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Research on Control System of Sequential Simulated Moving Bed Chromatography based on PLC and King View

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Abstract

The unit control of solenoid valve, pump and mass flowmeter instead of centralized control in sequential simulated moving bed chromatography(SSMB) for purification of xylitol mother liquor resulted in a low efficiency and other disadvantages. Therefore, a new SSMB separation system was developed based on PLC as controller and SSMB operation process as controlled object, adopting King View to design control screen, in order to achieve the control of industrial production process. Temperature, pressure, flow and mass concentration in separation process were input by measuring the analog connected with PLC and the results were output to an actuator following data processing and corresponding control operations. This paper described the compositions of the system and the realization of related functions including the control function. The creative system was validated by purifying xylitol mother liquor using SSMB and the excellent consequences revealed that the control system of SSMB based on PLC and King View had a positive effect on ensuring safe production and increasing production due to the suitable running temperature and simple operation and may have a realistic significance for improving modernization.

Keywords: *PLC, King View, Sequential simulated moving bed chromatography, Control system, Xylitol mother liquor*[1]

1. Introduction

Simulated moving bed chromatography (SMB) is a new type of separation technology, which can realize steady and continuous absorption separation so as to improve utilization rate of adsorbents, increase material handling and product purity[1]. Sequential Simulated Moving Bed chromatography (SSMB) is a significant innovation based on traditional SMB. The back mixing of substances in the reactor is avoided due to the efficient, continuous industrial separation and simple operation. SSMB has characteristics of good separation property, high recovery, superior efficiency, low cost and excellent stability. However, there are still some problems in the control of SSMB at present in our country, such as the absence of centralized control of solenoid valve, pump and mass flowmeter and low efficiency. Considering the

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background, the control system of SSMB based on PLC and King View was designed in order to enrich independent intellectual property rights and improve the stability and efficiency in production of purified sugar alcohols by SSMB in our country.

2. Materials and methods

2.1 Materials and instruments

Xylitol mother liquor, Shandong Longlive Bio-Technology. Strongly acidic cation ZG106Ca2+, Hangzhou Honor Resin. Preparative chromatography system, SMB-12E 1.2 L and SSMB-6Z6L, National Coarse Cereals Engineering Research Center. High performance liquid chromatography (HPLC)1200s, Agilent Technologies. Sacchari-meter WYT, Chengdu Haochuang Photoelectric Instrument.

2.2 Evaluation test for preparative chromatography

Preparative chromatography column was rinsed with deionized water, and the test conditions were set as following: column temperature 60℃, feed concentration 60%, feed amount 10 mL, flow rate 1.6 mL/min, and deionized water as the desorption agent. Sample were collected every 2 min, and the concentration was determined with WYT sugar meter, and then the purity of xylitol was determined by HPLC. The elution curve of xylitol motherliquid was drawn by the tube number as the abscissa and dry matter content as the ordinate.

2.3 Separation process of xylitol

The reliability of independent designed control system of SSMB was verified by separating xylitol mother liquor continuously for 10 h according to the purity and yield of xylitol. In the purification process, all the six chromatographic columns (35×1000 mm) in the system were under three steps successively, including Large cycle(S1), Small cycle(S2) and All in-out (S3). Consequently, eighteen procedures had to be taken in one complete operating cycle. Large cycle started from Column 1 and materials were only to be cycled without in and out. Small cycle was from Column 1 input desorbent D to Column 5 output hybrid sugar alcohols BD. All in-out was realized by adding desorbent D to the top of Column 1 and releasing xylitol AD from the bottom of Column 1 and then adding materials F to the top of Column 4 and releasing arabinitol BD from the bottom of Column 5. Hereafter transplanting to Column 2 and all the input and output of materials were moved to the next column and so on. The process flow of SSMB was shown in Figure 1 .

