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Article

Modelling and Feedforward Control of Pulsed Bed Adsorption Column for Colorant Removal in Sugar Syrup

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Abstract. In sugar refining, pulsed bed adsorption columns are used to remove colorants in sugar syrup. The pulsed bed adsorption column is a cyclic steady state operation that maximize the adsorbent utilization due to the increased driving force but requires special attention on control and optimization. The modelling and feedforward control of the pulsed bed adsorption columns is investigated in this article. The aim of this research is to develop the mathematical model of pulsed bed adsorption column by using the parameters obtained from breakthrough of fixed bed column and to investigate the optimum cycle time at different colorant concentrations in feed sugar syrup. The analysis showed that the optimum cycle time of pulsed bed adsorption column depended on feed flow rates and the colorant concentrations in feed sugar syrup. As the feed flow rate and colorant concentration increased, the optimum cycle time decreased. The feedforward control successfully compensated the effects of disturbances from colorant concentrations in feed syrup for both step and cosine functions and yielded the sugar syrup that met its specification.

Keywords: Pulsed bed, cyclic steady state, optimum cycle time, feedforward control, sugar refining.

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Nomenclature

C	The outlet concentration [dimensionless]
C_0	The concentration at initial time in liquid phase [ICUMSA]
C_{eq}	The concentration at equilibrium in liquid phase [ICUMSA]
C_{inlet}	The inlet concentration [ICUMSA]
$C_{model,i}$	The outlet concentration from experiment at time i [dimensionless]
$C_{exp,i}$	The outlet concentration from mathematical model at time i [dimensionless]
k	The mass transfer coefficient [1/s]
K	The equilibrium constant [dimensionless]
L	The height of pulsed bed column [m]
N	The number of cycles [cycle]
q	The concentration in adsorbent phase [dimensionless]
q_0	The concentration at initial time in adsorbent phase [ICUMSA]
t	Time [s]
v	The interstitial velocity of sugar syrup [m/s]
X_{pulse}	The ratio of adsorbent was removed and replaced per amount of all adsorbent bed
Z	The distance from entrance of column [m]
ε	The bed porosity [dimensionless]
τ	The dimensionless parameter in Klinkenberg's equation
τ_{cycle}	The cycle time of pulsed bed adsorber [min]
$\tau_{cycle,opt}$	The optimum cycle time of pulsed bed adsorber [min]
ξ	The dimensionless parameter in Klinkenberg's equation

1. Introduction

Sugar is the important constituent in the food industries, consumer products and other industries. Its color is one of the important specifications of high value-added refined sugar. In general, the color of the high quality exported super refined sugar must be less than 10 ICUSMA; hence, the colorant in sugar syrup for crystallizing super refined sugar must be less than 100 ICUMSA. The unit operations for removal of colorants in sugar syrup have long been developed and discussed [1-5].

Traditionally, carbonatation and ion exchange resin were used to remove the colorants in syrup. However, the wasted brine used in the regeneration of ion exchange resin was pollutant which need to be treated. Due to the less environmental impact and the lower cost for onsite reactivation, the application of granular activated carbon (GAC) pulsed bed adsorption columns for colorant removal in sugar refining is gradually increasing. In pulsed bed adsorption column, the high color sugar syrup leaving carbonatator (normally 700 – 900 ICUMSA) is fed at the bottom and low color sugar syrup (less than 100 ICUMSA) leaves at the top of the column. Periodically, the exhausted GAC is withdrawn from the bottom and fed to the onsite reactivation in the nearby furnace while the same amount of new GAC is fed at the top of the column [2-4]. Consequently, the pulsed bed adsorption column is the cyclic steady state operation in which the concentration of colorants in treated sugar syrup is a periodical function after certain number of cycles has reached [4]. The concentration gradient between phases in a pulsed bed adsorption column is higher than that in a fixed-bed adsorption column and the mass transfer in pulsed bed operation is much higher and the rate of mass transfer is enhanced. Because of the enhanced mass transfer, the pulsed bed operation generally requires lower capital investment, smaller space requirement, lower energy for regeneration and lower operating cost [4]. The cycle time is referred as the period of this periodical removal of exhausted and addition of new GAC [5, 6]. Because of the periodical simulated countercurrent movement of GAC, the driving force and, hence, mass transfer of colorants is enhanced when comparing with the conventional fixed-bed adsorption column [2, 7]. Although pulsed bed adsorption column has widely applied to the wastewater treatment and sugar refining as well as other industries, only limited number of publications has been available. For example, McKay modelled pulsed bed adsorption columns in wastewater treatment, predicted the wavefront rate and estimated cycle time based on the equations of the fixed bed model [11]. Morley studied the mathematical model of the ion-exchange columns with regeneration operation and performed numerical analysis to determine the optimum condition of color removal when varying the number of cycles, inlet concentration of feed flow rate and other parameters [12].

