     |
| Storage         | -      | -              | −164.8   |
| Utility         | Electricity | 261+81 kWh | 0.8      | −272.8   |
| Steam           | 2.89+2.4 tons | 190          | −1005.1  |
| Water           | 3 tons | 5              | −15      |
| Operation       | Labor and Management | - | - | −646.3 |
| Depreciation and Amortization | - | - | −1515 |
| Maintenance     | -      | -              | −697     |
| Income          | Cattle feed | 18.3 tons | 320.7    | 5868.8   |
| Fuel ethanol    | 1 ton  | 6270           | 6270     |
| Profit          | -      | -              | 2442     |

1 Using sulfur dioxide for storage [24]. 2 The latter value is the consumption in rectification and dehydration to extract ethanol from 26.3 wt% to 99.5 wt%. 3 1.87 tons residue of rectifying column are recovered as heating steam for CSSD process. 4 The depreciation life is 10 years, and the salvage rate is 5%. 5 The maintenance cost is calculated at 5% of the fixed asset.

4. Conclusions

The optimum CSSD column design factors were obtained through the FSSB batch distillation experiments and optimizing calculations. The whole distillation process was divided into two stages of heating and extracting. The mass and heat transfer model of the SSD heating process was similar with the packing rectifying column on the basis of analyzing the size distribution of sweet sorghum bagasse. The specific heat capacity and thermal conductivity data measured in practice were used to calculate the theoretical solid distillation packing height and ethanol concentration distribution in the packing at yielding point. The relative error between calculated and experimental results was 3.1%.

The extraction process is described as the ethanol desorption from porous media absorbent. The mass transfer model indicated that heating and extracting time both were mainly influenced by loading height and steam flow. Using regression analysis on experimental results could establish a relationship of loading height and steam flow rate with whole distillation time. Solving the objective function with SQP method could obtain the optimal solution as a loading height of 398 mm and a steam flow rate of 8.47 m³/h.

According to the optimization results, a CSSD column with a diameter of 4 m was designed. One heating tray and two extracting trays with the 400 mm effective height were stacked up in the column. The steam mass flow rate was 0.5 t/h in each tray and was further optimized to half that amount on the third tray based on the pseudo-first-order desorption equation.

The heat and mass transfer model of SSD was established for the first time, and the optimized loading height and steam flow rate experiment value were obtained in a scaled-up CSSD column, which is suitable for large-scale industrial bioethanol or distilled spirit production, improving the production efficiency of traditional batch distillation. Combined with ASSF, the energy consumption of bioethanol production can be reduced comparing with liquid state process. The wastewater generation could be diminished to nearly zero.

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