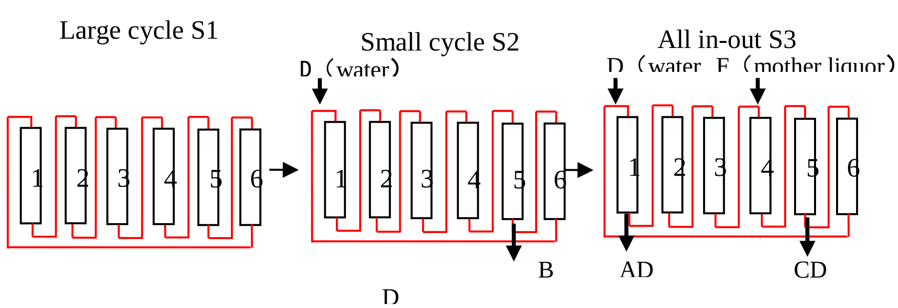

Figure 1. Process flow diagram of SSMB

Based on the evaluation test of preparative chromatography and the principle of material balance and SSMB, the process parameters in purification of xylitol mother liquor by SSMB were optimized according to the purity and yield of obtained xylitol so as to achieve the best purification effect[1][2].

2.3 Detection methods

1. The sugar concentrations were determined using saccharimeter WYT [1].
2. The purification of xylitol was determined by HPLC[1][3]. Specific conditions were as follows. Column, sugar column CHO-99-9453, Transgenomic, USA. Mobile phase: distilled water. Column temperature 80℃. Flow rate mL•min-1. Injection volume 10μL. Refractive Index Detector RI2001, Tianjin Lanbo.
3. The yield was calculated according to the formula.

$$\text{Yield（\%）} = \frac{\rho_1 \times V_1 \times C_1}{\rho_0 \times V_0 \times C_0} \times 100\%$$

C1-otal sugar concentration of xylitol after separation (mg·mL-1). C0-total sugar concentration of materials (mg·mL-1). ρ1-purity of xylitol after separation (%). ρ0-purity of xylitol in materials (%). V1-solution volume of xylitol after separation (mL). V0-solution volume of materials(mL).

2.4 Calculation method of resolution degree(Rs)[5]

Resolution degree is calculated according to the formula:

Rs=2（t2-t1）/(W2+W1)

In the formula, t2 is the retention time of xylitol; t1 is the retention time of oligosaccharides; W1 is the chromatographic peak width of oligosaccharides; W2 is the chromatographic peak width of xylitol.

3. Design and implementation of SSMB

3.1 Parameters and structures of SSMB

According to the process requirements above, configuration software, industrial computer, PLC and related electrical equipments were adopted to design a small scale of SSMB. Specific parameters were as follows. Separating column (34×300mm, n=6) volume 6L. Temperature 0℃~80℃. Pressure range 0 MPa~1MPa. Pump flow for materials 0.01mL~50.00 mL (H2O). Pump flow for water 0.01mL~50.00 mL (H2O). Circulating pump flow 0.01mL~80.00 mL (H2O).

The main structure of SSMB was as shown in Figure 2. SSMB was composed of six head-tail connecting chromatographic columns filled with solid adsorbent. The upper part of columns was liquid inlet and the lower part was outlet. Whether the inlet and outlet of columns were open or the pipe connections between columns worked depended on the controlling solenoid valves. Both computers and PLC were adopted to control the automatic switch of valves and the speed of pumps in order to control the rate, volume and direction of flow precisely.

The structure of control system of SSMB was shown in Figure 3.

3.2 Hardware design and implementation of SSMB

3.2.1 Hardware design

Hardware of SSMB should have the following functions after analysis.

1. Data collection, including parameters (pressure and flow) detection.
2. Data processing. Collected data had to be real-time stored and presented on the display.
3. Process control. Commands had to be sent to equipments including PLC according to the system settings and technological process to control each valve and adjust process temperature and flow rate.

Selected hardware were as follows according to requirements above.