Based on the literature, no research directly related to the application of GAC pulsed bed adsorption column on removal of colorants in sugar syrup was published. Furthermore, no previous attempt has been made on the process control of the pulsed bed adsorption even though it is widely accepted that disturbances in cyclic steady-state operations; for example, simulated-moving bed chromatography and pressure-swing adsorption do cause the deviation from the desired set point. As a result, the process control of cyclic steady-state operations should be studied and properly tuned [6, 9-11].

As a result, the aim of this research is to develop the mathematical model of pulsed bed adsorption column in sugar refining by using the parameters obtained from the breakthrough curve of fixed-bed column and the modification of the initial conditions at the beginning of each cycle. To investigate the pulsed-bed adsorption column, the numerical analysis is then performed and the optimum cycle time at different colorant concentration in feed sugar syrup is evaluated. Finally, the effectiveness of feedforward control when there are changes of ICUMSA in feed sugar syrup using cycle time as the manipulating variable is investigated for the disturbance of colorant in feed syrup for both step and cosine functions. The finding reported in this article should benefit engineers who work in sugar refining as well as academics who are interested in modelling, optimization and control of cyclic steady-state operation of adsorption processes. It should be pointed out that the linear driving force model is chosen to model the mass transfer of colorant between syrup and adsorbent since it was reported by Cortes [16] that the adsorption of colorants on GAC was linear when the ICUMSA of sugar syrup is less than 2,000. Since Cortes [16] reported that the adsorption equilibrium constant increased with temperature, the chemisorption of colorants on GAC played an important role.

2. Experimental Procedure

2.1. Material

Granular Activated Carbon (GAC) Norit Mag 30I from supplier in average particle size of 1.3 mm. (12x30 mesh size screen) was washed with deionized water and dried in an oven at 105°C for 24 hr. The feed sugar syrup of 700 ICUMSA and 65 Brix was prepared by dissolved refined sugar (Mitr Phol), brown sugar (Aro) in deionized water at temperature 70°C in a water bath.

2.2. Fixed-bed Experiment

The GAC was packed in the column by wet packing method in a column with diameter of 5 cm. and length of 20 cm. After packing, the sugar syrup was fed at bottom of column with the flow rates of 4, 6 and 8 ml/min, respectively. The temperature of 60°C was maintained by heating the feed syrup. The samples were collected at the top of column until the breakthrough curve was obtained. The samples were analysed by measuring the absorbance at 420 nm (with the cell length of 10 mm) according to the ICUMSA method (International Commission for Uniform Methods of Sugar Analysis) and shown in Eq. (1).

$$ICUMSA = \frac{Absorbance(420nm)x10000}{conc.\,of\,total\,solid(g/cm^3)\,x\,cell\,length\,(mm)} \tag{1}$$

2.3. Breakthrough Curve of Fixed-bed Column

Based on the linear driving force model and plug flow assumption, Ruthven [17] show that the concentration profile of eluent for linear adsorption isotherm can be explained by Klinkenberg's equation, Eq. (2) to (4).

$$\frac{C}{C_0} = \frac{1}{2} erfc\left(\sqrt{\xi} - \sqrt{\tau} - \frac{1}{8}\sqrt{\xi} - \frac{1}{8}\sqrt{\tau}\right) \tag{2}$$

where

$$\tau = k\left(t - \frac{Z}{v}\right) \tag{3}$$

and

$$\xi = \frac{kKZ}{v}\left(\frac{1-\varepsilon}{\varepsilon}\right) \tag{4}$$

The breakthrough curve from fixed-bed experimental was thus used to determine mass transfer coefficient and equilibrium constant from nonlinear regression by minimize the summation square of error of the experimental results and the values obtained from Klinkenberg's equation at the exit of fixed bed column (Z = L). The bed porosity (ε) for GAC bed was set at 0.4 as suggested by Ruthven [17] and Tangsathitkulchai [18].

