Dynamics and control of a heat pump assisted azeotropic dividing-wall column for biobutanol purification

DOI:

[10.1016/j.cherd.2019.04.029](https://doi.org/10.1016/j.cherd.2019.04.029)

Document Version

Accepted author manuscript

[Link to publication record in Manchester Research Explorer](#)

Citation for published version (APA):

Patracu, I., Bildea, C. S., & Kiss, A. (2019). Dynamics and control of a heat pump assisted azeotropic dividing-wall column for biobutanol purification. *Chemical Engineering Research & Design*.
<https://doi.org/10.1016/j.cherd.2019.04.029>

Published in:

Chemical Engineering Research & Design

Citing this paper

Please note that where the full-text provided on Manchester Research Explorer is the Author Accepted Manuscript or Proof version this may differ from the final Published version. If citing, it is advised that you check and use the publisher's definitive version.

General rights

Copyright and moral rights for the publications made accessible in the Research Explorer are retained by the authors and/or other copyright owners and it is a condition of accessing publications that users recognise and abide by the legal requirements associated with these rights.

Takedown policy

If you believe that this document breaches copyright please refer to the University of Manchester's Takedown Procedures [<http://man.ac.uk/04Y6Bo>] or contact uml.scholarlycommunications@manchester.ac.uk providing relevant details, so we can investigate your claim.

Dynamics and control of a heat pump assisted azeotropic dividing-wall column for biobutanol purification

Julian Patraşcu,¹ Costin Sorin Bildea,¹ Anton A. Kiss,^{2,3}*

¹ *University “Politehnica” of Bucharest, Polizu 1-7, 011061 Bucharest, Romania*

² *School of Chemical Engineering and Analytical Science, The University of Manchester, Sackville Street, Manchester, M13 9PL, United Kingdom*

³ *Sustainable Process Technology Group, Faculty of Science and Technology, University of Twente, PO Box 217, 7500 AE Enschede, The Netherlands*

** Corresponding author: TonyKiss@gmail.com, Tel: +44 161 306 8759*

Keywords

Downstream processing, distillation, dividing-wall column, optimal design, process control

Highlights

- Highly integrated design for ABE separation leading to process control challenges
- Efficient control structure ensuring stable operation of VRC assisted A-DWC
- Disturbances in feed flowrate and composition can be effectively rejected

Abstract

An efficient downstream process has been developed for the acetone-butanol-ethanol (ABE), allowing about 60% energy savings as compared to a conventional separation sequence. This is achievable in an azeotropic dividing-wall column (A-DWC) coupled with a heat pump and applying heat integration. The energy use is reduced to only 2.7 MJ/kg butanol (or 7.5% of the energy content of butanol), but to take full advantage of the energy savings offered by this highly integrated process, the process must be also controllable. This work presents the dynamics and control of this novel A-DWC process used for biobutanol purification.

A rigorous, pressure-driven dynamic simulation has been developed in Aspen Plus Dynamics. After adding the required PID controllers (e.g. flow, pressure, liquid level and temperature) to the base-case process, the system can handle small and short disturbances (e.g. 5% change of feed flow rate for 10 min.) but if the disturbance persists for a long time the process shuts down. To solve this problem, the controllability of the system is improved by adding an additional reboiler and condenser to control the temperature on the feed-side stripping section. Moreover, adding concentration controllers ensures that the products purity is kept constant when feed flowrate or composition disturbances occur.

1. Introduction

Biobutanol is a promising alternative fuel, with better physical and chemical properties than other biofuels. In addition to its role as biofuel, biobutanol can also be used to produce a wide range of chemicals. For its eco-efficient production, the selection of species, substrates, pre-treatment, genetic engineering techniques and various downstream processing techniques have been evaluated and reported in literature (Jin et al., 2011; Huang et al., 2014; Bharathiraja et al., 2017). Biobutanol is obtained in diluted form in the acetone-butanol-ethanol (ABE) fermentation process, with concentration lower than 3 %wt due to the severe butanol toxicity to micro-organisms. The efficiency of the ABE process can be improved by optimizing the upstream (e.g. pre-treatment and fermentation) and/or the downstream steps (e.g. product recovery and purification) by various techniques (Mariano et al., 2010; Garcia et al., 2011). Yet, the upstream changes rely heavily on modification of microorganisms by genetic engineering to increase the productivity, yield and concentration and thus reducing the production costs. While this remains a long-term challenge, the development of more efficient downstream processes for butanol recovery is actually a realizable option (Kujawska et al., 2015; Patrascu et al., 2017, 2018). In fact, achieving cost effective separations and process integration was shown to be an essential requirement for sustainable biorefineries (Kiss et al., 2015, 2016). A recent review paper provides more insights into the selection and design of fluid separation processes that are applicable also to biofuels (Blahusiak et al., 2018).