1. Industrial control computer (IPC), Advantech Store.
2. PLC. FX2N-128MR-001 and extended module from Mitsubishi were responsible for the control of solenoid valves and pumps and data collection.
3. Analog collecting modules FX2N-4AD.
4. Temperature sensor. Three-wire system of PT100 was chosen with accuracy of 0.5% and measurement ranging from 0℃ to 300℃.
5. Temperature transmitter SBWZ-PT100, output current 4~20mA, temperature ranging from 0℃to 300℃, measuring accuracy of ±0.2%FS, power voltage 24VDC±10%.
6. Heater. Preheating of materials was realized by traditional thermal resistance and solid state relay.
7. Pumps. ZWN60, a motor drive gear pump without brush.

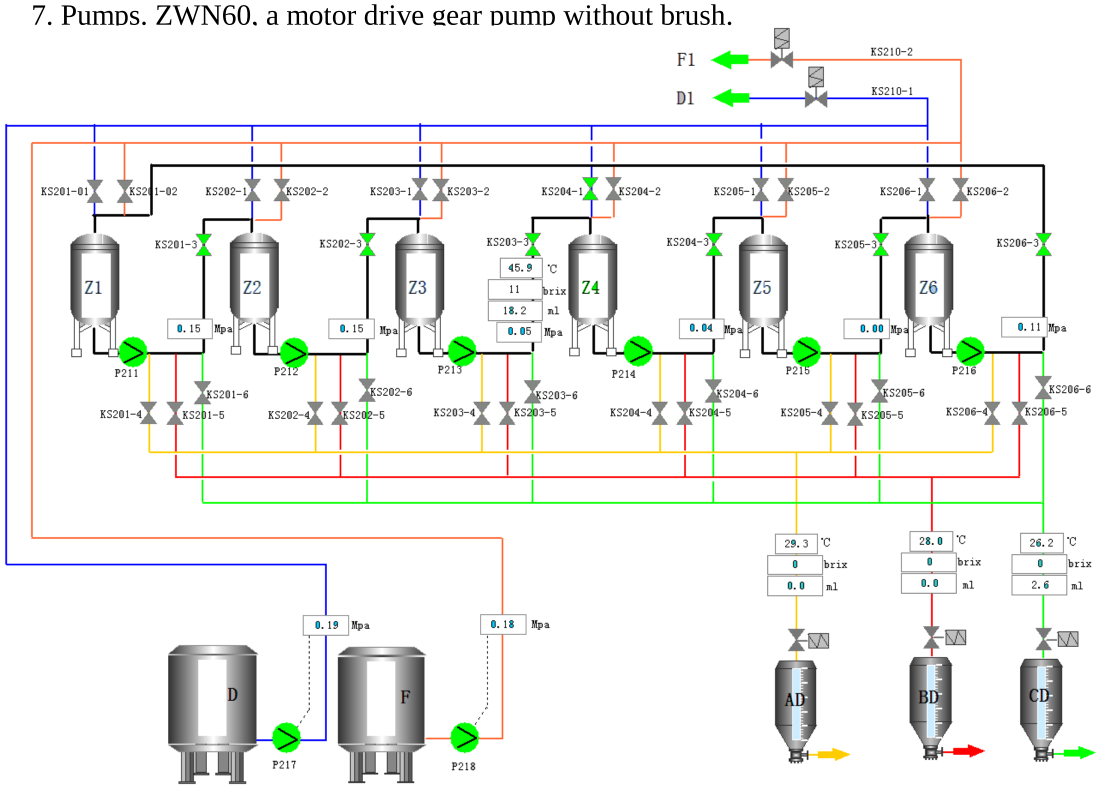

Figure 2. Main structure of SSMB

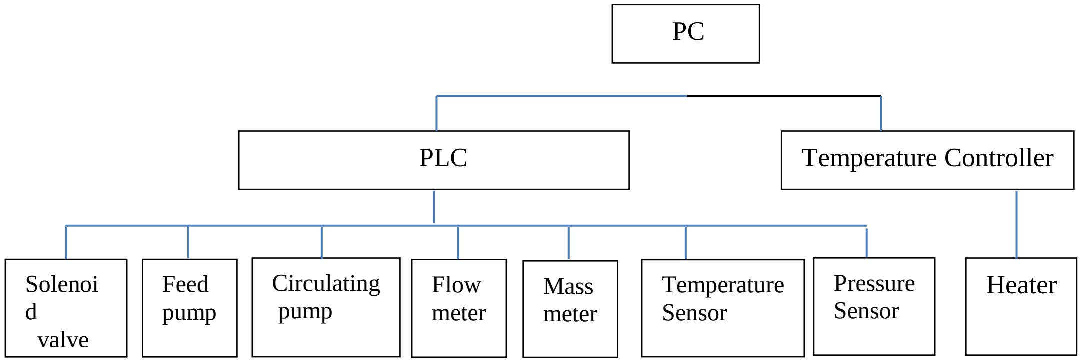

Figure 3. Structure of control system of SSMB

8. Pressure transmitter. PLS-200 from VOLKE contained an imported diffused silicon sensor. Range from -0.1MPa to 20MPa. Measurement accuracy 0.2%. Temperature effect ±0.07%FS/ 10℃. Stabilization ±0.10%FS/year. Reproducibility error ±0.10%FS. Working voltage DC24V. Output current 4mA ~ 20mA.

9. Mass flowmeter. Measurement ranging from 0 kg/h to 40kg/h. Measured pressure 0 MPa ~ 4MPa. Measurement accuracy 0.1% ~ 0.2%.

10. Temperature controller, MR13, Shimaden, Japan. The controller contained three-warning output and communication interface RS232 (RS485) which was responsible for communication with host computer. Manual temperature control system with the function of analysis and without records was rapidly built with the help of MR13. Furthermore, MR13 also promoted the construction of multi-zone automatic heating control system together with configuration software of host computer and PLC, which could be applied to record, analyze and process data[1].

3.2.2 Function implementation

1. Automatic operation