2.4. Modelling and Numerical Analysis of Fixed-bed and Pulsed Bed Adsorption Column

With the assumptions of linear adsorption isotherm, linear driving force model, plug flow assumption, the mathematical model of fixed bed column was shown in Eq. (5) and (6)

$$\frac{\partial C}{\partial t} = -v\frac{\partial C}{\partial Z} - \frac{q_0}{C_0}\left(\frac{1-\varepsilon}{\varepsilon}\right)\frac{\partial q}{\partial t} \tag{5}$$

$$\frac{\partial q}{\partial t} = k(C - C_{eq}) \tag{6}$$

The initial conditions of fixed-bed column are

$$C(Z, t = 0) = 0 \text{ and } q(Z, t = 0) = 0 \tag{7}$$

The boundary conditions of fixed-bed column is

$$C(Z = 0, t) = C_0 \tag{8}$$

Equations (5) and (6) were solved by numerical method of lines (NMOL) with spatial derivative terms approximated by 5 point biased up-winding approximation with the step sizes of 0.01 m [19-20]. The 4th-order Runge Kutta method was then used to solved the resulted ordinary differential equation, using SCILAB [21]. The accuracy of the mathematical model is validated by comparing with the breakthrough curve obtained from experiment and numerical analysis using the mean absolute error (MAE) which was defined in Eq. (9).

$$MAE = \frac{\sum_{i=1}^{n}\left|C_{model,i} - C_{exp,i}\right|}{n} \tag{9}$$

Because the mass transfer in the fixed bed and in the pulsed bed are exactly the same, the mathematical model of pulsed bed adsorption column was adapted from that of fixed-bed column with the modification of initial conditions at the beginning of each cycle so that the pulse of exhausted GAC and the addition of new GAC was taken into account.

The initial conditions at the first cycle of pulsed bed are

$$C(Z, t = 0) = 0 \text{ and } q(Z, t = 0) = 0 \tag{10}$$

The initial conditions at the beginning of N+1th cycle which represent the 10% removal of GAC at the bottom

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of the column and 10% addition of novel GAC at the top of the column are

$$C = \begin{cases} 0 \; when \; Z \geq (1-0.1)L \\ C(t = Nt\tau_{cycle}, Z - 0.1L) \; if \; Z \leq (1-0.1)L \end{cases} \quad (11)$$

$$q = \begin{cases} 0 \; when \; Z \geq (1-0.1)L \\ q(t = N\tau_{cycle}, Z - 0.1L) \; if \; Z \leq (1-0.1)L \end{cases} \quad (12)$$

The boundary condition after the first interval is

$$C(Z = 0, t) = C_0 \quad (13)$$

Equations (5) and (6) along with the initial and boundary conditions of pulsed bed adsorption column were solved by numerical method of lines using the value of K and k obtained from fitting with the breakthrough curve.

2.5. Estimating the Optimum Cycle Time of Pulsed Bed Adsorber Column

The optimum cycle time was first estimated from the rate of mass transfer zone expansion at the breakthrough. By using the pulse of 2 cm (corresponding to the 10% pulse - the 10% pulsing is within the normal operating range of 5 – 15% as reported by Liang [3]), the initial guess of pulse time is the ratio of 0.02 and rate of mass transfer zone expansion. The iteration of optimum cycle time was then performed by small increases of the first estimate until the concentration of colorants in product syrup reached its specification of 100 ICUMSA. In order to study the effect of colorant concentrations in feed syrup on optimum cycle time, the concentration (C_{inlet}) of colorants in feed syrup was varied from 500 ICUMSA to 1200 ICUMSA and the optimum cycle time ($\tau_{\text{cycle,opt}}$) of pulsed bed column at each flow rate was determined by using the same approach. The relationship of the optimum cycle time and colorant concentration in feed syrup was correlated by linear regression.

2.6. Feedforward Control of Pulsed Bed Adsorption Column

The feedforward control of pulsed bed adsorption column when there is the disturbance of colorant in feed sugar syrup using the cycle time (τ_{cycle}) as the manipulated variable and the outlet concentration of colorants (C) as the controlled variable as shown in Fig. 1 was investigated. When there is the change in C_{inlet}, the cycle time was adjusted based on the relationship of $\tau_{cycle,opt}$ and C_{inlet} obtained in Section 2.5.