To avoid separating a diluted biobutanol solution, the concentration of the ABE produced can be further increased by in-situ product recovery (ISPR) methods, such as gas stripping technology, to: 4.5 %wt acetone, 18.6 %wt butanol and 0.9 %wt ethanol (Xue et al., 2013). Many techniques are available for ABE separation, e.g. distillation, reverse osmosis, adsorption, liquid-liquid extraction, pervaporation and others (Abdehagh et al., 2014). Among the methods used for recovery of biofuels, advanced distillation techniques (e.g. heat pump assisted distillation and/or dividing-wall column) proved capable of obtaining significant energy savings (Kiss, 2013; Luo et al., 2015; Kiss and Infante Ferreira, 2016).

A novel heat pump assisted azeotropic dividing-wall column (A-DWC) was suggested for the ABE separation (Patrascu et al., 2018), which allows up to 60% energy savings compared to a conventional separation sequence. For a production of 40 ktpy butanol, the total equipment cost was evaluated at 5250 k\$, while the operating cost was 1435 k\$/year. Although using a heat pump increases the capital cost due to the addition of a compressor (1581 k\$), considering the energy savings (1.69 MJ/kg butanol) evaluated at 1893 k\$/year, the payback time of the added compressor is less than one year. This work continues the endeavour by

1 considering the dynamics and control of the heat pump assisted azeotropic dividing-wall
2 column, with the aim of proving that such a highly integrated system is controllable and thus
3 the energy savings are also achievable in practice. Adding a heat pump into this process
4 integration brings additional control challenges on top of those encountered in the classic
5 control of DWC (Kiss and Bîldea, 2011; Rewagad and Kiss, 2012).

6 Although there are significant differences regarding the configuration of the DWC, the
7 challenge is somewhat similar to the tert-butanol / water separation in a vapor recompression
8 assisted DWC using cyclohexane as entrainer in heterogeneous azeotropic distillation
9 (Luyben, 2016), or the bioethanol dehydration in a heat pump assisted extractive DWC
10 (Patrascu et al., 2017). However, instead of exploiting the heterogeneity of the cyclohexane /
11 water / tert-butanol azeotrope (Luyben, 2016) or the change of ethanol / water relative
12 volatility induced by ethylene glycol (Patrascu et al., 2017), the design analysed here exploits
13 the heterogeneity of the n-butanol – water azeotrope, without adding a mass separation agent.
14 Crossing the distillation boundary by the liquid-liquid split takes place in a side-decanter.

16 **2. Problem statement**

17 Combining heat pumps (e.g. vapor recompression) with azeotropic dividing-wall column (A-
18 DWC) technology offers major advantages in terms of annualized costs and large energy
19 savings of up to 60%. But such an integrated system might prove difficult to control and this
20 is just as important as the cost savings, considering that in practice the plant must be available
21 and operable to attain the expected design savings. The problem is that a highly integrated
22 system has reduced flexibility and fewer degrees of freedom, thus being more difficult to
23 control. To solve this problem, we investigate here the dynamics of the process and propose a
24 simple but effective process control structure that is able to reject common disturbances,
25 within short settling times and low overshooting.

27 **3. Process design**

28 An efficient control structure is developed in this work by addressing the main task of
29 plantwide control, namely the strategy of solving the dynamic problems of a process under the
30 production (rate and quality) and safety constraints, while minimizing the costs. Achieving this
31 goal requires dynamic simulations (carried out in Aspen Plus Dynamics) to understand the
32 dynamics of the process and to prove the effectiveness of the proposed control structures. The
33 starting point is the heat integrated VRC assisted A-DWC design proposed by Patrascu et al.
34 (2018), which achieves 59.75% energy savings compared to the conventional separation

sequence. The reader is referred to the original paper (Patrascu et al., 2018) for more details concerning the principle of operation and the efficiency of the system.

