Dynamic study in enhancing the controllability of an energy-efficient double side-stream ternary extractive distillation of acetonitrile/methanol/benzene with three azeotropes

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Abstract: The exploration of control strategy for the double side-stream ternary extractive distillation (denoted as DSTED) process is essential because it has a significant potential for energy-saving. However, how to effectively control the energy-efficient DSTED process is a unique and complex issue due to the additional coupled variables of double side-streams. Consequently, in this study, control schemes of the energy-efficient DSTED process for separating acetonitrile/methanol/benzene with multi-azeotrope are investigated. Firstly, a dual-temperature control strategy is proposed based on the existing single temperature control with feedforward scheme. Temperature difference control strategy is then studied to effectively reduce the offset of product purities for the disturbances of feed composition. Following that, a double-temperature difference control strategy is studied to maintain the product purities. Finally, the comparisons of product purities and energy consumption are introduced to clearly assess the stability and controllability of the different temperature control structures. Dynamic performances show that the

temperature difference control structure has the best performance facing the ±10% feed disturbances.

1. Introduction

Extractive distillation [1-6], pressure-swing distillation [7-9] and azeotropic distillation [10-13] could be used to separate azeotropic system to recover the available resources and protect the environment. The pressure-swing distillation process has higher equipment requirements and it is limited to separate the pressure-sensitive systems [14] while the multiple steady-state issue and huge reflux rate exist in the azeotropic distillation process [15]. Extractive distillation could increase the relative volatility of azeotropic systems by introducing a third component (also called as entrainer), which makes separation easier. Therefore, the extractive distillation is more suitable to separate azeotropic or close-boiling mixture [16-21], for example, binary azeotropic system involving tert-butanol and isopropanol dehydration process [22, 23]. Several separation configurations are explored to separate ternary system acetonitrile/methanol/benzene with multiple azeotropes [24]. Subsequently, a systematic method for separating ternary mixture ethyl acetate/ethanol/water with multiple azeotropes via the extractive distillation is reported by Yang et al. [25].

The intensified distillation processes should be developed because more and more attention has been paid to energy, environment, economy and sustainable development [26-29]. For example, the separation of ethanol/water via the extractive dividing wall column (EDWC) scheme is reported and they illustrated that the energy consumption of the EDWC configuration could decrease about 17% [30]. The EDWC configuration could also been applied to separate benzene/cyclohexane [31] and they proved that the total annual cost (TAC) of the intensified scheme is decreased by 4.8%.

Following that, a novel extractive distillation configuration by the combination of the EDWC and decanter is developed to separate complex heterogeneous system methanol/toluene/water and they illustrated that the TAC could be decreased by 15.14% [32]. Furthermore, an intensified side-stream extractive distillation configuration for the separation of binary systems is proposed [33, 34] and their results shown that the proposed process has significantly advantages in energy-saving and emission reduction. Afterwards, the side-stream extractive distillation configuration has been further investigated to separate acetonitrile/methanol/benzene mixture [24] and the calculation displayed that the energy-saving of the side-stream scheme is the best in all alternative configurations (i.e., thermally coupled and side-stream). In summary, energy consumption of the intensified separation techniques could be effectively reduced, however, it will make the control strategy of the intensified scheme become more complex than the conventional scheme. Therefore, it has far-reaching significance to explore an effective control strategy for the energy-saving extractive distillation separation sequences.

In addition, dynamic controllability investigations of the intensified distillation configurations have been also extensively explored in literatures. For instance, a novel control scheme with non-linear solvent/feed for extractive pressure-swing distillation is studied by Luyben [35]. Two basic and an improved robust control schemes are reported to explore the dynamic controllability of the EDWC process for separating azeotropic mixture 2-methoxyethanol/toluene [36] and then several temperature control structures are also studied [37]. Economics and dynamic controllability for the side-stream and EDWC are compared and only 5% of feed flowrates disturbances are considered by Wang et al. [38]. Subsequently, Yang et al. [25] discussed several effective temperature control schemes to achieve the control of product purities for the triple-column extractive distillation scheme.

Lately, the dynamic comparison of the conventional and the intensified extractive distillation schemes are explored by Zhang et al. [39]. The dynamic investigation of the triple-column extractive distillation with heat integration has been explored by Shi et al. [40].

In recent, several alternative intensified separation sequences (e.g., side-stream distillation process) have been proposed for the separation of ternary mixture with multiple azeotropes and they demonstrated that the intensified side-stream distillation configuration is the most promising among those intensified configurations from the economic and environmental protection aspects [41]. Afterwards, a control strategy with several composition control controllers for the intensified DSTED scheme is explored to maintain the product purities [38]. Nevertheless, the composition controller have to be abandoned because the real time online device is expensive and time consuming [42]. Therefore, four simple temperature control strategies are explored to handle the product purities in our recently work [43]. However, the transient deviations and offsets of product purities for two disturbances have not achieving the expected effect for the intensified DSTED scheme because the temperature of sensitive stage is fixed, and the flow rate of the side-stream has not be controlled. Consequently, more attention should be received for the exploration of the control strategy to accelerate realizing the industrial application of the intensified process.

In this work, we aim to explore the dynamic controllability of the intensified DSTED scheme via a cheap and responsive temperature control structure. Three temperature control schemes (i.e., dual temperature, temperature difference, and double temperate difference control strategies) for the intensified DSTED scheme are explored to handle the product purities. Temperature-sensitive and reference stages of three proposed control structures are determined via the open-loop sensitive analysis and criteria averaged absolute variation magnitudes. To assess the stability and robustness of

the three improved control schemes, the comparisons of product purities and reboiler duty are used when the disturbances are introduced.