The feedforward control performance when there were step disturbance and cosine disturbance were then investigated. The step disturbance, when C_{inlet} changed 700 to 500, and 700 to 900 ICUMSA represented the scenario when the colorant concentration in feed sugar syrup in the actual raw sugar processes decreased and increased respectively. While the cosine function with the average of 800 ICUMSA, the amplitude of 100 ICUMSA and the period of 4 hours represented the scenario in the real sugar refining plants that the colorant concentrations in feed syrup fluctuated.

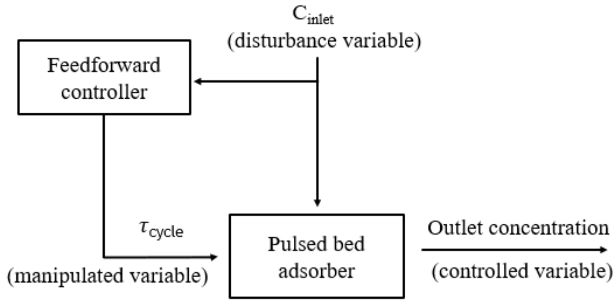

Fig. 1. Block diagram of feedforward control of pulsed bed adsorption column.

3. Result and Discussion

The equilibrium constant (K) and mass transfer coefficient (k) from nonlinear regression with Klinkenberg's equation were shown in Table 1. Because the mass transfer coefficient increased when feed flow rate increased, the mass transfer coefficient at 4 ml/min was the lowest due to the lowest value of Reynold number.

Table 1. Parameter estimated from breakthrough curve of fixed-bed column.

Flowrate [ml/min]	K [-]	k [1/s]	Mean Absolute Error (MAE)
4		$2.35\text{x}10^{-4}$	$6.66\text{x}10^{-2}$
6	1.58	$8.08\text{x}10^{-4}$	$1.08\text{x}10^{-1}$
8		$8.09\text{x}10^{-4}$	$6.86\text{x}10^{-2}$

By using the parameters listed in Table 1, the breakthrough curve at 4, 6 and 8 ml/min were able to be correlated as shown in Fig. 2. The small MAE may probably cause by the axial dispersion that was neglected in this model. Further communication on the effect of axial dispersion will be published subsequently. It should be noted that the breakthrough time generally decreased when the flow rate increased.

Since the linear adsorption isotherm of colorants on GAC was reported [16], it was expected that the mass transfer zone was not constant [17]. Consequently, the concentration profile inside the column at different flow rates was shown in Fig. 3. Based on Fig. 3, it was obvious that the width of mass transfer zone was not constant. By using the value of C/C_0 at breakthrough point of 0.143, which was the specification, the mass transfer zone width and the rate of mass transfer zone expansion were shown in Fig. 4 and Fig. 5.