The azeotropic dividing-wall column (Figure 1) combines three distillation columns in one single unit. The new design has two stripping section and one fractionation section. The cold fresh feed is preheated and partially vaporized from 25 °C to 97 °C, near the boiling point of the heterogeneous azeotrope butanol-water. This requires an important amount of heat (5.77MW), almost half of the total heating requirements. The feed-side stripping section (DWC-L) separates as distillate the acetone, ethanol and butanol (with some water involved in the ethanol-water and butanol-water azeotropes) and delivers high purity water as bottom product. Therefore, the most-plentiful component is obtained here. The rectifying section (AEW) provides the light components (acetone and ethanol, with some water involved in the ethanol-water azeotrope) as distillate. The liquid flowing down from the fractionation section is routed to the second stripping section (DWC-R, opposite to feed). A large fraction of the liquid (with near azeotropic composition) is cooled to 40 °C and sent to decanting. Here, the distillation boundary is crossed. The organic phase, which is returned to the column, is on the butanol-side of the azeotrope. Therefore, high-purity butanol can be obtained as bottoms product of the DWC-R section. The aqueous phase obtained from the decanter is mixed with the feed and sent to the water-removal section. In order to achieve energy savings, the top vapor stream is compressed from 1 bar to 5.8 bar. This way, the temperature increases from 60 °C to 150 °C, which is high enough to drive the reboiler of the water-removal stripping section and to preheat the fresh feed. Moreover, the hot water product is used to preheat the organic and aqueous phases obtained from decanting, before being returned to the column. In this way, 69% energy savings (57% due to use of heat pump, 12% due to process-process heat exchange) is achieved compared to a simple A-DWC column. Figure 1 shows the mass balance, with some additional technical details. Figure 2 illustrates the liquid composition profile and the temperature profile of the A-DWC unit. In the bottom stages of the two stripping sections the purity of water (99.8 %wt. – dash line) and the purity of butanol (99.4 %wt. – line) can be noticed. The distillate contains small amount of water because of the ethanol – water azeotrope. The middle part of the column (stages 10, 11, 13) contains the butanol-water azeotrope. Figure 3 presents the temperature – duty and vapour fraction - duty profiles along the two heat exchangers where the compressed vapours are first de-superheated and partially condensed to 60 % vapor fraction (Hex1) and then totally condensed and subcooled (Hex2). Further sub-cooling occurs in the last heat exchanger Cool4.

4. Results and discussion

4.1 Process dynamics

The pressure driven simulation is built in Aspen Plus, then exported to Aspen Plus Dynamics. Two RADFRAC units are used to model the water removal section (DWC-L) and the combined rectifying (AEW) and butanol purification sections (DWC-R). The reboiler on the water-removal side is represented as a counter-current heat exchanger followed by a flash. More details concerning the type of models used for dynamic simulation and their specifications are given in Table 1. Note that Table 1 includes an auxiliary reboiler and a condenser, which will be added later to the flowsheet, in order to improve the controllability. Several pumps and valves are provided on following streams: bottom stream of the water-removal stripping section, side stream, water and butanol product streams, distillate, reflux, organic and aqueous phase stream. No valves are necessary on the streams (internal flows) connecting the water-removal to the fractionation section, the vapor-liquid separator (FLASH) to the water-removal section, and the top main column to compressor. The characteristics of the pumps and valves are shown in Table 2.

The basic control is ensured with the following flow rate, liquid level, pressure and temperature loops:

- Fresh feed flow rate, by manipulating the valve position of the feed stream.
- Liquid level of the sump, by manipulating the valve position of the bottom stream (both water removal DWC-L and butanol purification DWC-R sections).
- Liquid level of the vapour-liquid separator, by manipulating the valve position of the water product stream.
- Liquid level of the organic phase in decanter, by manipulating the valve position of the organic stream.
- Liquid level of the aqueous phase in decanter, by manipulating the valve position of the aqueous stream.
- Liquid level of the reflux drum, by manipulating the valve position of the reflux stream.
- Pressure of the main column, by manipulating the compressor brake power.
- Temperature on stage 5 of the fractionation section, by manipulating the valve position of the distillate stream.
- Temperature on stage 40 of the butanol-purification section (DWC-R), by manipulating the reboiler duty.

