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Extraction of bioethanol from fermented sweet sorghum bagasse by batch distillation

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Abstract−Extraction of bioethanol, a potential alternative to fossil fuel in the transport industry, from sweet sorghum stems [*Sorghum bicolor* (L.) Moench] using solid-state fermentation (SSF) technology has become a popular research topic worldwide. Because SSF technology can directly convert fermentable sugars into target products without juice squeezing and water input, this method can potentially reduce energy and water consumption. However, ethanol extraction from fermented sweet sorghum bagasse requires further investigation. We used batch solid-state distillation to investigate the optimal operating parameters in a distillation column (diameter, 400 mm) via a single-factor experiment. Results showed that the optimal steam flow rate and loading height were 8-10 $kg\cdot h^{-1}$ and 700-1,000 mm, respectively. Under optimal conditions, an energy consumption of 3.82 tons of steam per ton of ethanol and distillate concentration of 60.9% (v/v) were obtained. The pseudo-first-order rate equation was used to describe the distillation kinetics, and good correlations were obtained. Therefore, solid-state distillation can be effectively used to extract ethanol from fermented sweet sorghum bagasse.

Keywords: Ethanol Extraction, Sweet Sorghum Bagasse, Batch Solid-state Distillation, Condition Optimizing, Pseudo-first-order Rate Equation

INTRODUCTION

Biofuels can potentially solve the shortage of fossil fuels caused by continuous depletion of natural resources and rapid industrial development [1-3]. Bioethanol production from sweet sorghum stems using advanced solid-state fermentation (ASSF) technology has been proven to be relatively cost-effective [4]. Sweet sorghum stems are used as a feedstock in ASSF because sweet sorghum is a rapid-growing C_4 crop that can be planted in tropical, subtropical, and temperate zones, marginal lands, and even alkaline and saline lands [5-7]. The plant is a non-food crop with a rich germplasm resource, high biomass yield, rapid growth, wide adaptability, and rich sugar content in stems [8-10]. Solid-state fermentation has the advantages of no squeezing process required, which saves energy, nearly no wastewater produced, which saves on equipment investments, limited sugar loss, and high product yields [11].

Two of the most important challenges in extracting ethanol from fermented sweet sorghum bagasse (FSSB) are the poor fluidity of the feedstock and its low ethanol concentration. Ethanol production from FSSB occurs in the solid-state and the product shows extremely irregular fiber-like particles with different diameters and lengths. Therefore, the classical distillation method [12], which is widely used to separate liquid systems in the chemical industry, cannot be used for FSSB. In ethanol-water system, classical distillation method will be a better separation method [13] and many novel methods, such as diffusion distillation [14,15], heat pump assisted distillation [16] and so on, are also developed. However, in the solid-ethanol-water system, there is now free water in it. The most efficient method of extracting ethanol from FSSB involves direct heating with vapor; this method can be referred to as the Chinese liquor distillation system [17]. These methods all employ solid-vapor systems. Whereas solid-state particles in the liquor system present in the form of grains and are regular, those produced in the FSSB distillation system only appear fiber-like and are relatively irregular. Therefore, further studies in this area remain an urgent necessity.

Several liquor distillation systems have been widely studied. Tang et al. [18] found that both steam flow rate and loading height affected the distillation yield of fermented grains. The optimal steam flow and loading height in a distiller with a diameter of 1,000 mm were found to be 96 $kg\cdot h^{-1}$ and 1,000 mm, respectively. Nie et al. [19] analyzed the effect of temperature distribution in the distiller on distillate quality and yield during distillation of fermented grains named Nongxiang Baijiu (liquor); in this work, the heating rate decreased progressively in a circular fashion from the distiller edge to its center. Liu et al. [20] investigated the effects of various distillation conditions, including different distiller heights, and found that loading height affected the distillation yield during distillation of luzhou-flavor liquor from mature solid-state fermented grains. Li et al. [21,22] investigated the optimal operating conditions and mass transfer model in batch distillation of Chinese liquor from fermented grains; a film theory model that agreed well with the industrial-scale experimental data was subsequently proposed and developed. Du et al. [23] established a batch distillation model and explained the mechanism of Chinese liquor distillation. Yuan [24]

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theoretically investigated the distribution and moving pattern of alcohol together with other flavor ingredients during grain distillation and proposed mathematical models to describe the distillation process.