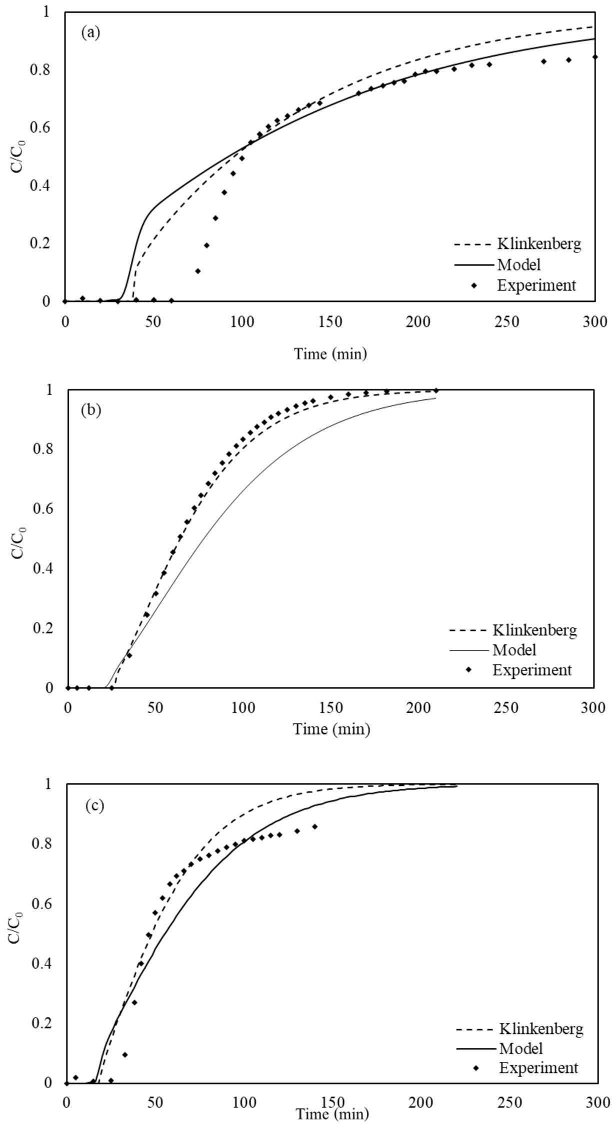

Fig. 2. Comparison between breakthrough curve from experiment, Klinkenberg and the model of fixed-bed column at different flow rates (a) 4 ml/min (b) 6 ml/min and (c) 8 ml/min.

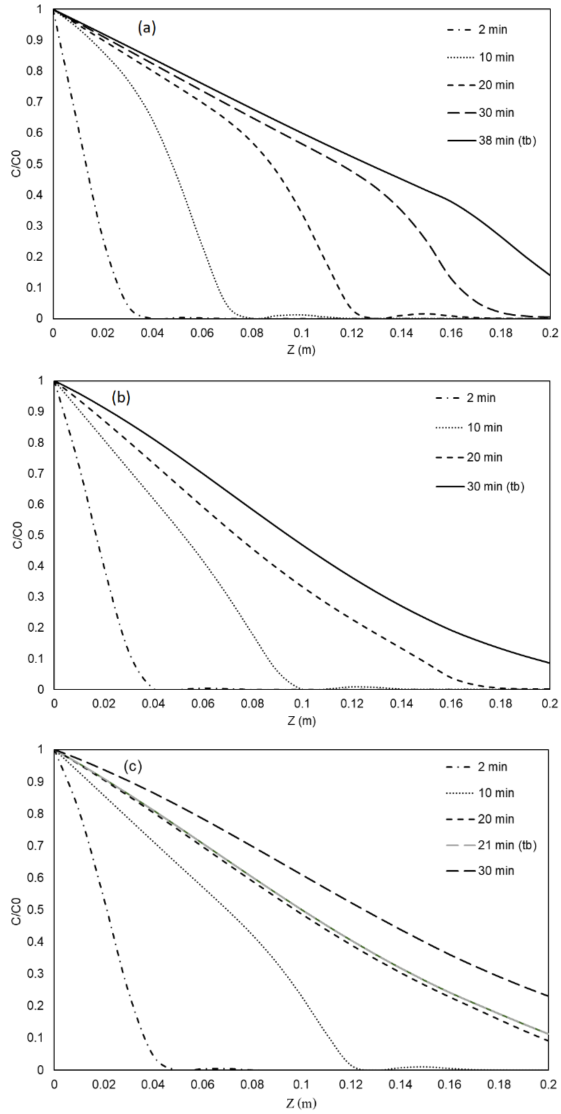

Fig. 3. Concentration profiles inside a fixed bed column obtained from a model at different flow rates (a) 4 ml/min (b) 6 ml/min and (c) 8 ml/min.

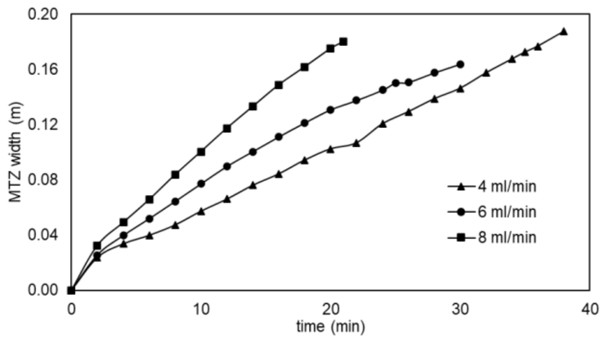

Fig. 4. Mass transfer zone width in a fixed bed column at flow rate of 4, 6, and 8 ml/min.

From Fig. 5, it was obvious that the rate of mass transfer zone expansion depended on the flow rate. At the flow rate of 8 ml/min, the mass transfer zone expanded at the highest speed than the other flow rates and would result in the lowest breakthrough time. Since the mass transfer zone must not move beyond the pulse bed adsorption column for the successful run of pulsed bed adsorption, the breakthrough time and the rate of mass transfer zone expansion usually affected the optimum cycle times in the pulsed bed adsorption column which would be discussed next.