- Temperature of the mixture sent to decanting (the side stream, butanol-water azeotrope), by manipulating the cooler duty (Cool1).
- Temperature of the subcooled condensate, by manipulating the cooler duty (Cool4).
- Temperature of the bottom products, by manipulating the corresponding cooler duties.
- Side stream flow rate, by valve position of the side stream.
- Ratio control between the side stream and feed, set to the steady state value

The settings of these controllers are detailed in Table 3. These basic controllers (Figure 4) can stabilize the process against small and non-persistent disturbances. For example, when the feed flow rate changed by $\pm 5\%$ for 10 minutes (Figure 5), the process returns to the initial operating point when the disturbance is removed. However, when the feed flow rate is ramped by 5% over one hour (a persistent disturbance, Figure 6), the stream entering the column cools down, the pressure and the temperature in column drop, less vapours are sent to rectifying section and to compressor. This leads to further temperature decrease in the column. In the meantime, the temperature controller of stage 40 temperature of the butanol-purification tries to compensate the disturbance by increasing the reboiler duty. This affects the temperature of stage 5, so the top temperature controller closes the distillate stream valve. Further, the pressure controller reduces the brake power, trying to increase the column pressure. As a result, the vapours flow is too small to drive the water removal section. Therefore, some new manipulated variable is needed to provide the additional heat required to reject this disturbance.

When the feed flow rate is decreased by 5% and the disturbance persists for a long time (also shown in Figure 6), the feed enters the column at higher temperature. In this case more vapours are sent to the rectifying section and more vapours are compressed, leading to continually temperature increase in the water-removal stripping section. As a result, more water is vaporized and eventually reaches the butanol product. Such disturbance could be rejected by reducing the temperature of the stream entering the column, but no manipulated variable is available.

4.2 Process control

To overcome these control difficulties, two new variables to be manipulated were added (Figure 7). First one is a small additional duty (Duty-aux, 0.3 MW) on the vapor-liquid separator (the reboiler of the water-removal section), which represents 10% of the energy required. The second one is an additional cooler (Cond-aux, -0.68 MW) placed on the

1 compressed vapor stream, between the reboiler and the feed preheating. Compared to the
2 original design (Patrascu et al., 2018), the area of Hex1 decreases from 491 m² to 456 m²,
3 while the area of Hex2 increases from 301 m² to 345.9 m². As a result of the changes, the
4 temperature, pressure and composition of the key streams presented in Figure 1 do not
5 change. The two new duties are used in new control loops as follows:

- 6 • Temperature of the preheated feed, by manipulating the duty of the additional cooler.
- 7 • Temperature on stage 5 in water-removal section (DWC-L), by manipulating the
8 additional duty of vapor-liquid separator.

9 By introducing these controllers, the process can also handle large feed flow rate disturbances
10 for long time. Figure 8 shows the behaviour of the system when the feed flow rate is increased
11 or decreased by 10% (the disturbance, twice the disturbance applied in Figure 6, is applied
12 after 2 hours of steady state operation). As long as the right vapor flow is ensured by the new
13 variables (additional duties), the pressure and temperature in top of the main column can be
14 easily controlled by the compressor brake power and the distillate rate, respectively. The
15 pressure on the top of the column (left diagram, DWC-R top) remains unchanged, while only
16 small deviations from the steady value are observed at the top of the water-removal section
17 (DWC-L top). The corresponding temperatures (right diagram) also show small changes.
18 Figure 9 shows the performance of the control system for feed flow rate (top diagrams) and
19 feed composition (bottom diagrams) disturbances. The transient regime takes about 5 hours.
20 As the feed rate changes, the water purity and the mass fraction of water in the distillate are
21 practically unaffected, while the butanol purity deviates by only 0.1 %wt. from the design
22 value. When the fraction of butanol in the feed stream changes (from 18.6%wt. to 17 %wt. or
23 20 %wt., bottom diagrams), the behaviour is similar: water and distillate compositions are
24 practically unchanged, while the concentration of the butanol product deviates by about 0.1
25 %wt. from the design value. The performance of the control system is excellent, considering
26 that only temperature measurements were used.