The FSSB distillation system has also been studied extensively. Chen et al. [25] studied bioethanol extraction from sweet sorghum stems by using a gas-stripping method during solid-state fermentation. The optimal initial gas-stripping time, gas-stripping temperature, fermentation time, and particle thickness were 10 h, 308 K, 28 h, and 15 mm, respectively, and the corresponding ethanol recovery was 77.5%. Zhang et al. [26] designed a novel equipment of cellulose fuel ethanol distiller to meet the requirements of liquid and solid distillation, and found that the optimal extraction conditions of steam pressure, loading coefficient, and reflux ratio were 0.3 Mpa, 0.7, and 0.7, respectively; in this work, the maximum ethanol recovery was 78.9%.

Several studies on FSSB distillation systems have been performed [27], but research on the influence of steam flow rate and loading height on distillation efficiency and vapor consumption is rarely reported. Studies on a kinetic extraction model that suitably describes the FSSB distillation system are also needed. The present study aims to address these research gaps by investigating the influence of steam flow rate and loading height on the energy consumption and distillate concentration of a batch solid-state distillation (BSSD) system. A kinetic model that can describe the distillation process rules is also determined.

MATERIAL AND METHODS

1. Material

Sweet sorghum, named "Energy & Feed 1#" (i.e., "Nengsi 1#" in Chinese), was harvested in Hebei Province, China, in November 2014. The sweet sorghum stems were stored in natural conditions outside the laboratory. Together with leaves and husks, the stems were smashed into particles 1-5 mm in diameter and 3-50 mm in length by a pulverizer before fermentation. Solid-state fermentation of the sweet sorghum stems was conducted following the methods presented by Li [4] and Du [28] et al.. Solid-state FSSB, as shown in Fig. 1, was fed into the BSSD system immediately after fermentation. The ethanol concentration and moisture content of the FSSB were determined to be 6.6±0.4 wt% and 73.5±1.0 wt%, respectively.

Fig. 1. Photograph of fermented sweet sorghum bagasse (FSSB).

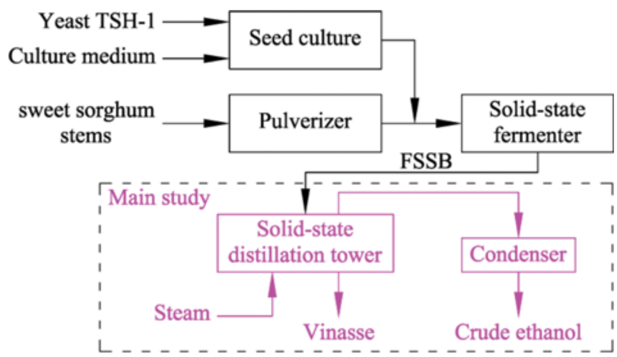

Fig. 2. Flowsheet of ethanol extraction process from sweet sorghum stems to crude ethanol.

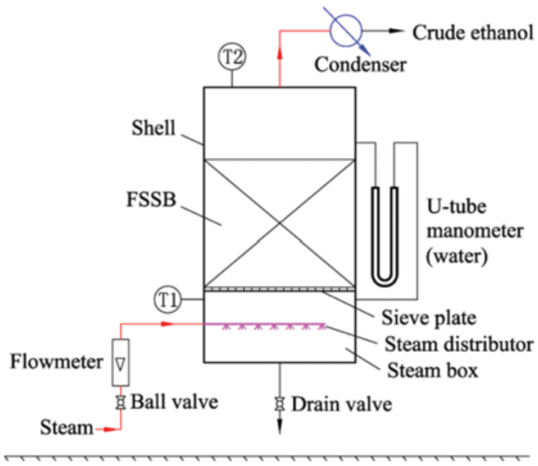

Fig. 3. Structure diagram of distillation tower and the BSSD process flowsheet.

2. Apparatus and Procedure

To have a complete picture of the process, a complete flowsheet of the process is shown in Fig. 2. The complete process include FSSB preparation introduced in 2.1, and solid-state distillation, which is the main study.

The main BSSD apparatus was a stainless-steel distillation tower with an inner diameter of 400 mm. The height of the tower can be changed from 100 mm to 1,500 mm by combining various sections with different lengths. Fig. 3 shows the structure of distillation tower and the BSSD process flowsheet.