The output state and switch time of PLC set up by IPC. The switch state of solenoid valves depended on the output of PLC in this system.

2. Manual operation

Manual operation could be implemented as well in the system. Single step including experiment, rinse and detection was implemented following the setup of PLC and output control of PLC.

3. Flow control

High-accuracy motor drive gear pump without brush, ZWN60, was utilized in this design. According to the set value, the operation state and rotating speed of ZWN60 were controlled by pulse signal sent by PLC.

4. Collection and control of temperature

Pt resistance temperature transducer PT100 was used as the temperature sensor in the system. PT100 transformed the resistance variations induced by temperature into current signal from 4mA to 20mA. Then the current signals were sent to PLC by the collecting module and to the host computer by PLC sequentially. Temperature controller, MR13, was responsible for adjustment of electric heating to control the temperature.

5. Pressure collection

Pressure transmitter transformed the change of pressure into current signal from 4mA to 20mA. Then the current signals were sent to PLC by the collecting module and to the host computer by PLC sequentially. Alarm limit was set up by IPC.

6. Collection of material

Mass flowmeter was applied to collect the information concerning the quality of input and output materials. Data collection was realized by serial mode of RS232 and IPC was responsible for the display, alarm and record of data.

3.3 Software design and implementation of SSMB

Software of host computer are developed in two forms in current industrial control research. One is basic development through advanced visual programming language such as VB, VC and Delphi. The other is secondary development based on configuration software including WinCC, King View and MCGS. King View was adopted in this system and secondary development was expected[2][1][3].