27 Note that choosing a stage from the upper part of the water-removal (DWC-L) section for
28 temperature control is critical. For example, one could choose a stage in the lower part (for
29 example, stage 25) with the idea that this would keep the purity of the water product closer to
30 the required value, avoiding the use of a concentration control loop. However, dynamic
31 simulations showed that disturbances in the fresh feed are seen by the control system too late,
32 after they propagate from stage 1 to stage 25 of the DWC-L section. By now, the rectifying
33 section is also seriously affected. Thus, increasing the duty of the auxiliary reboiler duty is not
34 effective, and the compressor power must also be employed for temperature control purposes

(override control should be included to avoid that the compressor-outlet temperature does not increase above a certain limit). Then, the only way to control the column pressure is by using the sub-cooled reflux. While such control structure works, it is more complex and does not provide better performance compared to the control structure presented in Figure 7.

If one wants to keep the purity of the butanol and water products exactly at their setpoints, concentration measurements are necessary. The slow dynamics of these measurement was taken into account by using the Aspen Plus Dynamics “sensor” model with 5 minutes sampling period and 5 minutes dead time. Just one concentration controller is provided. This adjusts the setpoint of the temperature controller of the butanol-purification section (stage 40). Figure 11 shows dynamic simulation result for the control structure employing the concentration controllers. The diagrams are organized similarly to Figure 9 – top diagrams for feed flow rate disturbances, bottom diagrams for feed composition disturbances. The deviation of butanol purity from the steady state value is quite small. Eventually, 4 hours after the disturbance, the butanol purity comes back to the design value (99.4 %wt.). Water purity is practically unchanged, while the mass fraction of water in the distillate is acceptable.

5. Conclusions

The downstream processing of biobutanol in the ABE process can be effectively performed in a heat pump assisted azeotropic dividing-wall column that is heat integrated and allows 59.75% energy savings as compared to a conventional separation sequence. Such integrated processes have more interactions between variables and fewer degrees of freedom as compared to conventional DWC systems, making it challenging to control and questioning the expected benefits.

The dynamics and control of this highly integrated process was successfully explored by using rigorous, pressure-driven dynamic simulations developed in Aspen Plus Dynamics.

- When adding the required PID controllers (e.g. flow, pressure, liquid level and temperature) to the base-case process, the system can handle only small and short disturbances (e.g. 5% of feed flow rate for 10 min.) but if the disturbance persists for a long time the process shuts down, hence it would be uncontrollable in practice.
- The controllability of this integrated system can be significantly improved by adding an additional reboiler (to control one temperature on the water removal stripping section; it is essential that the temperature measurement is place on the upper part of this section) and an additional cooler (to control the temperature of the stream entering the column). The energy penalty for improving the controllability is only 4%.

- The inferential control using only temperature measurements provides excellent performance (very small purity offset), for rather large feed flowrate or feed composition disturbances. The offset can be completely removed by adding one concentration controller for the butanol product stream.

Acknowledgement

Financial support of the European Commission through the European Regional Development Fund and of the Romanian state budget, under the grant agreement 155/25.11.2016 (Project POC P-37-449, acronym ASPIRE) is gratefully acknowledged. AAK gratefully acknowledges the Royal Society Wolfson Research Merit Award. The authors also thank the reviewers for their insightful comments and suggestions.

References

1. Abdehagh N., Tezel F. H., Thibault J., Separation techniques in butanol production: Challenges and developments, *Biomass and Bioenergy*, 60 (2014), 222-246.
2. Bharathiraja B., Jayamuthunagai J., Sudharsanaa T., Bharghavi A., Praveenkumar R., Chakravarthy M., Yuvaraj D., Biobutanol - An impending biofuel for future: A review on upstream and downstream processing techniques, *Renewable and Sustainable Energy Reviews*, 68 (2017), 788-807.
3. Blahusiak M., Kiss A. A., Babic K., Kersten S. R. A., Bargeman G., Schuur B., Insights into the selection and design of fluid separation processes, *Separation and Purification Technology*, 194 (2018), 301-318.
4. Garcia V., Pääkilä J., Ojamo H., Muurinen E., Keiski R. L., Challenges in biobutanol production: How to improve the efficiency?, *Renewable and Sustainable Energy Reviews*, 15 (2011), 964-980.
5. Huang H. J., Ramaswamy S., Liu Y.-Y., Separation and purification of biobutanol during bioconversion of biomass, *Separation and Purification Technology*, 132 (2014), 513-540.
6. Jin C., Yao M., Liu H., Lee C. F., Ji J., Progress in the production and application of n-butanol as a biofuel, *Renewable and Sustainable Energy Reviews*, 15 (2011), 4080-4106.
7. Kiss A. A., Novel applications of dividing-wall column technology to biofuel production processes, *Journal of Chemical Technology and Biotechnology*, 88 (2013), 1387-1404.
8. Kiss A. A., Grievink J., Rito-Palomares M., A systems engineering perspective on process integration in industrial biotechnology, *Journal of Chemical Technology and Biotechnology*, 90 (2015), 349-355.
9. Kiss A. A., Bildea C. S., A control perspective on process intensification in dividing-wall