In the BSSD process, steam with a gauge pressure of 0.3 MPa flows through the flowmeter and is distributed into the steam box by the distributor. Steam then enters the FSSB packing bed through the sieve plate and initiates direct heat and mass transfer with the FSSB. When the temperature of FSSB reaches the bubbling point of the liquid it holds, ethanol begins to volatilize into the vapor phase but condenses upon contact with the upper portion of the FSSB, which features a temperature lower than that of ethanol. Given continuous steam inputs, ethanol is volatilized continuously, and this volatilization and condensation process occurs repeatedly until the

Table 1. Parameters of flowmeter, U-tube manometer and thermometer

Name	Range	Accuracy	Function	Manufacturer
Vortex flow meter	1.5-15 $kg\cdot h^{-1}$	1.5	Detect and adjust steam flow	XinruiStrong Technology (Beijing) Co., Ltd.
U-tube manometer (water)	0-1000 mm	0.1	Detect pressure drop of FSSB bed	Wuqiang country Jingda meter plant essence of instrument
Thermometer (PT100)	−100-450 °C	0.2	Detect temperature of steam and ethanol vapor	Kunlun Beijing Industrial Control Technology Development Co., Ltd.

vapor leaves the FSSB bed and reaches the condenser. Finally, in the condenser, the vapor is condensed to form a distillate product.

The BSSD system is equiped with one steam flowmeter, one U-tube manometer (water) and two thermometers. The parameters are listed in Table 1. One thermoneter (T1), located in steam box, is used to detect the temperature of steam put into the FSSB bed. Another thermometer (T2), located in the top of the distillation tower, is used to detect the temperature of ethanol vapor left the FSSB bed.

The BSSD procedure is described as follows:

a) The BSSD system is connected correctly and preheated in advance.

b) The FSSB is loaded into the distillation tower without pressure or vibration until the intended loading height and smooth surface are achieved.

c) When the steam valve is turned on, the timer begins. When the distillate flows out of the condenser, it is collected at different time periods. These times are recorded.

d) When the distillate concentration is very close to 0.0% (v/v), as tested by an alcohol refractometer, the steam valve is turned off. The concentration of the distillate samples is accurately analyzed by gas chromatography.

3. Experimental Method

The FSSB is a kind of solid-state irregular fiber-like material with multiholes and with different size. The moisture content in FSSB is about 74% w/w, and the water-holding capacity can reach up to 80% w/w. BSSD is a kind of unsteady state operation. Therefore, the reflux ratio, the number of stages and hydraulics are not designed. This has referred to similar systems. Steam flow and loading height are the most significant influence factors on distillation in the similar systems [18,20,22,23,26,29]. Furthermore, the composition and content of FSSB are almost fixed. Thus, we investigated the influence of steam flow and loading height by using the single factor method.

In the steam flow influence experiment, steam flows ranged from 3.5 $kg\cdot h^{-1}$ to 15.0 $kg\cdot h^{-1}$. The loading height (500 mm) and other parameters were invariant. In the loading height influence experiment, loading heights ranged from 300 mm to 1,500 mm. The steam flow (the steam flow value is going to be optimized from steam flow influence experiment) and other parameters were invariant. The distillation efficiency was assessed by energy consumption and total distillate concentration. Each experiment was repeated by 3-5 times.

4. Extraction Model

The distillation process of ethanol desorbed from the FSSB can be regarded as a desorption process. The pseudo-first-order rate equation is one of the most widely used kinetic models and often applied to correlate the adsorption kinetics curve [30,31]:

$$\ln(q_e - q_t) = \ln q_e - k_1 t'$$

where q_t ($g\cdot kg^{-1}$, i.e., g ethanol per kg FSSB) is the desorption amount at a given time t' (min), q_e is the equilibrium desorption amount, k_1 is the pseudo-first-order rate constant (min^{-1}), and t' (min) is the distillation time; here, t=0 is defined as the moment the distillate flows out of the condenser.

5. Analytical Methods

5-1. Moisture Content

The moisture content of FSSB was determined as the difference in weight (loss) measured before and after the sample was dried in the oven at 378±2 K for about 3 h [32].

5-2. Ethanol Concentration

Ethanol in FSSB was first extracted by placing 50 g of FSSB with 200 mL of distilled water into a 500 mL round-bottomed flask for distillation, which yielded 100 mL of distillate. Ethanol concentration was determined [4] by a gas chromatograph (SHIMADZU GC-14C) equipped with a flame ionization detector and an SS column (0.125 cm i.d., 2 m). Nitrogen gas and hydrogen gas were used as flaming and carrier gases, respectively. The injector temperature was 353 K and the detector temperature was 493 K. The running time was 18 min. Propanol was applied as the internal standard.