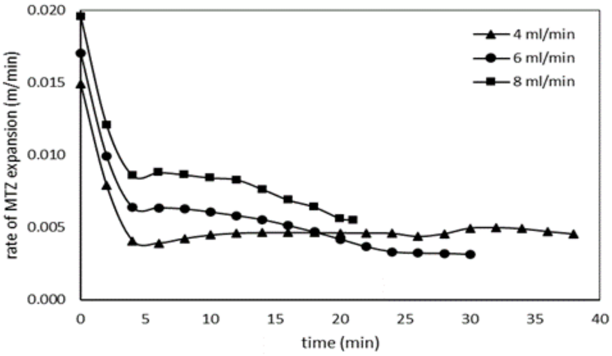

Fig. 5. Rate of mass transfer zone (MTZ) expansion in fixed bed at flow rate of 4, 6 and 8 ml/min.

It should be further noted that the rate of mass transfer zone expansion shown in Fig. 5 was from the beginning of fixed bed operation to the breakthrough point. The rate of mass transfer zone expansion probably decreased further after the breakthrough. Nevertheless, these rates of mass transfer zone expansion at the breakthrough were used as the initial guesses of optimum cycle time in the pulsed bed operation.

The numerical analysis of pulsed bed adsorption column was performed based on the parameters listed in Table 1 and shown in Fig. 6.

From Fig. 6, the cyclic steady state behavior of pulsed bed adsorption column was clearly established after 23 to 30 cycles. It should be noted that the cyclic steady states were also reached after 23 to 30 cycles at the flow rate of 4 and 8 ml/min. By using the initial guess of cycle time from the rate of mass transfer zone expansion obtained in fixed bed, it was observed that there was the optimum cycle time at the corresponding flow rate and the colorant concentration in feed sugar syrup. For the feed sugar syrup of 700 ICUMSA and at the flow rate of 6 ml/min, the optimum cycle time is 7.68 min.

When the cycle time was higher than the optimum cycle time, the exit concentration of colorants at the cyclic steady state will be higher than its specification of 100 ICUMSA. On the other hand, if the cycle time is less than the optimum one, the GAC will not be used efficiently – the removal of GAC which could still adsorb colorants occurred. Therefore, it is particularly of engineers' interests to investigate the optimum cycle time at different flow rates and different colorants concentration.

It should be further pointed out that this optimum cycle times were higher than the initial guesses due to the fact that the rate of mass transfer zone expansion decreased after the breakthrough time. Using the approach similar to the one described above, the relationship of the optimum cycle time and the colorant concentrations in feed sugar syrup at different flow rate were shown in Fig. 7.