- columns, *Chemical Engineering and Processing: Process Intensification*, 50 (2011), 281-292.
10. Kiss A. A., Infante Ferreira C. A., *Heat pumps in chemical process industry*, CRC-Press, Taylor & Francis Group, 2016.
11. Kiss A. A., Lange J. P., Schuur B., Brilman D. W. F., van der Ham A. G. J., Kersten S. R. A., *Separation technology - Making a difference in biorefineries*, *Biomass and Bioenergy*, 95 (2016), 296-309.
12. Kujawska A., Kujawski J., Bryjak M., Kujawski W., *ABE fermentation products recovery methods - A review*, *Renewable and Sustainable Energy Reviews*, 48 (2015), 648-661.
13. Luo H., Bîldea C. S., Kiss A. A., *Novel heat-pump-assisted extractive distillation for bioethanol purification*, *Industrial & Engineering Chemistry Research*, 54 (2015), 2208-2213.
14. Luyben W. L., *Control of an azeotropic DWC with vapor recompression*, *Chemical Engineering and Processing: Process Intensification*, 109 (2016), 114-124.
15. Mariano A. P., Costa C. B. B., de Angelis D. D. F., Mauger Filho F., Atala D. I. P., Wolf Maciel M. R., Maciel Filho R., *Optimisation of a continuous flash fermentation for butanol production using the response surface methodology*, *Chemical Engineering Research and Design*, 88 (2010), 562-571.
16. Patraşcu I., Bîldea C. S., Kiss A. A., *Dynamics and control of a heat pump assisted extractive dividing-wall column for bioethanol dehydration*, *Chemical Engineering Research and Design*, 119 (2017), 66-74.
17. Patraşcu I., Bîldea C. S., Kiss A. A., *Eco-efficient butanol separation in the ABE fermentation process*, *Separation and Purification Technology*, 177 (2017), 49-61.
18. Patraşcu I., Bîldea C. S., Kiss A. A., *Eco-efficient downstream processing of biobutanol by enhanced process intensification and integration*, *ACS Sustainable Chemistry & Engineering*, 6 (2018), 5452-5461.
19. Rewagad R. R., Kiss A. A., *Dynamic optimization of a dividing-wall column using model predictive control*, *Chemical Engineering Science*, 68 (2012), 132-142.
20. Xue C., Zhao J-B., Liu F-F., Lu C-G., Yang S-T., Bai F-W., *Two-stage in situ gas stripping for enhanced butanol fermentation and energy-saving product recovery*, *Bioresource Technology*, 135 (2013), 396-402.

1 **Tables**

2

3 **Table 1.** Units sizing and dynamic simulation models (see Figure 7 for the whole flowsheet)