2. Simulation of steady-state and existing control scheme

Steady-state simulation for separating the mixture acetonitrile/methanol/benzene (A/M/B) via the energy-efficient DSTED configuration is reported by Wang et al. [41]. Well-known, the determination of thermodynamic model is a key to the accuracy of the calculation [44, 45]. Following the study of Wang et al. [41], the Wilson model is selected to calculate the vapor-liquid equilibrium of A/M/B using chlorobenzene (CB) as solvent. The built-in binary interaction parameters for the A/M/B/CB in Aspen Plus are shown in Table S1. Fig. 1 represents the thermodynamic features (e.g., residue curve maps) for quaternary system A/M/B/CB under 1 bar.
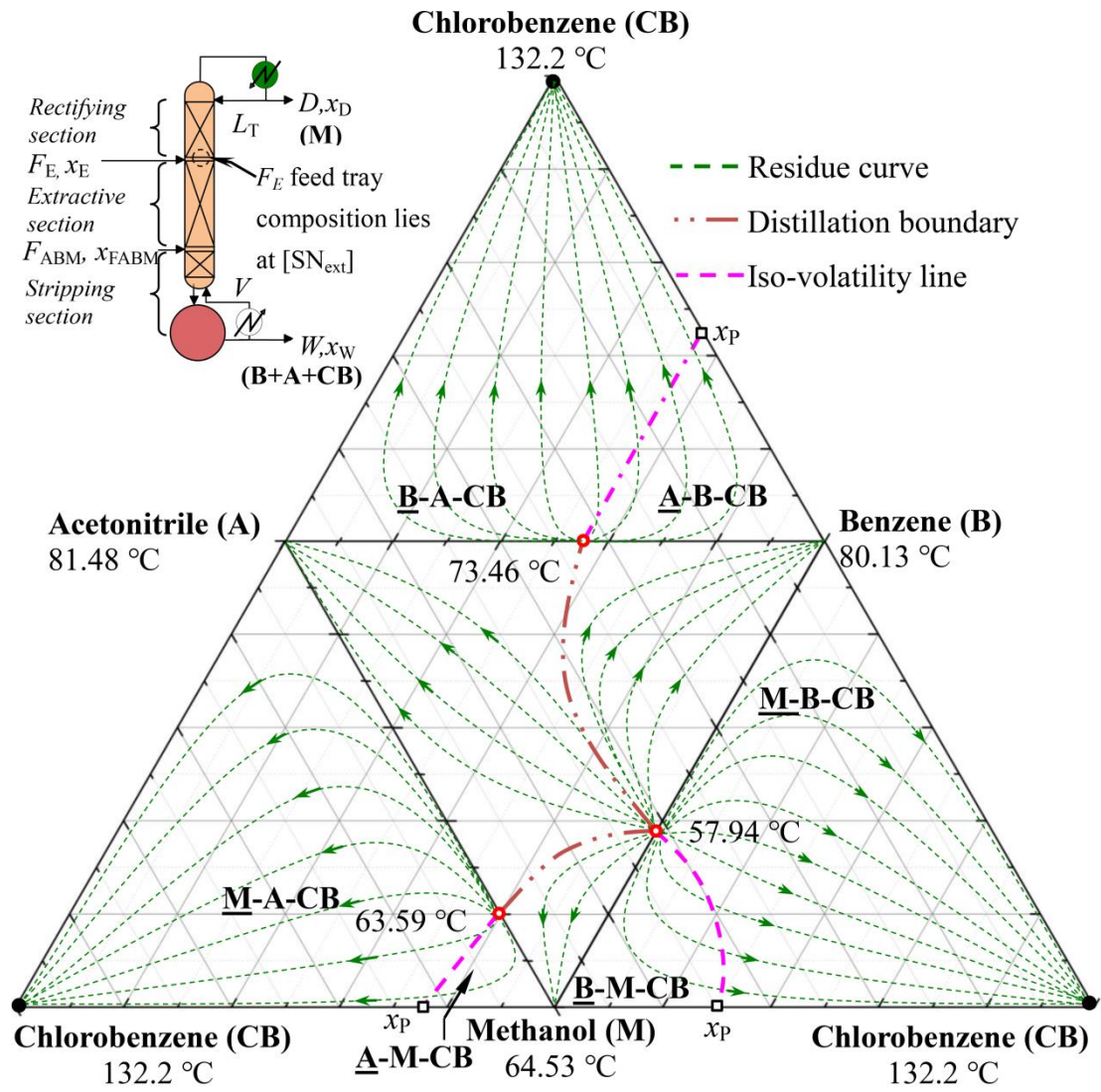
Fig. 1. Thermodynamic features of class for ternary mixture A/M/B using CB as solvent at 1 bar
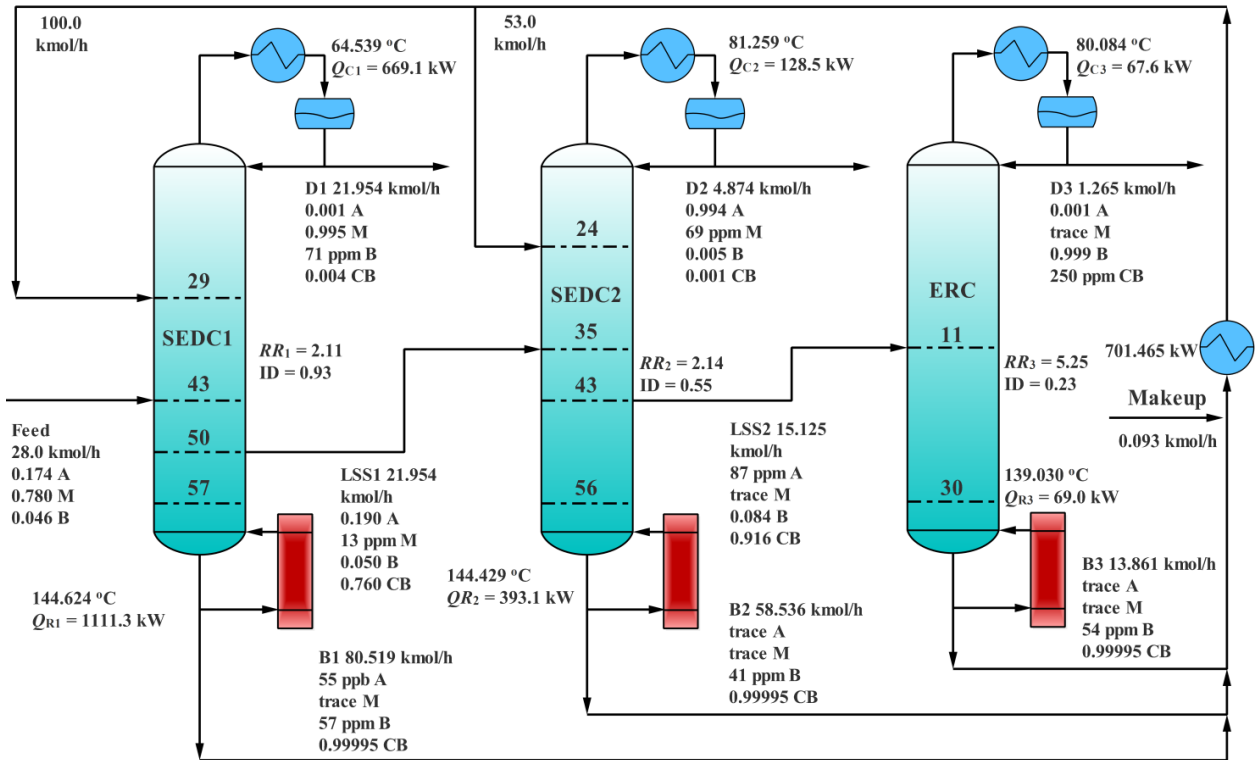
Fig. 2. The flowsheet of the intensified DSTED process for the separation of A/M/B

The scheme of the intensified DSTED process with detailed operating conditions for separating the ternary azeotropic mixture A/M/B using entrainer CB is illustrated in Fig. 2. The flow rate of fresh feed is 28.0 kmol/h with 17.4 mol% A, 78.0 mol% M, and 4.6 mol% B. The first side-stream extractive distillation column (SEDC1), second side extractive distillation column (SEDC2) and entrainer recovery column (ERC) have 58, 57, and 31 theoretical stages, respectively. The solvent and fresh feed of the SEDC1 are 29th and 43th tray, respectively. Withdraw stage and the flow rates of side-stream are 50th stage and 21.954 kmol/h. To achieve the separation, reflux ratio of 2.11 is needed for the SEDC1. Similar observations can be made for SEDC2 and ERC from Fig. 2.
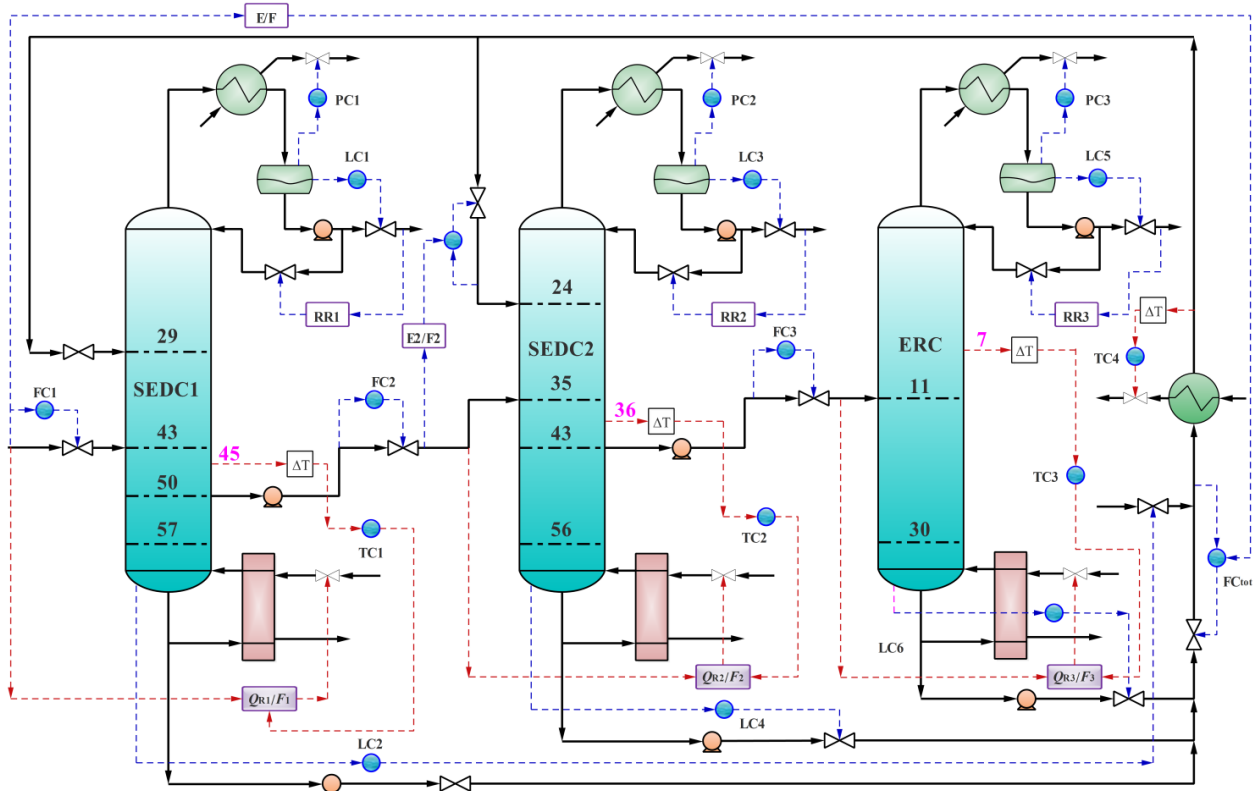
Fig. 3. The existing temperature and feedforward control structure