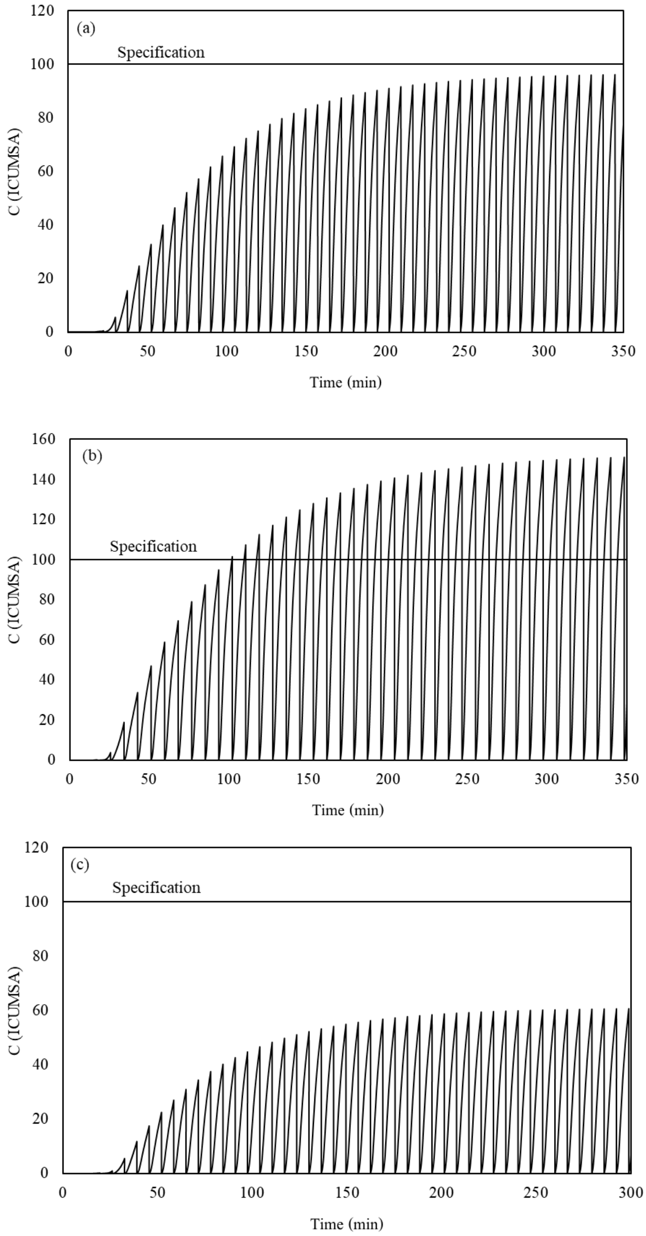

Fig. 6. The predicted performance of pulsed bed column at inlet concentration 700 ICUMSA of feed flow rate 6 ml/min. (a) optimum cycle time of 7.68 min (b) cycle time < optimum cycle time and (c) cycle time > optimum cycle time.

Based on Fig. 7, it was obvious that the optimum cycle time increased when the inlet concentration decreased since the mass transfer zone would move at the lower speed and the frequency of pulsing would definitely be less. Furthermore, when the inlet concentration was 100 ICUMSA which was the specification of the process, there was no need to run the pulsed bed adsorption columns and the correlation yielded the optimum cycle time of infinity which reflected this fact.

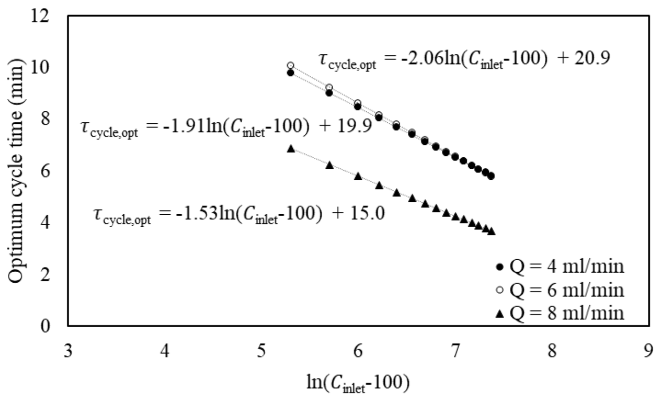

Fig. 7. Relationship between inlet concentration and optimum cycle time of pulsed bed adsorber column at feed flow rates of 4, 6 and 8 ml/min.

In addition, the optimum cycle time at 4 ml/min and 6 ml/min were very close to each other, especially at higher colorant concentration in feed sugar syrup while the optimum cycle time at 8 ml/min was significantly less than at 4 or 6 ml/min. It should be pointed out that the longer breakthrough time in fixed bed column would result in the shorter optimum cycle time. Furthermore, the mass transfer zone at 8 ml/min moved at the higher speed and the lower optimum cycle time was thus required. Consequently, the control of feed flow rate of syrup must be applied in order to operate the pulsed bed adsorption column efficiently. Alternatively, the more advance control of industrial pulsed bed adsorption column where the feed flow rate was the disturbance must be investigated in the future.