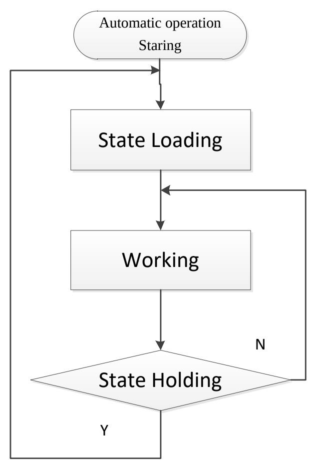

Figure 4. Automatic operation flow

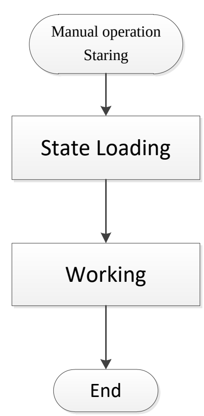

Figure 5. Manual operation flow

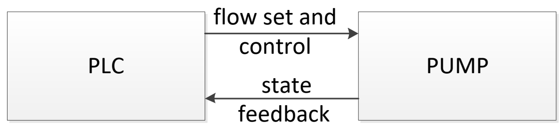

Figure 6. Flow control flow

3.3.1 Functions of system software

The functions of host computer monitoring system were as follows.

1. System picture configuration was beneficial to be monitored, tested, data collected and analyzed by users.
2. Setting related parameters of the system, for example temperature and flow.
3. Data display. The database of King View were adopted to collect and restore the real-time data including temperature, pressure and quality.
4. Controlling the running state by setting and switching parameters manually and automatically.

3.3.2 Design of system configuration

The configuration of controlling system consisted of main picture, operating parameters picture, loading picture. The specific structure of system configuration was shown in Figure 7.

The main picture was shown in Figure 8.