The existing feedforward control structure with reboiler duty-to-feed flow rate ratio (Q_R/F) in Fig. 3 has been explored in our previous work [43]. Six level controllers (LC1, LC2, LC3, LC4, LC5, and LC6) with gain (K_C) of 2 and integral time (τ_I) of 9999 min [46, 47]. Distillate rate-to-reflux rate ratios of three columns (RR1, RR2, and RR3) are kept as constant in the control process. The flow rate of fresh feed, side-stream 1 and side-stream 2 are controlled via flow controllers FC1, FC2, and FC3 with $K_C = 0.5$ and $\tau_I = 0.3$ min [48-50]. Operating pressures of columns SEDC1, SEDC2 and ERC are controlled by adjusting the flow rate of cooling water (PC1, PC2, and PC3). Temperature of sensitive-stage could be controlled by manipulating the ratio of Q_{R1}/F_1, Q_{R2}/F_2, and Q_{R3}/F_3 (TC1, TC2, and TC3). According to Luyben and Chien [51], the Tyreus–Luyben rule (see in Eqs. (1) and (2)) is used to obtain the K_C and τ_I for all temperature controllers.
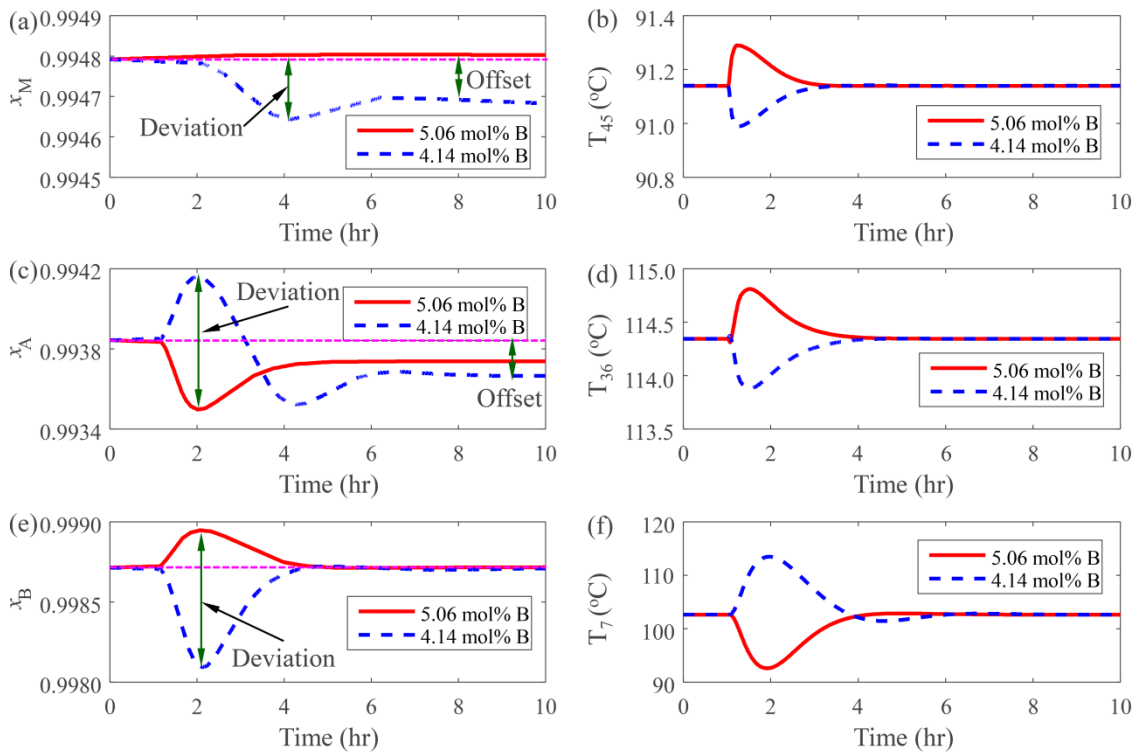
$$K_C = K_U / 3.2 \tag{1}$$

$$\tau_I = 2.2 P_U \tag{2}$$
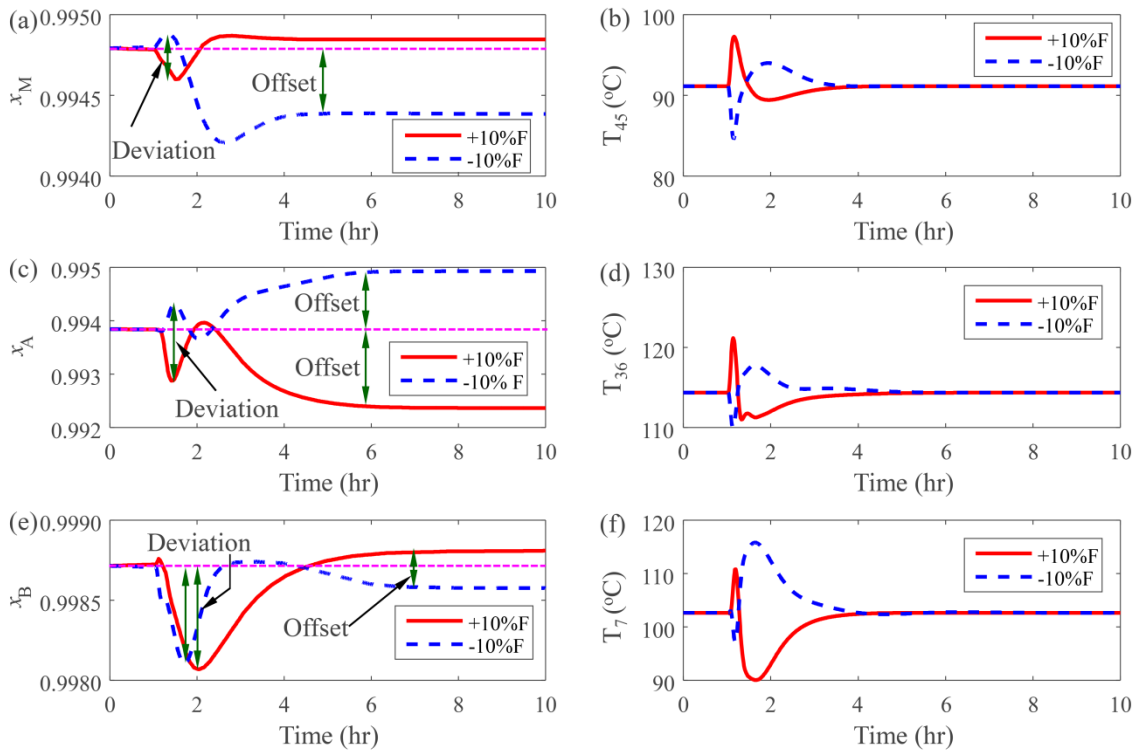
Fig. 4. Dynamic performances of the existing control strategy for ±10% feed flow rate disturbances


Fig. 5. Dynamic performances of the existing control strategy for ±10% feed composition disturbances

Dynamic responses of the existing temperature control structure (TC) for ±10 feed flow rate and

composition disturbances are displayed in Figs. 4 and 5, respectively, which is investigated in our recently work [43]. Form the observation of transient deviations and offsets in Figs. 4 and 5, the TC structure could not well deal with the product purities for both flow rate and composition disturbances because the temperatures of sensitive stages are fixed and the existing control structure only has a flowrate controller for the side-stream. Therefore, more robust control strategy will be explored in the next section.

3. Dynamic control of double side-streams ternary extractive distillation

3.1 Determination of sensitive stage and reference stage
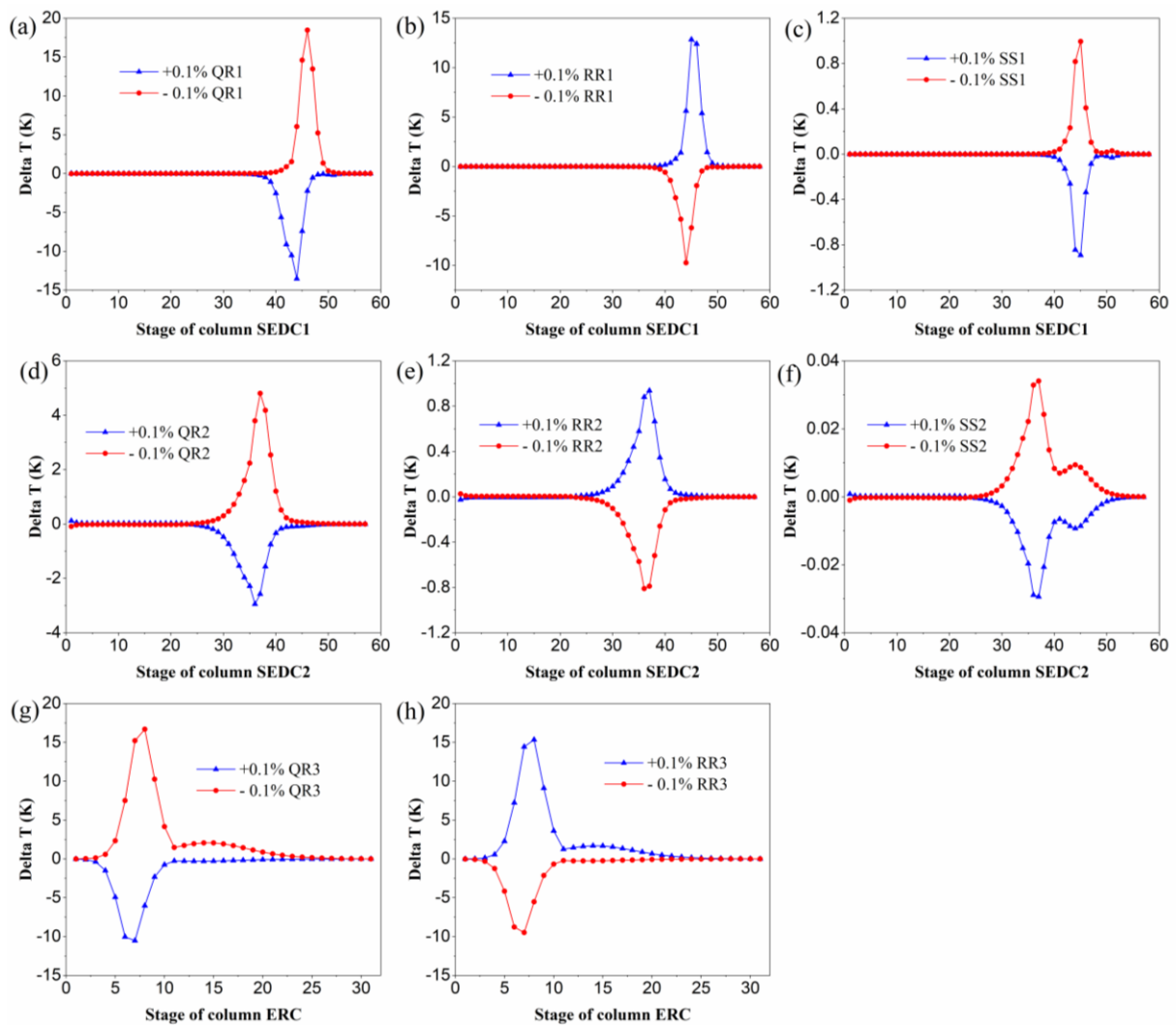
Fig. 6. Open-loop sensitivity analysis by varying manipulated variables