Flowsheet unit (Aspen Plus model)	Specifications
DWC-L (RadFrac)	32 stages. Top pressure: 1.13 bar; Pressure drop: 0.01 bar/stage Hydraulics: simple tray, 1.06 m diameter; stage area 0.808 m ² Sump: Height 2.6 m, Diameter 1.3 m
DWC-R and AEW (RadFrac)	45 stages. Top pressure: 1 bar; Pressure drop: 0.01 bar/stage Hydraulics: <ul style="list-style-type: none"> - simple tray (1–12), 2.08 m diameter; stage area 3.076 m² - packed tray (13 – 14), 0.98 m diameter; stage area 0.762 m² - simple tray (15 – 45), 1.09 m diameter; stage area 0.847 m² Sump: Height 2.76 m, Diameter 1.38 m Reboiler duty: 1971 kW
Hex1 (HeatX)	Simulation mode: instantaneous; Countercurrent Hot valid phases: vapor-liquid; Cold valid phases: vapor liquid Area 456.4 m ² ; U = 0.338 kW/m ² /K; LMTD=17.48 °C; Duty = 2.7 MW
Hex2 (HeatX)	Simulation mode: instantaneous; Countercurrent Hot valid phases: vapor-liquid; Cold valid phases: vapor-liquid Area 345.9 m ² ; U = 0.914 kW/m ² /K; LMTD=18.31 °C Duty = 5.79 MW
Hex3 (HeatX)	Simulation mode: instantaneous; Countercurrent Hot valid phases: vapor-liquid; Cold valid phases: vapor-liquid Area 56.27 m ² ; U = 0.85 kW/m ² /K; LMTD=13.67 °C Duty = 0.65 MW
Hex4 (HeatX)	Simulation mode: instantaneous; Countercurrent Hot valid phases: vapor-liquid; Cold valid phases: vapor-liquid Area 70.86 m ² ; U = 0.85 kW/m ² /K; LMTD=13.40 °C Duty = 0.81 MW
FL (Flash2)	Valid phases: vapor-liquid Height 2.12 m, Diameter 1.06 m Duty: 0.301 MW
Cond aux (Heater)	Simulation mode: instantaneous; Valid phases: vapor-liquid Out Temp: 115.5 °C; Pres. drop: 0.01 bar Duty: -0.68 MW
Cool1 (Heater)	Simulation mode: instantaneous; Valid phases: liquid Out Temp.: 40 °C; Pres. drop: 0.01 bar

	Duty: -1634 kW
Cool2 (Heater)	Simulation mode: instantaneous; Valid phases: liquid Out. Temp.: 25 °C; Out. pres.: 1 bar Duty: -471 kW
Cool3 (Heater)	Simulation mode: instantaneous; Valid phases: liquid Out. Temp.: 25 °C; Out. pres.: 1 bar Duty: -622 kW
Cool4 (Heater)	Simulation mode: instantaneous; Valid phases: liquid Out Temp.: 31.73°C; Pres. drop: 0.01 bar Duty: -319 kW
Comp (Compressor)	Model: Isentropic Discharge pressure: 5.8 bar Brake power: 1653 kW
Decanter (Decanter)	Valid phases: liquid-liquid Pressure: 1.12 Height 3.29 m; Diameter 1.64 m
Reflux drum (Mixer)	Simulation mode: mixer Valid phases: liquid Pres.: 5.76 bar Height 3.57 m; Diameter 1.78 m

1

2

1

2 **Table 2.** Pumps and valves characteristics (pressure driven simulation)

Pressure change devices			Pump				Valve		
			$F_{\max}$	$H_{\max}$	BPower	ΔP		$C_{0,\max}$	ΔP
			[m ³ /h]	[m]	[kW]	[bar]		[m ^{1.5} kg ^{0.5} /h·bar ^{0.5}]	bar
	FEED						V0	1012.08	5.99
DWC-L	BOTTOM	P1	27.85	22.70	2.78	2.02	V1	1678.28	2.00
	FLASH	P2	22.51	56.21	5.85	5.00	V2	830.68	5.00
DWC-R	BOTTOM	P3	7.17	72.72	2.69	5.00	V3	240.39	5.00
	SIDE STREAM	P4	20.93	48.65	4.43	4.00	V4	858.42	3.99
	REFLUX						V7	1893.14	4.76
	DISTILLATE						V8	70.20	4.76
DECANTER	ORGANIC	P5	10.70	48.10	2.75	4.00	V5	461.00	3.97
	AQUEOUS	P6	8.78	42.27	2.43	4.00	V6	5399.49	3.98