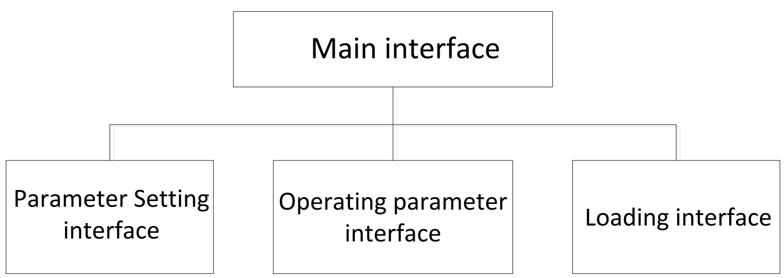

Figure 7. Structure of system configuration

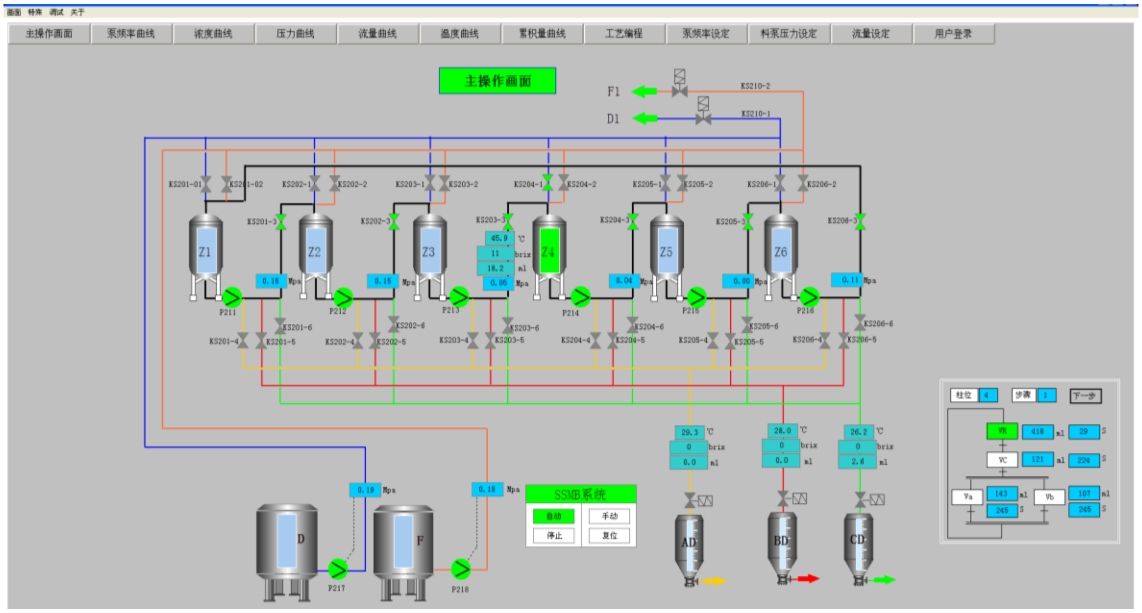

Figure 8. Main picture

Figure 9. Operating parameters picture

3.3.3 Application of communication technology

Communication technologies including computer and PLC, computer and intelligent thermostat were applied in the system. Furthermore, PLC could communicate following setting the communication parameters including baud rate, data bits, stop bits and parity check instead of special configuration due to the integration of PLC driver from Mitsubishi and RS232 communication mode between COM and PS/2. The communication parameters in the system were as follows: baud rate 9600, data bits 7, stop bits 1 and even parity. The communication mode between computer and intelligent thermostat was RS232 [1][2].

3.3.4 Design of controller

Temperature controller was responsible for controlling operating temperature and connected with computer in serial mode. Better accuracy depended upon proper parameters in this system. Segmented parameter design was adopted as shown in Figure 10.

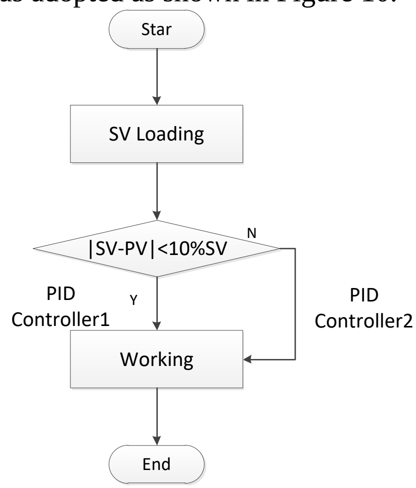

Figure 10. Temperature control flow

When the system started heating, let Ti is 0, Td is 0 and set the 60%~70% of the maximum value allowed as input in field debugging. Increase the proportional gain P from 0 until oscillation was observed, then decrease the proportional gain P until oscillation disappeared in turn and record the proportional gain P at this moment. Set the 60%~70% of the P value as the proportional gain of PID. After the proportional gain was determined, set a larger value of integration time constant Ti and reduce it until oscillation was observed. Then increase Ti gradually until oscillation disappeared and record the Ti at the moment. Set the 150%~180% of the Ti value as the integration time constant of PID. Similarly, Td was determined as the 30% of value recorded when oscillation disappeared. All these above were controller parameters in I segment. After being steady, readjust the parameters of PID controller and determine the parameters in II segment to ensure the timely response, high accuracy and excellent control effect of the system[13][14].

4. Results and analysis of verification test

4.1 Analysis of test results

The results of HPLC of raw materials liquid were shown in Figure 11 and Table1.

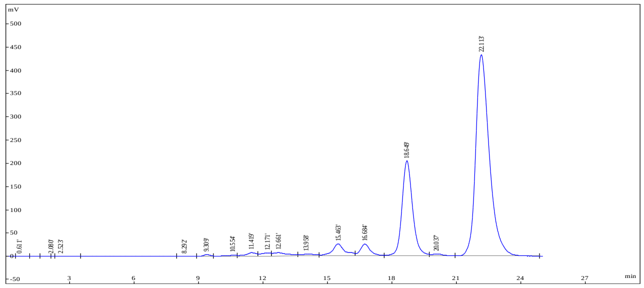

Figure 11. HPLC of Xylitol mother liquor

Table 1. HPLC analysis of Xylitol mother liquor

Code	Retention time(min)	Contents(%)	Composition
1	15.463	6.60	Hybrid sugar alcohols
2	16.684	5.54	Hybrid sugar alcohols
3	18.649	23.56	Arabinitol
4	20.037	0.62	Hybrid sugar alcohols
5	22.113	63.68	Xylitol

4.2 Results of preparative chromatography evaluation experiment

The evaluating results of preparative chromatography column of xylitol mother liquor were shown in Table2, and the elution curve was shown in Figure 12.