45th, 36th and 7th stages are selected as sensitive stages for controlling the reboiler duty of SEDC1, SECD2 and ERC because they have the large peaks in the result of open-loop sensitivity analysis [43].. 51th and 44th stages are selected as another sensitivity stages to control the flow rate of side stream because they have two peaks in the SEDC1 and SEDC2 and near the side stream as shown in Fig. 6.

Recently, a novel criteria averaged absolute variation magnitudes (ASVM) in Eq. (3) is proposed by Yuan et al. [52] to select the reference temperature stages for temperature difference (TDC) and double-temperature difference (DTDC) control schemes.

$$\text{ASVM} = \frac{1}{2\text{NC}} \sum_i^{2\text{NC}} \alpha_i \left| \Delta \left(T - T_{SS} \right)_i \right| \tag{3}$$

where the number of targeted components is denoted as NC; α_i (i = 1, …, NC) represents the weighting coefficients of rejecting the feed composition disturbances via the potential robust control strategy; T represents the temperature remaining stages; T_{SS} denotes the temperature of sensitive stage.

Following the study of Yuan et al. [49], the selection of reference stage for the TDC and DTDC schemes should be initiated from the sensitive stage, and the nearest stage with a local ASVM minimum should be chosen as the reference stage. The difference between sensitive and reference stages as a constant is employed to handle the feed flowerate and composition disturbances [53].
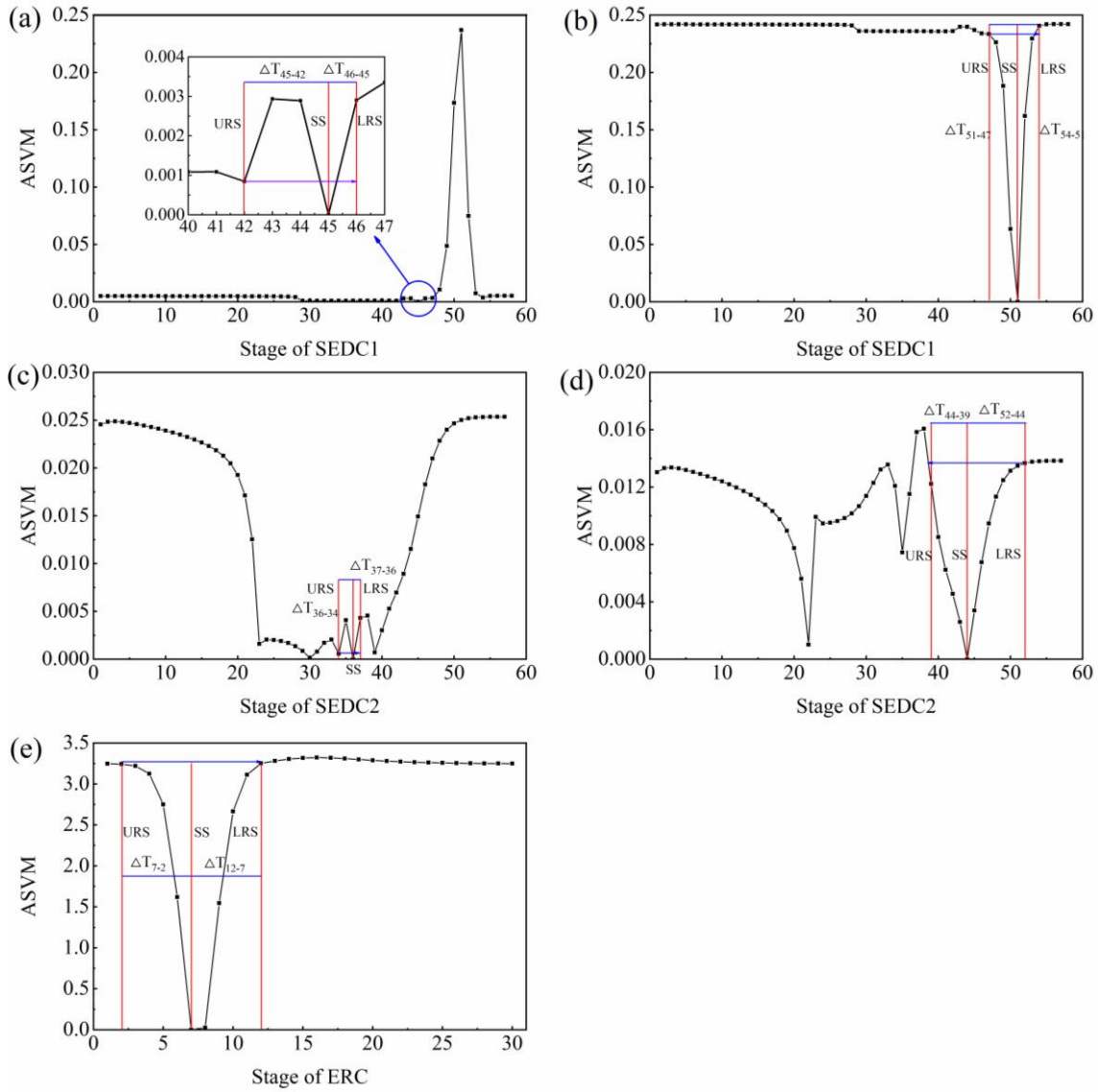
Fig. 7. ASVM for the (a,b) SEDC1, (c,d) SEDC2 and (e) ERC

The calculation of the ASVM for SEDC1, SEDC2 and ERC are displayed in Fig. 7. ASVM of

the sensitive-stage is zero. The first reference stage is determined via searching a slowly change or a

minimum value of ASVM point. The second reference stage could be selected by combing the first

reference stage and sensitive-stage. In addition, the control points of different control loops should

avoid crossing. Finally, the ΔT_{45-42} and ΔT_{51-47} in SEDC1, ΔT_{36-34} and ΔT_{52-44} in SEDC2, and ΔT_{7-2} in

ERC are determined for the temperature difference control (TDC) structure. In addition,

$\Delta T_{(45-42)-(46-45)}$ and $\Delta T_{(51-47)-(54-51)}$ in SEDC1, $\Delta T_{(36-34)-(37-36)}$ and $\Delta T_{(44-39)-(52-44)}$ in SEDC2 and

$\Delta T_{(12-7)-(7-2)}$ in ERC are selected for the DTDC structure.

3.2 Dual temperature control scheme
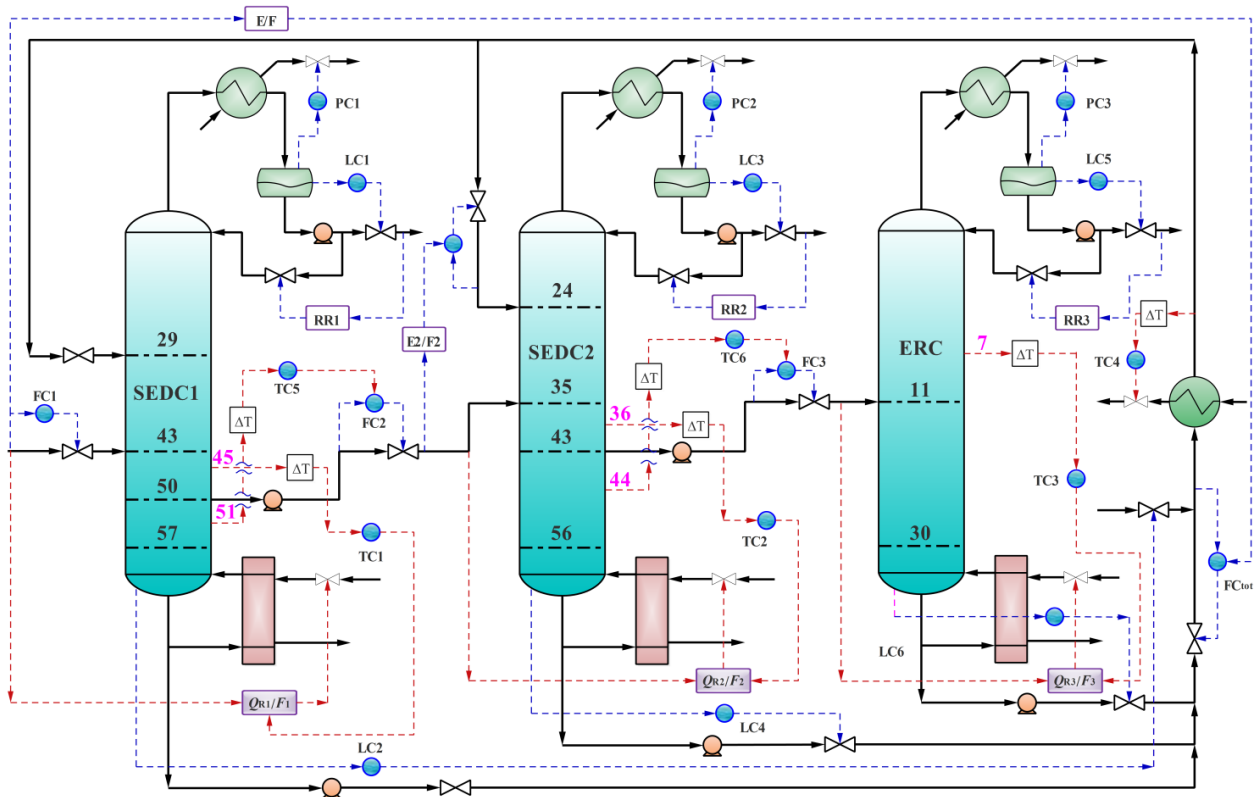
Fig. 8. The dual temperature control scheme for the DSTED process