Energy consumption (EC) was calculated as:

$$EC = \frac{F t_{0.2}}{60 C_D V_D}$$

where EC (t) is the amount of steam based on 1 ton of absolute ethanol, F ($kg\cdot h^{-1}$) is the steam flow, $t_{0.2}$ (min) is the distillation time required to decrease the ethanol concentration in the vinasse to 0.2%, and C_D (g/mL) and V_D (L) are the total distillate concentration and total distillate volume at time $t_{0.2}$, respectively. Because $t_{0.2}$ could not be directly obtained from the experimental data, it was calculated via the interpolation method using the corresponding data of distillation time and ethanol concentration in the vinasse.

Ethanol recovery (ER) was calculated as:

$$ER = \frac{C_D V_D}{C_i m} \times 100\%$$

where C_i (%, kg ethanol per 100 kg FSSB) is the initial ethanol concentration in FSSB and m (kg) is the initial weight of the loaded FSSB.

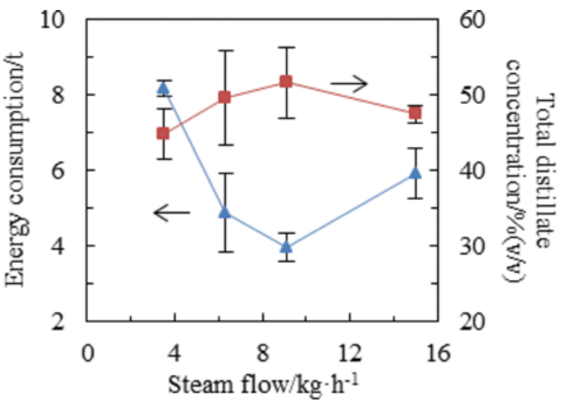

Fig. 4. The effect of steam flow on energy consumption and total distillate concentration.

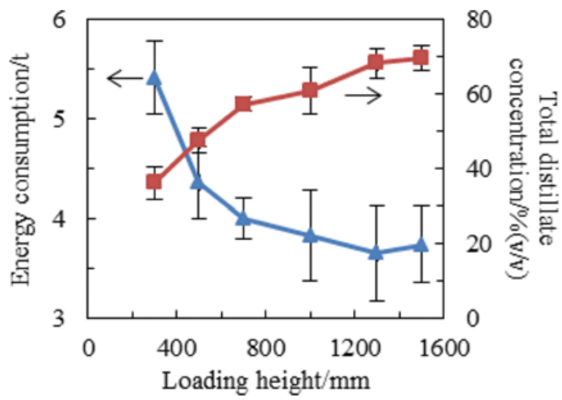

Fig. 5. The effect of loading height on energy consumption and total distillate concentration.

RESULTS AND DISCUSSION

1. Effects of Steam Flow Rate

Steam flow is one of the most critical parameters in distillation. The effect of steam flows ranging from 3.5 kg·h^{-1} to 15.0 kg·h^{-1} at a loading height of 500 mm (Table 2) on energy consumption and total distillate concentration was investigated. Fig. 4 shows the results (with standard deviation error bars).

Energy consumption initially decreased and then increased with increasing steam flow. The total distillate concentration showed the opposite results. The lowest energy consumption and the highest total distillate concentration were obtained when the steam flow was approximately 9 kg·h^{-1}, likely because longer distillation times (and more heat loss) are needed in the presence of lower steam flows.

Furthermore, the steam is changed into water by heat loss and stays in the packing, causing the moisture content to increase and the ethanol concentration to decrease accordingly. Thus, the distillate concentration decreased according to the vapor-liquid equilibrium. As the steam flow increased, the distillation time decreased and heat loss was reduced, leading to an increase in total distillate concentration. In general, not all of the steam molecules are utilized to conduct mass and heat transfer because the mass and transfer rates are not as equal to the rate at which steam flows over the packing bed. This unutilized steam is condensed into the distillate and decreases the distillate concentration. As such, a steam flow in the range of 8-10 kg·h^{-1} is recommended.

2. Effects of Loading Height

Loading height is another important operating parameter during distillation. The effect of loading heights ranging from 300 mm to 1,500 mm with a steam flow of 8.1±0.4 kg·h^{-1} (Table 2) was investigated and the results are shown in Fig. 5.

Energy consumption decreased and total distillate concentration increased with increasing loading height. This variation tendency intensified at heights less than 700 mm and weakened at heights exceeding 1,000 mm. FSSB can be considered as a type of packing material in classical distillation. Classical distillation is operated in a continuous and steady-state manner, and increasing the loading height of the packing could improve the distillate concentration. However, the present BSSD system is conducted in batch mode and no reflux, which can improve the distillate concentration during classical distillation, is observed in the BSSD system. Consequently, the distillate concentration cannot be continuously improved and is limited by the vapor-liquid equilibrium. Therefore, the distillate concentration shows gradual changes with increasing loading height. This finding supports the rationale behind the use of loading heights of 700-1,000 mm in traditional liquor distillation [29].