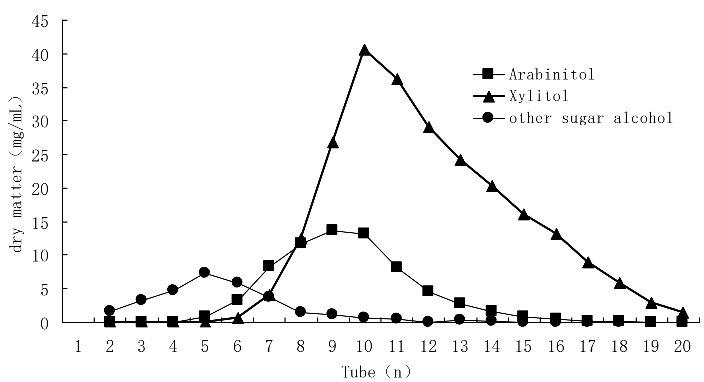

Figure 12. Elution curve of xylitol

Table 2. Results of preparative chromatography evaluation experiment

Tube	Volume（mL）	Concentration（%）	arabinitol purity（%）	Xylitol purity（%）	other sugar alcohol（%）	Arabinitol dry matter（mg）	Xylitol dry matter（mg）	other sugar alcohol dry matter（mg）
15	48	0.5	0	0	100	0	0	1.6
16	51.2	1	0	0	100	0	0	3.2
17	54.4	1.5	1.09	0	98.89	0.05328	0	4.74672
18	57.6	2.5	9.51	0	90.48	0.7616	0	7.2384
19	60.8	3	33.22	6.1	60.77	3.17952	0.5856	5.83392
20	64	5	51.34	25.15	23.41	8.2304	4.024	3.7456
21	67.2	8	45.75	48.26	5.7	11.712	12.43136	1.4592
22	70.4	13	32.73	64.34	2.77	13.65728	26.7904	1.15232
23	73.6	17	24.18	74.72	1.09	13.15392	40.65312	0.59296
24	76.8	14	19.18	80.98	0.94	8.09984	36.27904	0.42112
25	80	10.5	13.49	86.36	0.14	4.53264	29.01696	0.04704
26	83.2	8.5	9.98	88.79	1.23	2.71456	24.15088	0.33456
27	86.4	7	6.97	90.59	0.44	1.56128	20.29216	0.09856
28	89.6	5.5	4.88	91.04	0.08	0.85888	16.02304	0.01408
29	92.8	4.5	3.36	91.64	0	0.48384	13.19616	0
30	96	3	2.38	92.27	0.36	0.22848	8.85792	0.03456
31	99.2	2	1.66	92.19	0.15	0.10624	5.90016	0.0096
32	102.4	1	1.52	92.65	0.83	0.04864	2.9648	0.02656
33	105.6	0.5	1.99	92.41	0.6	0.03184	1.47856	0.0096

As was shown in Table 2 and Figure 12, the retention time of xylitol was significantly different from that of other sugar alcohol. Following calculating, the index of separation was only 0.33, which indicated that there was no complete separation between xylitol and other sugar alcohol in spite of the trendency of separating. However, the effect of separation was able to be improved by modifying the conditions of process such as lengthening the distance and time of separation and increasing the volume of elution water. Further optimization of process parameters resulted in better separation effect[15][16].

4.3 Analysis of SSMB results

Process parameters and test results of xylitol mother liquid purification by SSMB were shown in Table3.

Table 3. The optimized small scale operation parameters of SSMB

Code	Feed quantity (g·h-1)	Water consumption (g·h-1)	Circulation（mL）	Concentration (%)	Glucose purity (%)	Glucose yield (%)
1	346.00	546.00	345.80	42.4±0.1[b]	93.2±0.1[d]	90.1±0.2[d]
2	346.00	728.00	364.00	37.0±0.3[f]	90.2±0.2[f]	92.3±0.2[a]
3	455.00	682.50	364.00	43.8±0. 2[a]	92.1±0.3[e]	90.5±0.2[bc]
4	455.00	910.00	318.50	42.2±0.2[bc]	94.2±0.1c	90.8±0.1[b]
5	546.00	819.00	382.20	41.2±0.2[d]	94.8±0.3[ab]	90.6±0.3[bc]
6	546.00	819.00	391.30	39.67±0.3[e]	95.3±0.3[a]	88.6±0.3[e]