Fig. 8 represents the dual temperature control (DTC) scheme for the intensified DSTED process.

In this structure, two controllers FC2 and FC3 are used to control the flow rate of two side-streams,

which is cascade control scheme by using the temperature of 51th and 44th stages in SEDC1 and

SEDC2, respectively. Tuning parameters of the DTC structure for the all temperature controllers and

controller action are summarized in Table 1.

**Table 1**. Tuning parameters of the DTC structure for the intensified DSTED process

	TC1	TC2	TC3	TC4	TC5	TC6
Ultimate gain, K_U	1.4038	2.0339	1.0522	0.5165	81.1485	0.5165
Ultimate period, P_U (min)	10.2000	14.4000	12.0000	3.6000	6.0000	3.6000
Gain, K_C	0.4387	0.6356	0.3288	0.1614	25.3589	0.1614
Integral time, τ_I (min)	22.4400	31.6800	26.4000	7.9200	13.2000	7.9200
Controller action	Reverse	Reverse	Reverse	Reverse	Direct	Reverse

Figs. 9 and 10 illustrate the dynamic responses of the DTC strategy for feed flow rate and

composition disturbances. Transient deviations and offsets of three product purities could be effectively reduced compared with the existing TC scheme.
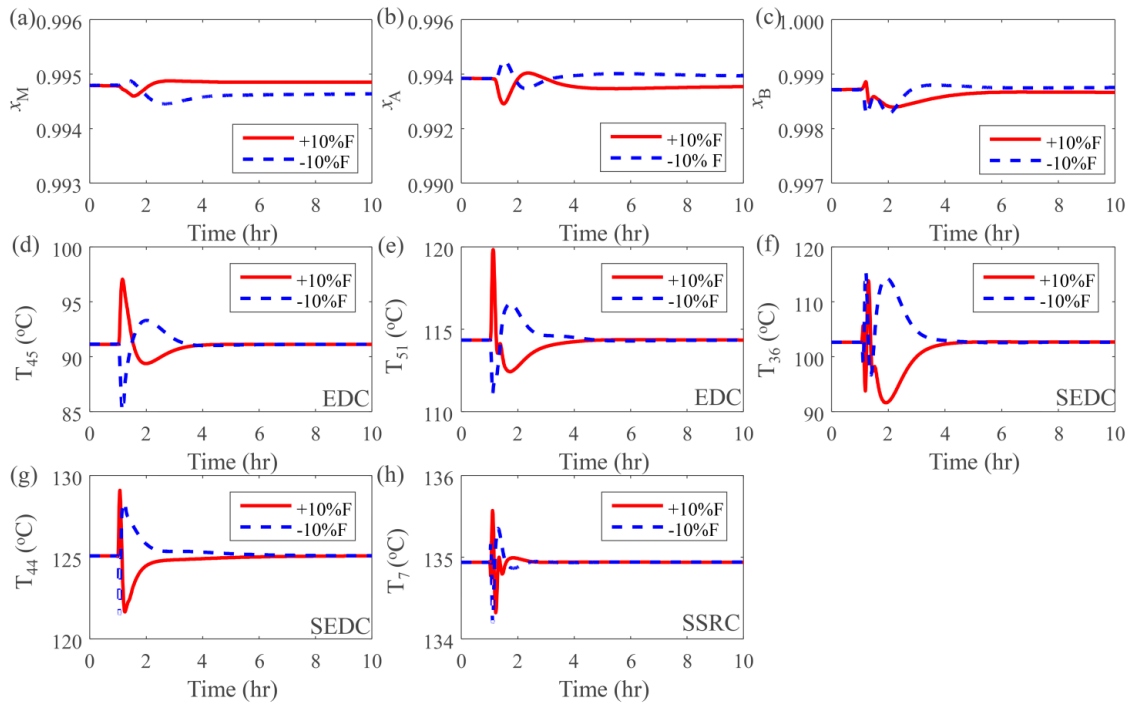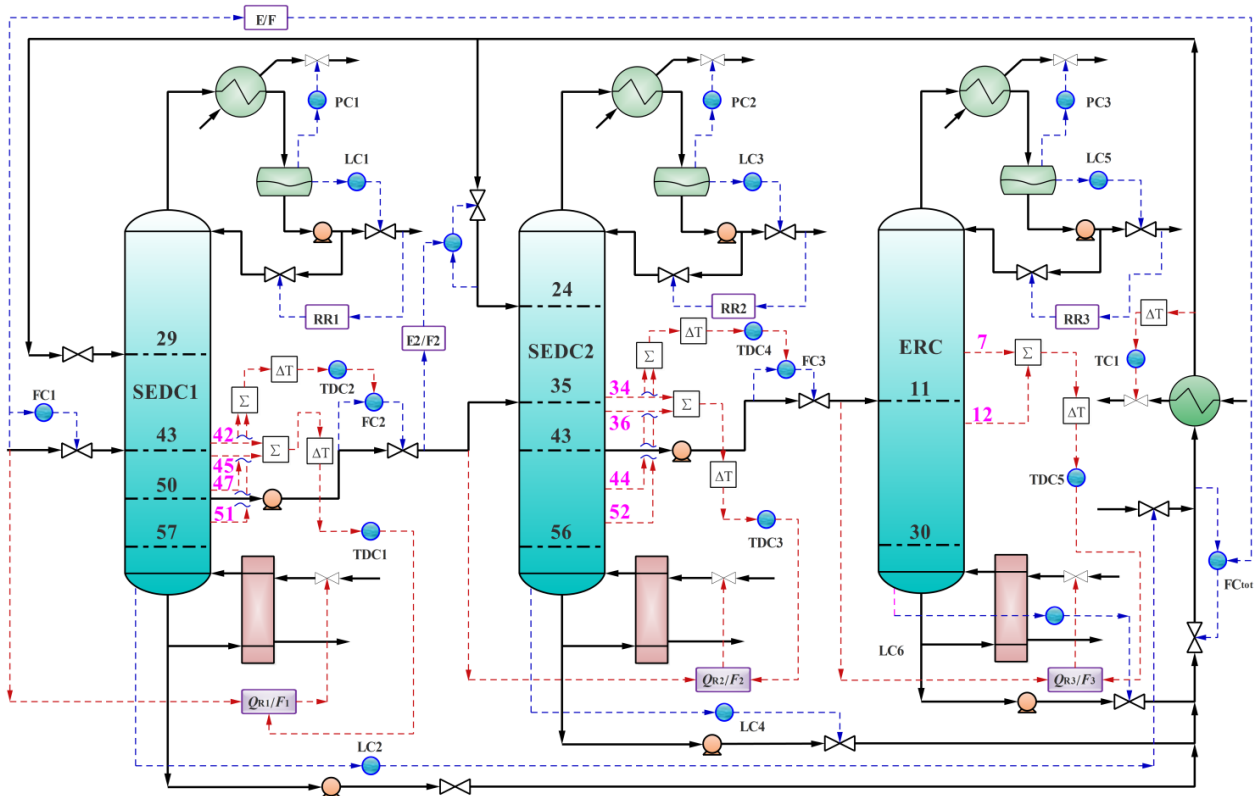