The packing density and pressure drop increased with increasing loading height (Fig. 6). Increasing the packing density will in-

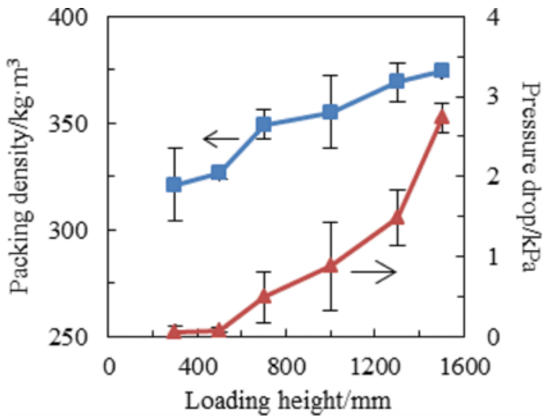

Fig. 6. The effect of loading height on packing density and pressure drop.

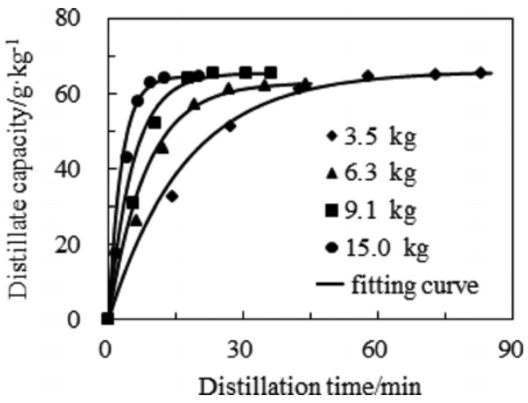

Fig. 7. The effect of steam flow on distillation kinetics.

Table 2. Fitting parameters of distillation kinetics in different steam flow and loading height by using pseudo-first-order rate equation

H/mm	F/kg·h^{-1}	C_i/% (v/v)	$t_{0.2}$/min	C_d/% (v/v)	EC/t	ER/%	k_1/min^{-1}	R^2
300	8.2	6.8	27.8	36.1	5.42	97.0	0.181	0.992
700	8.4	7.0	31.9	47.5	4.36	96.8	0.099	0.968
1000	8.2	6.5	45.6	57.2	4.00	96.8	0.064	0.952
1300	7.7	6.6	74.5	60.9	3.82	97.1	0.050	0.942
500	3.5	6.5	90.1	44.8	8.18	96.9	0.059	0.981
500	6.3	6.3	48.8	49.7	4.88	96.8	0.120	0.973
500	9.1	6.6	32.3	51.7	3.97	96.9	0.185	0.940
500	15.0	6.5	18.3	47.5	5.93	96.8	0.352	0.952

Note: C_d is the total distillate concentration with the unit of % (v/v)

crease the resistance the steam must overcome to pass through the FSSB packing, which, in turn, will require more steam and increase the pressure drop. Thus, the energy consumption of the system cannot be decreased continuously with increasing loading height.

3. Kinetic Parameters of Extraction

Fig. 7 displays the effect of steam flow on the distillation kinetics of the present system. Table 2 lists the fitting parameters.

The distillation time decreased and the distillation rate increased with increasing steam flow. This finding was supported by the data of distillation time and first-order rate constants listed in Table 2.

Fig. 8 displays the influence of loading height on the distillation kinetics of the present system; Table 2 also lists the relevant fitting parameters. The distillation time increased and distillation rate decreased with increasing loading height; k_1 also decreased with increasing loading height.

Table 2 shows that the average R^2 is 0.962, which means the pseudo-first-order rate equation can suitably describe the distillation kinetics in the present system. Based on the ethanol content in the vinasse, the average ER was 96.9 wt%.

The model of pseudo-first-order rate equation suitably used in this study is similar to the model presented by Li et al. (Li's model) [21]. Their research objects are FSSB and grains, respectively. The parameter of q_e in this study means equilibrium desorption amount. It was calculated by iteration method. But in Li's model, q_e means the initial amount of ethanol. It was detected. When the latter was used in this study, the R^2 was lower. Thus, the calculatin of q_e by using iteration method is relatively suitable. Maybe that is because the FSSB has more holes and more specific surface area than grains.