As seen in Table 3, the 5th test group was better than the other groups considering the treatment quantity, ratio of material to water, concentrations, xylitol purity and yield. Therefore, the optimum separation parameters were feed concentration 60%, feed quantity 546.00g·h-1, water consumption 819.00 g·h-1, circulation 382.2mL, and the output concentration reached 41.2%, the purity and yield were up to 94.8% and 90.6%, respectively under the conditions stated above.

The HPLC results of AD and BD compositions were shown in Figure 13 and Figure 14 respectively after purifying for 10h by SSMB. The purity of xylitol in AD was 94.67% and that of arabinitol in BD was 43.31%. The yield of xylitol was up to 90.52%. Therefore, the excellent effect of purification provided an evidence that the new control system ran steadily and controlled precisely.

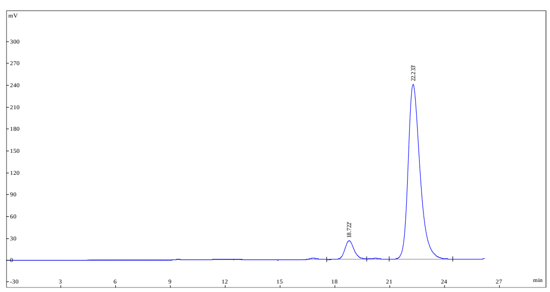

Figure 13. HPLC of AD composition

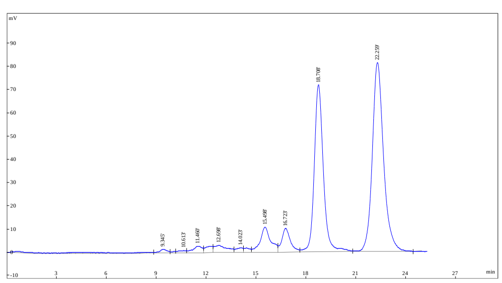

Figure 14. HPLC of BD composition

4.4 Comparative analysis the effect of purification of SMB and SSMB for xylitol motherliquid

Main indicators in separation process of SSMB and SMB were comparative analyzed, in order to determine the separation advantages and disadvantages of SSMB and SMB technology, the results are shown in Table 4.

Table 4. The comparison of SMB and SSMB seperation conditions

Item	SMB	SSMB
Number of chromatographic columns	12	6
The addition amount of resin	1.2 L	6 L
The ratio of material to water	3.0:1	1.5:1
Feed concentration	50%	60%
processing capacity	1.5 kg·d-1	13.1kg·d-1
glucose concentration	25.5%	41.2%
Glucose purity	91.4%	94.8%
Glucose yield	87.2%	90.6%

As seen in Table 4, main indicators in separation process of SSMB were superior to SMB separation process. The number of chromatographic column SSMB were six less than that of SMB, so the equipment investment is reduced. The water consumption of SSMB was 60% lower than SMB, which reduced the operation cost. The feed concentration and outlet sample concentration in the SSMB process were higher than that of SMB process, which increased processing capacity, decreased the cost of material concentration, and reduced the operation cost[17][18]. In addition, the purity and yield of xylitol purity with SSMB process was 94.8% and 90.6%, respectively. Which were significantly higher than the purity and yield of xylitol purity was 91.4% and 87.2% with SMB process, respectively.

5. Conclusions

A creative control system of SSMB was designed and proved to run steadily and control precisely in the purification of xylitol mother liquor for 10h continuously. The obtained results were purify 94.8% and yield 90.6% in verification test, significantly better than the current results of purify 92% and yield 85%[17][18]. The innovation of control system based on PLC and King View improved the purification effect of SSMB and realized the steady and automatic running for long hours, which laid a solid foundation for the research and application of SSMB control system in our country.

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