Fig. 9. Dynamic responses of the DTC for the ±10% feed flow rate disturbances
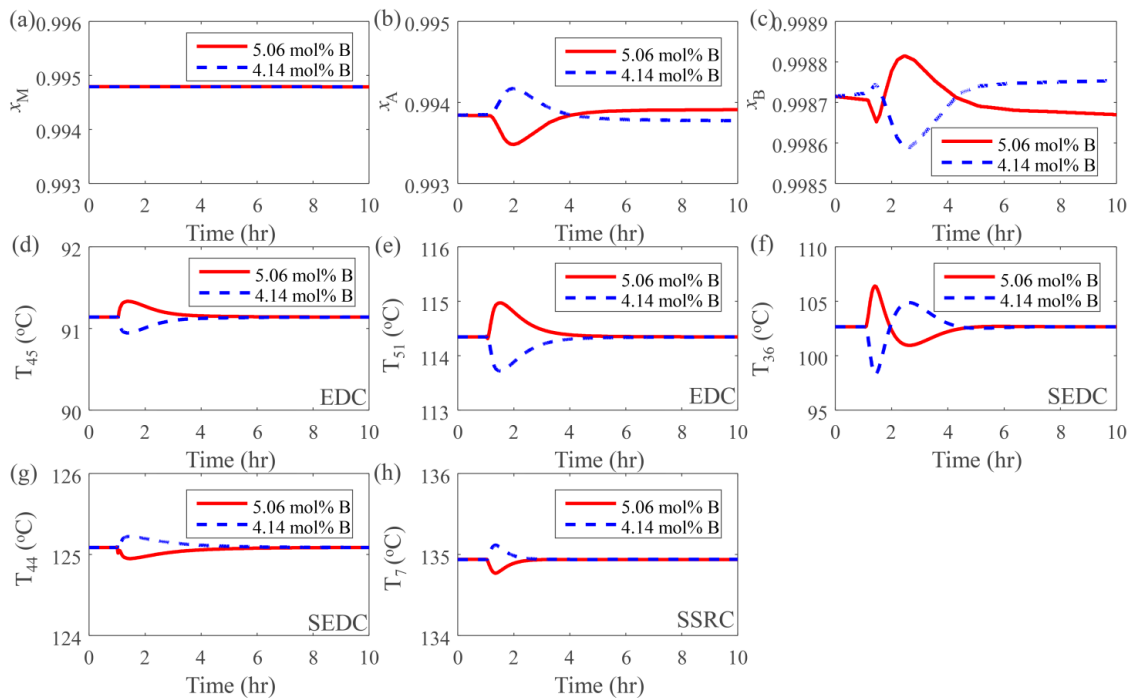
Fig. 10. Dynamic responses of the DTC for the ±10% feed composition disturbances

3.3 Temperature difference control structure

Fig. 11. The TDC structure for the double side-streams ternary extractive distillation process

The TDC structure for the DSTED process is displayed in Fig. 11. Five temperature difference controllers (TDC1, TDC2, TDC3, TDC4, and TDC5) are used in the TDC structure. Of note is that, the controller action of TDC4 and TDC5 is direct and TDC1, TDC2 and TC1 are reverse. Tuning parameters of all temperature controllers are listed in Table 2.

Table 2. Tuning parameters of the TDC structure for the intensified DSTED process

	TDC1	TDC2	TDC3	TDC4	TDC5	TC1
Ultimate gain, K_U	0.2656	0.8173	0.2467	7.0563	0.4314	0.5165
Ultimate period, P_U (min)	9.0000	7.8000	13.8000	5.4000	9.6000	3.6000
Gain, K_C	0.0830	0.2554	0.0771	2.2051	0.1348	0.1614
Integral time, τ_I (min)	19.8000	17.1600	30.3600	11.8800	21.1200	7.9200
Controller action	Reverse	Reverse	Reverse	Direct	Direct	Reverse

To evaluate the control performances, two kinds of disturbances are introduced in the TDC scheme. Dynamic performances for flow rate disturbances are given in Fig. 12. Offsets of three product purities could be decreased compared with the DTC scheme. However, the peak transient

deviations will increase for flow rate disturbances. From Fig. 13, the offsets and the peak transient deviations could be further reduced for the dynamic performances of feed composition disturbance.
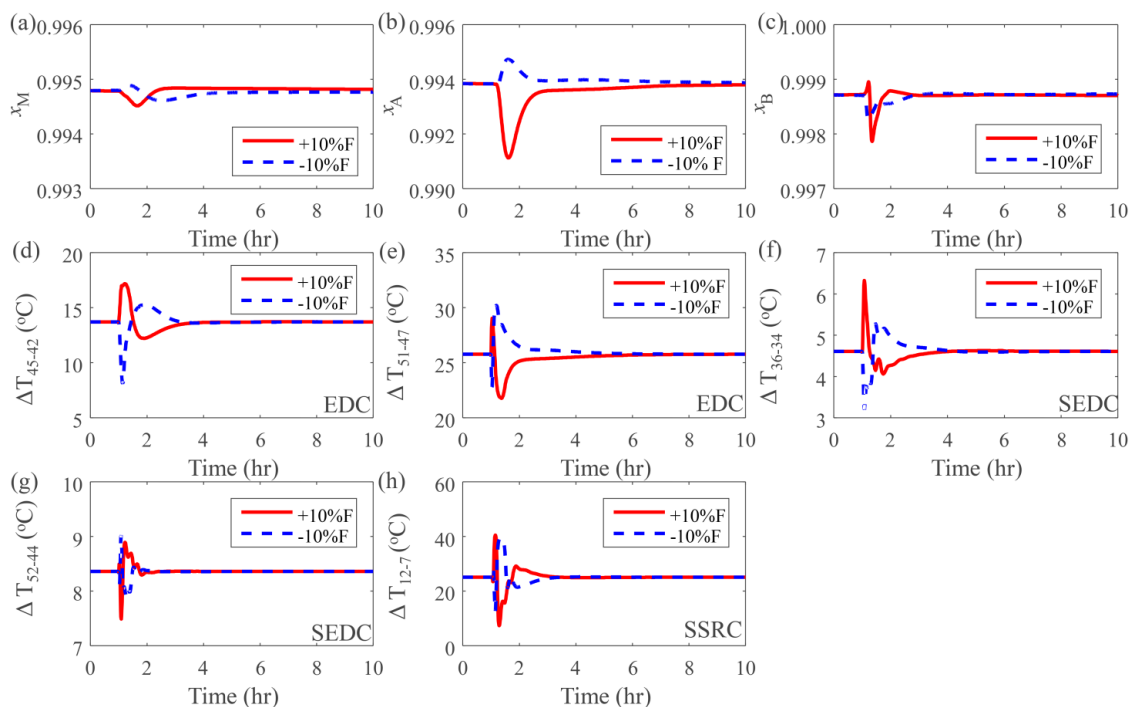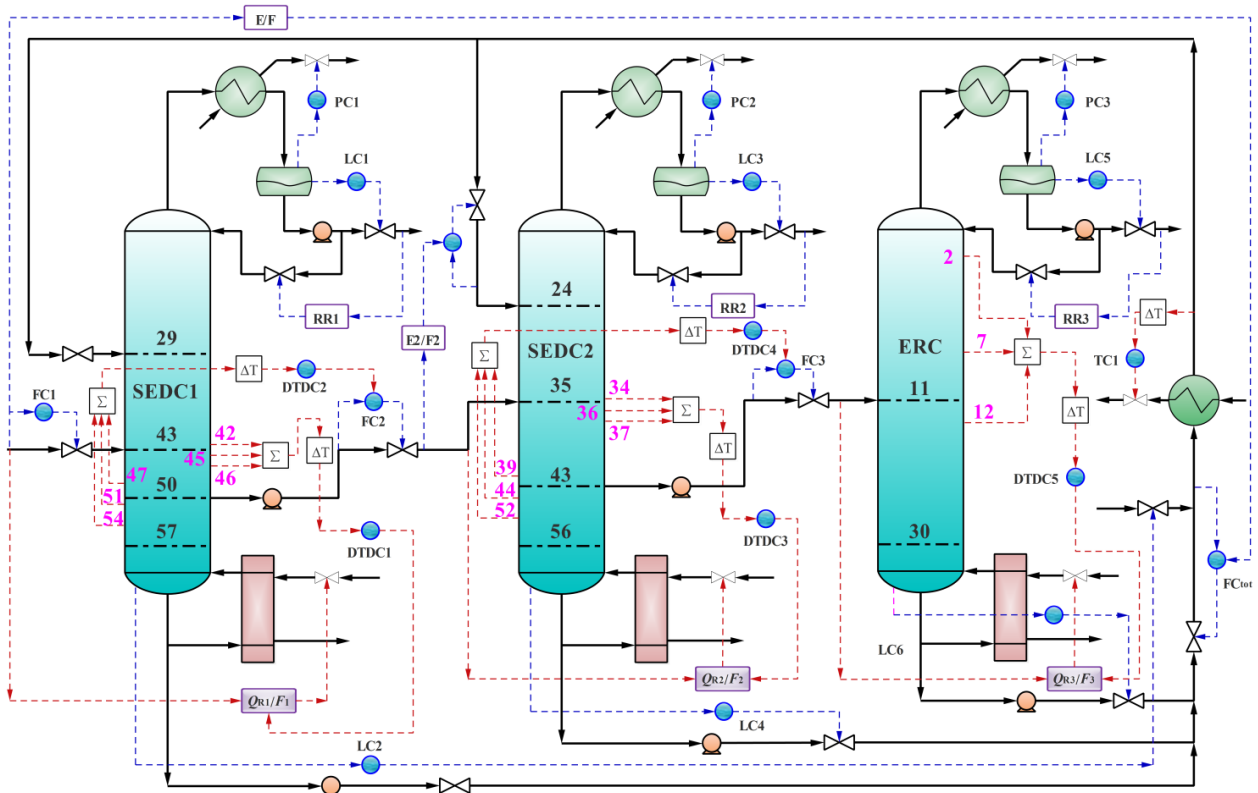