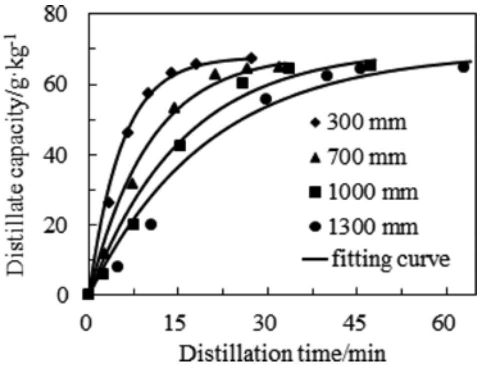

Fig. 8. The effect of loading height on distillation kinetics.

4. Economic Analysis

Economic analysis of producing 1 ton absolute ethanol from sweet sorghum stems by using solid-state fermentation and BSSD was listed in Table 3. The ethanol yield is about 1,500 tons per year and the calculation was based on the demonstration study [4] and a new program underway in the east of China. One part of the vinasse was used to generate steam, which was just enough for the ethanol production. The left part was used as animal feed, which could make a profit about $20 per ton vinasse [4].

Table 3 presents that the cost of 1 ton ethanol production is $697.9,

Table 3. Economic analysis of producing 1 t absolute ethanol from sweet sorghum stems

Item	Amount	Unit price/$	Cost/$
Feedstock			
Sweet sorghum stems[a]	16 tons	30	480
Transport[b]	16 tons	2.13	34.08
Storage[c]	16 tons	5	80
Utility			
Electricity	332 kW·h	0.1	33.2
Steam[d]	3.97+1.5 tons	8	43.76
Water	4 tons	1	4
Yeast & Enzymes			26.5
Labor[e]			20.09
Maintenance			20
Depreciation			64.3
Management			18
Finance			30
Vinasse	7.8 tons	−20	−156
Total			697.93

[a]The price of 1 ton sweet sorghum stems is about $30 [4]
[b]Biomass load and transport cost is $1.10 and $1.03 per ton ethanol, respectively. The average transport distance is 10 km and the cost is $0.103 per km [33]
[c]Using SO_2 for storage, the cost is around $ 5 per ton ethanol [28]
[d]1.5 t steam is used to extract ethanol from 60.9% (v/v) to 99.5% (v/v) by using extractive distillation method in the device of rotating packed bed (internal data)
[e]Refered to ABF Economics report [34]. The other cost refered to Du et al. [28]

which is cost competitive compared with that of corn-based ethanol ($841.7), cassava-based ethanol ($778.1) and wheat-based ethanol ($869.9) [35]. These results showed that it is economically viable to produce ethanol from sweet sorghum stems by using solid-state fermentation and BSSD method. As shown in Table 3, the feedstock of sweet sorghum stems costs the most, which accounts for 66.1% of the total cost. Therefore, to reduce the feedstock cost, the planting structure and management are suggested to be optimized; the high yield sweet sorghum seed and lower cost land (Such as saline-alkali land) are suggested to be developed.

CONCLUSIONS

Ethanol extraction from FSSB by BSSD was investigated. The operating conditions were optimized and the distillation kinetics was simulated. The results show that:

a) The optimal steam flow range is 8.0-10.0 $kg\cdot h^{-1}$; under this condition, the energy consumption, distillate concentration, and ethanol recovery were 3.97 t, 51.7% (v/v), and 96.9%, respectively.

b) Higher loading heights can reduce the energy consumption of the system and increase the distillate concentration. This variation tendency was intensified at heights less than 700 mm and weakened at heights exceeding 1,000 mm. Therefore, a loading height of 700-1,000 mm is recommended. Under this condition, the energy consumption and distillate concentration range from 4.36 t to 4.00 t and from 47.5% (v/v) to 57.2% (v/v), respectively; ethanol recovery at 700-1,000 mm is 96.8%.

c) The distillation kinetics of the studied system fits the pseudo-first-order equation well. This result is useful for applications in industrial design.

One valuable point of this study is that, fundamentally, it provides important and useful data supporting our industrial demonstration study. It is meaningful to enhance the technology. The other valuable point is that the mass transfer model in Chinese spirits (from grains) distillation was revised by using pseudo-first-order rate equation, which was suitable to BSSD. The main difference is that the feedstock of the most published works on the subject is grains, and not only the quantity but also the steam consumption is considered as a indicator in this study to evaluate the distillation efficiency.

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