3

Table 3. Controller tuning parameters

Controller	PV. value & range	OP. value & range	Kc. %/%	Ti. min
FEED				
FC	Flow rate = 26971 kg/h 0...53942 kg/h	Valve opening = 50% 0...100%	10	20
TC	Preheated feed temperature 97.16 = kg/h 75...115 °C	Aux cool duty = -2.47 GJ/h -12.94...-0.2	20	5
DWC-L				
TC-5	Stage 25 temperature = 108.69 °C 95...120 °C	Duty LV = 1.08 GJ/h 0.2 ... 15.16	1.5	5
LC	Liquid level= 1.62 m 0...3.24 m	Valve opening = 50% 0...100%	10	60
FLASH (vas) LV				
LC	Liquid level = 1.32 m 0...2.65 m	Valve opening = 50% 0...100%	10	60
COMPRESSOR				
PC	Pressure in column = 1 bar 0 ... 2 bar	Compressor Power = 1653.3 kW 0...3306.3 kW	5	20
DWC-R				
Ratio-feed stream:side sstream				
Ratio = 0.49521026				
FC side stream	Side stream flow rate = 17551.2 kg/h 0...35100 kg/h	Valve opening = 50% 0...100%	1	20
TC - 5	Stage 5 temperature = 79.45 °C 60...100 °C	Valve opening = 50% 0...100%	5	20
TC-40	Stage 40 temperature = 126.78 °C 100...150 °C	Duty = 7.297 GJ/h 0 ... 14.59 GJ/h	10	10
LC	Liquid level (sump) = 1.73 m 0...3.46 m	Valve opening = 50% 0...100%	1	20
LC	Liquid level (reflux drum) = 2.23 m 0...4.47 m	Valve opening reflux = 50% 0...100%	5	20
CC-B	Butanol product purity = 0.994 %wt. 0.99...0.998 %wt.	Stage 40 temperature = 126.78 °C 100...150 °C	0.5	55
CONDENSER				
TC	Temperature = 31.735 °C 20...40 °C	Duty = -1.151 GJ/h -25...-0.2	20	20
DECANTER				
LC1	Liquid 1 level = 2.057 m 0...4.11 m	Valve opening = 50% 0...100%	10	60
LC2	Liquid 2 level = 1.004 m 0...2.01 m	Valve opening = 50% 0...100%	10	60
TC	Temperature = 40 °C 20...60 °C	Duty = -5.9 GJ/h -11.78...0 GJ/h	5	20
PRODUCT COOL				
TC	Temperature butanol = 25 °C 10...40 °C	Duty = -1.69 GJ/h -3.387...0 GJ/h	1	20
TC	Temperature water = 25 °C 10...40 °C	Duty = -2.252 GJ/h -4.507...0 GJ/h	1	20

Figure captions

Figure 1. Process flowsheet and mass balance of heat pump assisted A-DWC (heat integrated process) for butanol purification

Figure 2. Temperature (left) and mass composition (right) profiles

Figure 3. Temperature-duty diagrams of the heat-exchangers

Figure 4. Control structure of the heat-integrated, heat pump assisted A-DWC for butanol purification – basic controllers

Figure 5. Stability test of the basic control structure, for non-persistent disturbance - 5% change of the feed flow rate for 10 min

Figure 6. Stability test of the basic control structure, for persistent disturbance - 5% disturbance in feed flow rate

Figure 7. Control structure of the heat-pump assisted A-DWC, after including the additional reboiler and condenser

Figure 8. Stability test of the control structure shown in Figure 7, for persistent disturbance - 10% change of the feed flow rate

Figure 9. Performance of the control shown in Figure 7, in terms of product purities. Top - 10% feed flow rate disturbances. Bottom - feed concentration disturbances.

Figure 10. Control structure of the heat-pump assisted A-DWC, using concentration control

Figure 11. Performance of the control structure shown in Figure 10. Top - 10% feed flow rate disturbances. Bottom - feed concentration disturbances.

Figure 1. Process flowsheet and mass balance of heat pump assisted A-DWC (heat integrated process) for butanol purification.

Figure 2. Temperature (left) and mass composition (right) profiles

Figure 3. Temperature-duty diagrams of the heat-exchangers Hex1 and Hex2

Figure 4. Control structure of the heat-integrated, heat pump assisted A-DWC for butanol purification – basic controllers

Figure 5. Stability test of the basic control structure, for non-persistent disturbance - 5% change of the feed flow rate for 10 min

Figure 6. Stability test of the basic control structure, for persistent disturbance - 5% disturbance in feed flow rate

Figure 7. Control structure of the heat-pump assisted A-DWC, after including the additional reboiler and condenser

Figure 8. Stability test of the control structure shown in Figure 7, for persistent disturbance - 10% change of the feed flow rate

Figure 9. Performance of the control shown in Figure 7, in terms of product purities. Top - 10% feed flow rate disturbances. Bottom - feed concentration disturbances.

Figure 10. Control structure of the heat-pump assisted A-DWC, using concentration control

Figure 11. Performance of the control structure shown in Figure 10. Top - 10% feed flow rate disturbances. Bottom - feed concentration disturbances.