Fig. 12. Dynamic responses of the TDC for the ±10% feed flow rate disturbances
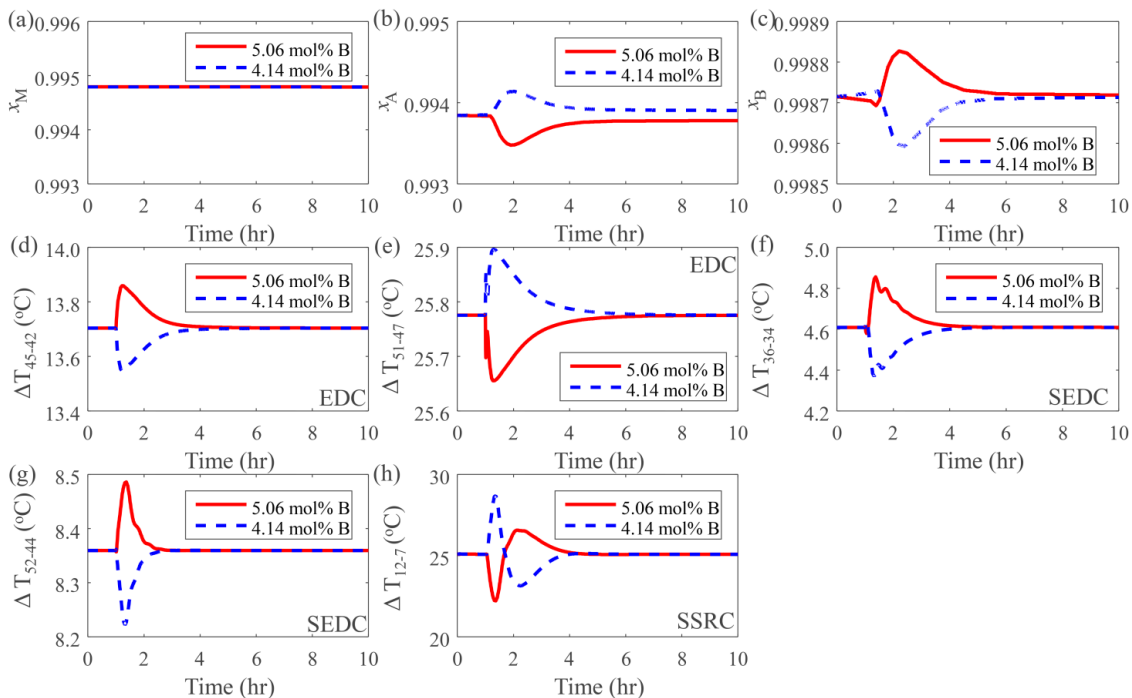
Fig. 13. Dynamic responses of the TDC for the ±10% feed composition disturbances

### 3.4 Double-temperature difference control strategy

Fig. 14. The double-temperature difference control structure for the double side-streams ternary extractive distillation process

Following the study of Yuan et al. [52], Wu et al. [54], and Ling and Luyben [53], control performances may be improved via the DTDC structure. Hence, a DTDC structure in Fig. 14 for the DSTED process is explored. In this scheme, five double-temperature difference controllers (DTDC1, DTDC2, DTDC3, DTDC4, and DTDC5) are used to control the reboiler duty-to-feed flow rate ratio. All tuning parameters of the DTDC scheme are displayed in Table 3.

Table 3. Tuning parameters of the DTDC structure for the intensified DSTED process

	DTDC1	DTDC2	DTDC3	DTDC4	DTDC5	TC1
Ultimate gain, K_U	0.1274	0.1274	0.1274	0.8540	0.1274	0.5165
Ultimate period, P_U (min)	9.6000	7.8000	13.2000	8.4000	11.4000	3.6000
Gain, K_C	0.0398	0.0398	0.0398	0.1825	0.0398	0.1614
Integral time, τ_I (min)	21.1200	17.1600	29.0400	18.4800	25.0800	7.9200
Controller action	Reverse	Reverse	Reverse	Reverse	Direct	Reverse
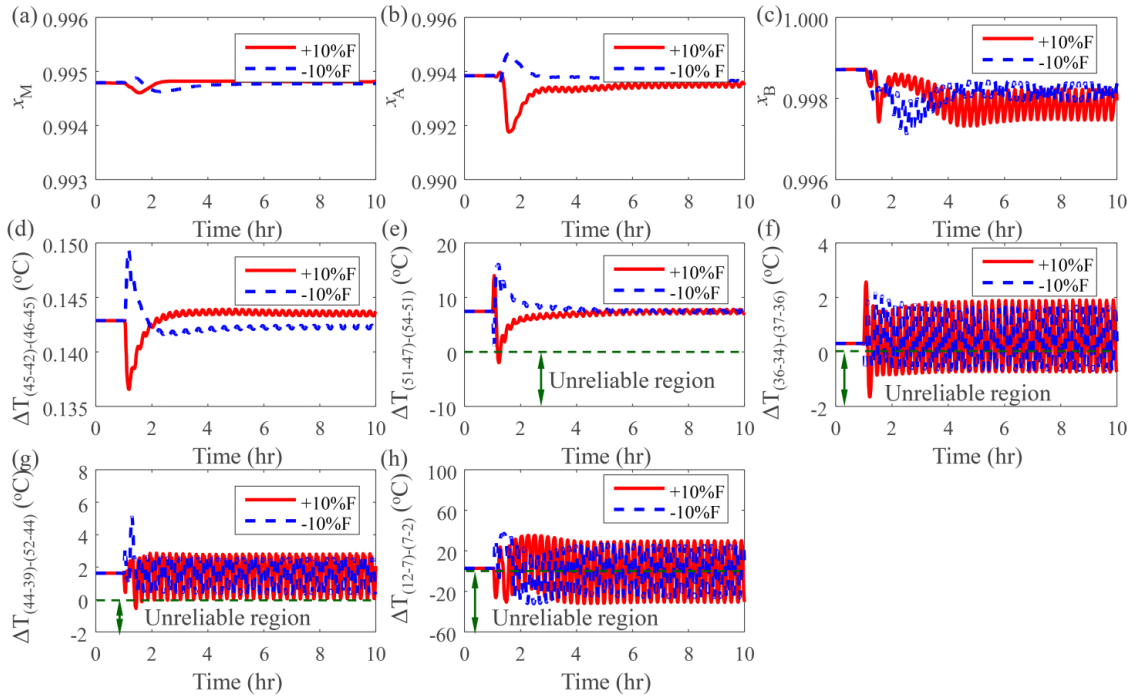
Fig. 15. Dynamic responses of the DTDC for the ±10% feed flow rate disturbances
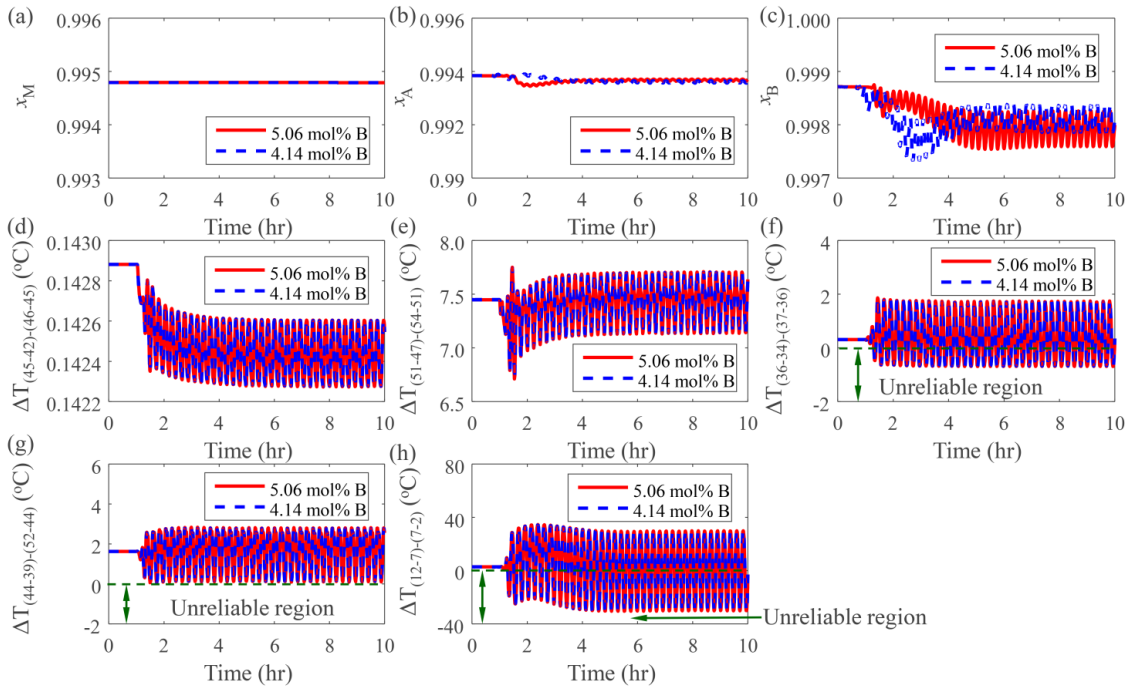

Fig. 16. Dynamic responses of the DTDC for the ±10% feed composition disturbances

Herein, the disturbances of feed flow rate and composition are introduced to assess the dynamic performances of DTDC structure (see Figs. 15 and 16). From Fig. 15e, an unreliable region is occurred for double temperature difference controller because of the value of $\triangle T_{(51-47)-(54-51)} =$

$(T_{51}-T_{47})-(T_{54}-T_{51})$ or $(2\times T_{51}-T_{47}-T_{54})$ is lower than zero. Hence, the output signal of DTDC2 controller is unreliable. Similar observation can be made for other controllers from parts f-h of Figs. 15 and 16. In summary, the product purities could not be well handled via the DTDC structure.

4. Results and discussion
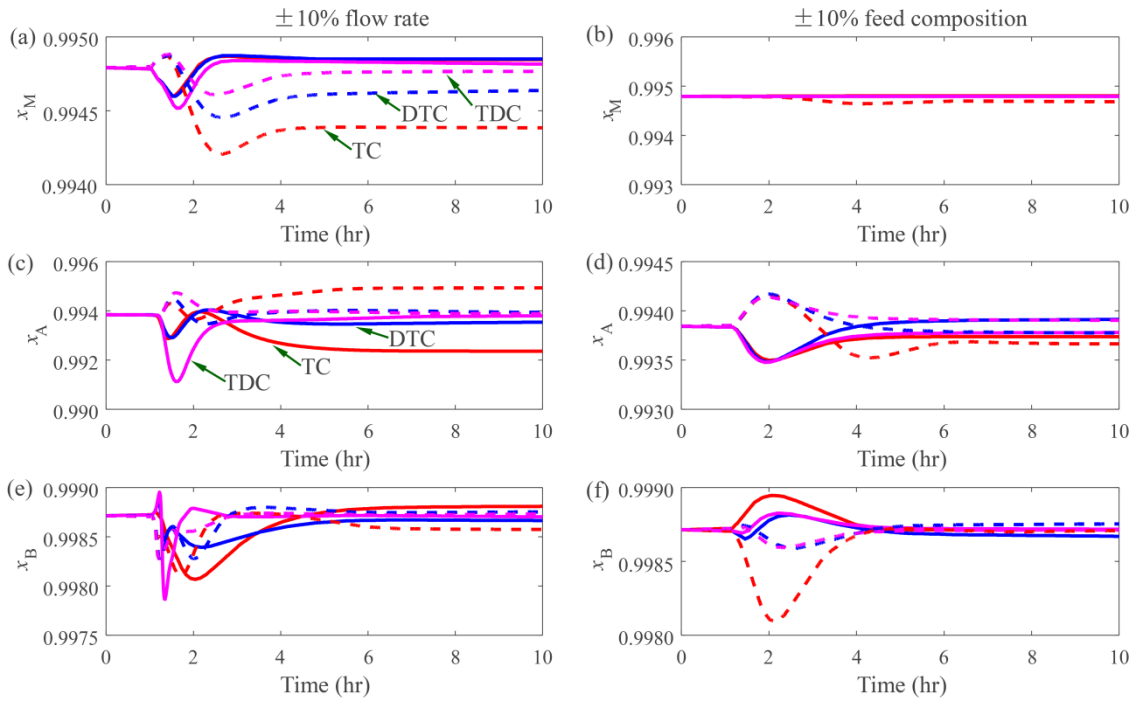
Fig. 17. Comparison of dynamic response for TC, DTC, and TDC schemes

Comparisons of dynamic responses for TC, DTC, and TDC schemes are illustrated in Fig. 17. Form Fig. 17a,c,e, transient deviations of TC and DTC schemes are better than that of the TDC strategy while offsets of the TDC is better that those of TC and DTC schemes. Similar observation of the feed composition disturbances can be made from Fig. 17b,d,f. In summary, the purities of A, B, and M could be well controlled via the improved TDC strategy.
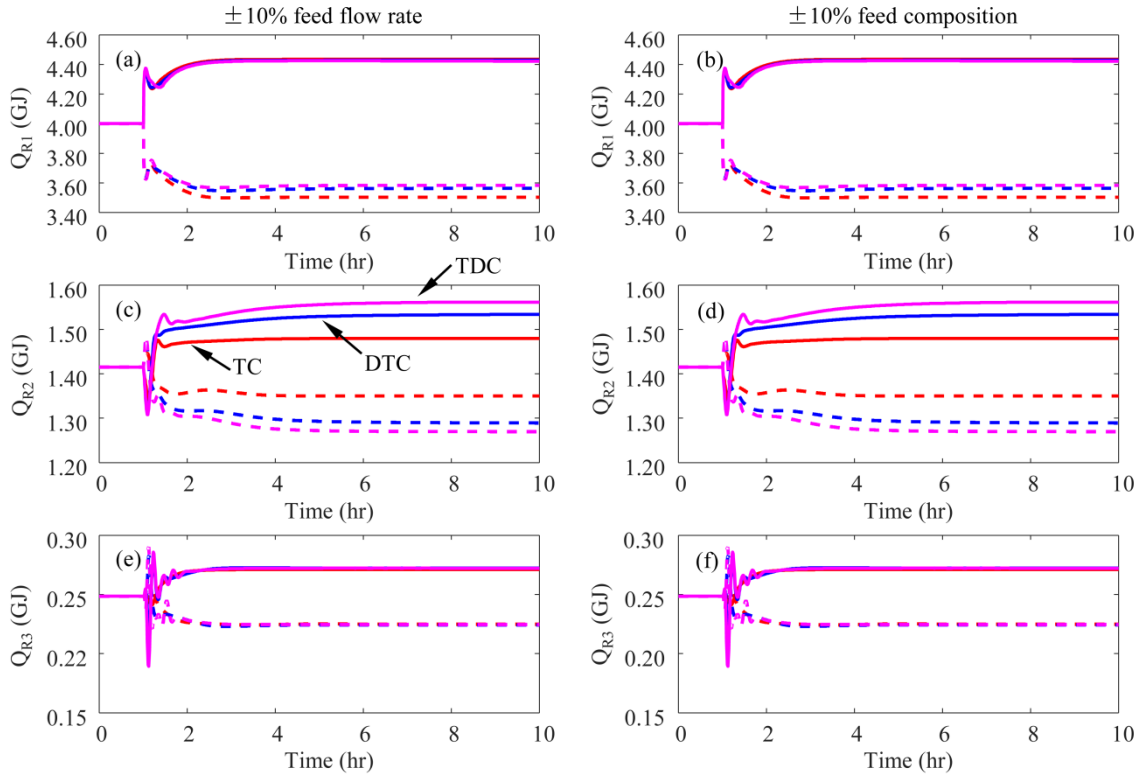
Fig. 18. Comparison of reboiler duty for TC, DTC, and TDC schemes

Table 4. Total energy consumption of the process facing feed flow rate and composition disturbances

	TC	DTC	TDC
+10% Feed flow rate	6.1858	6.2361	6.2566
-10% Feed flow rate	5.0777	5.0767	5.0780
+10% Feed composition	5.6754	5.6741	5.6742
+10% Feed composition	5.6530	5.6551	5.6551

Energy consumptions of three control strategies are also compared to evaluate the economics as displayed in Fig. 18 and Table 4. Reboiler duty of SEDC1 (Q_{R1}) for the TC scheme is higher than that of DTC and TDC structures (see Fig. 18a,b). Of note is that the reboiler duty of SEDC2 for the TDC strategy is higher than other two schemes. Energy consumptions of ERC (Q_{R3}) after smoothing are similar for TC, DTC and TDC strategies, which are shown in Fig. 18e,f. In a word, TDC and TC scheme have the highest and lowest energy consumption, respectively, and the corresponding control effect is opposite.

5. Conclusion

In this work, three enhanced temperature control strategies have been investigated to realize robust control of the intensified DSTED process. To obtain a better control performance, a dual temperature control strategy is firstly investigated based on the existing temperature control scheme. Two improved temperature difference and double temperature difference control structures are then studied to further handle the composition disturbances. Product purities and energy consumption are introduced to compare the dynamic performances of the existing and proposed control schemes. Dynamic responses indicated that product purities could be well controlled via the temperature difference control strategy when the two kinds of disturbances are introduced. In summary, the present investigation will promote the practical application of the energy-saving DSTED process to commerce or industry.

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Notes

The authors declare no competing financial interest.

Supporting Information

Built-in binary interaction parameters of the studied system acetonitrile/methanol/benzene could be found in Table S1.

Nomenclature

A   acetonitrile

ASVM   averaged absolute variation magnitudes

B   benzene

CB   chlorobenzene

DWC   dividing wall column

DTC   dual temperature control

DTDC   double temperature difference control

EDWC   extractive dividing wall column

FC   flow rate controller

K_C gain

M   methanol

PCi   pressure controller i

Q_R/F reboiler duty-to-feed flow rate ratio

SEDC   side extractive distillation column

ERC   entrainer recovery column

TAC   total annual cost

TC   temperature control

TDC   temperature difference control

TCi   temperature controller i

T_{SS} temperature of sensitive stage

τ_I integral time, min

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