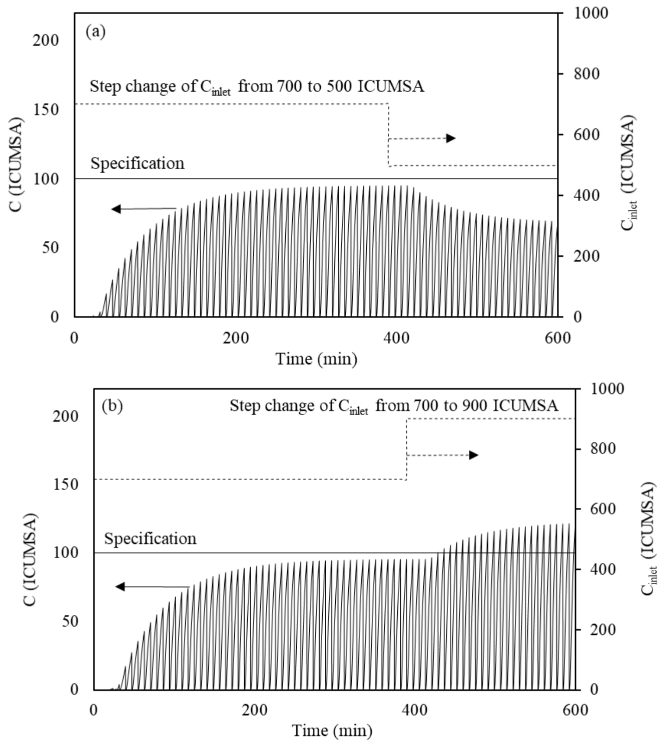

Fig. 8. The predicted performance of pulsed bed column at constant cycle time of flow rate 6 ml/min (a) inlet concentration changed 700 to 500 ICUMSA and (b) inlet concentration changed 700 to 900 ICUMSA.

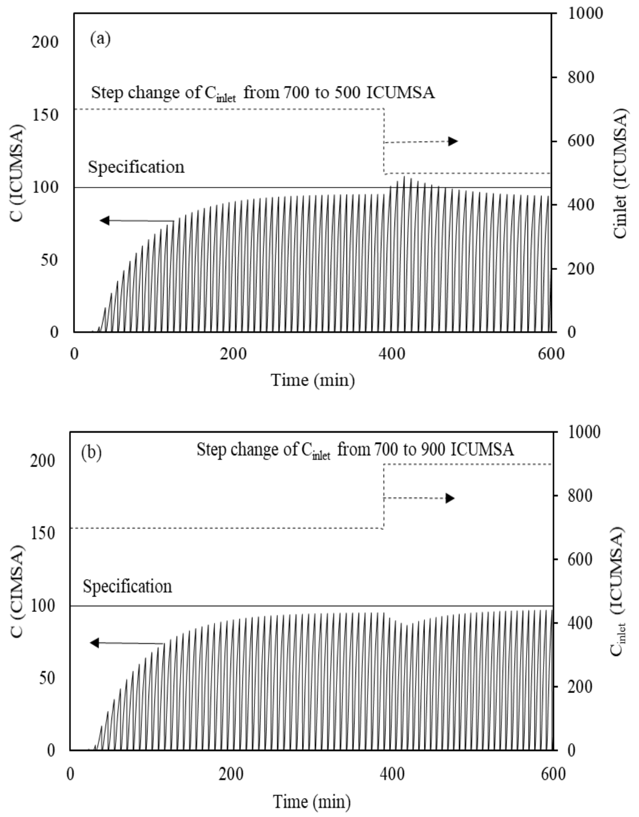

Fig. 9. The predicted performance of pulsed bed model for step change at flow rate 6 ml/min (a) cycle time adjusted immediately for step change of inlet concentration from 700 to 500 ICUMSA and (b) cycle time adjusted immediately for step change of inlet concentration from 700 to 900 ICUMSA.

In order to investigate the advantage of using feedforward control on pulsed bed adsorption column, the numerical investigation of pulsed bed adsorption column and pulsed bed adsorption column without and with feedforward control were shown in Fig. 8 and Fig. 9, respectively. In Fig. 8, the performance of pulsed bed adsorption column with the step change of colorant concentration in feed sugar syrup without feedforward control was shown and it was obvious that the product did not meet its specification since the product would have the color of 120 ICUMSA. On the other hand, when the colorant concentration in feed syrup decreased from 700 to 500 ICUMSA, the GAC was not fully utilized. Hence, the application of feedforward control of pulsed bed adsorption column should be beneficial.

Therefore, the concept of feedforward control by using the cycle time as the manipulated variable was investigated and their performances were illustrated in Fig. 9. From Fig. 9a and 9b, the performance of feedforward control with the immediate adjustment of the cycle time when there was a change in colorant concentration in feed sugar syrup was shown. It was found that there was the undesirable effect between the first and the eighth cycle after the disturbance since the

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product had the color exceeded 100 ICUMSA for approximately 7 cycles before it decreased to 100 ICUMSA after reaching the new cyclic steady state. This temporally negative effect probably caused by the large time constant of the pulsed bed adsorption column – the effect of colorant concentration in feed sugar syrup was normally observed only after 4 to 8 cycles (depending on flow rates). The same phenomenon was observed in the fixed bed adsorption column.

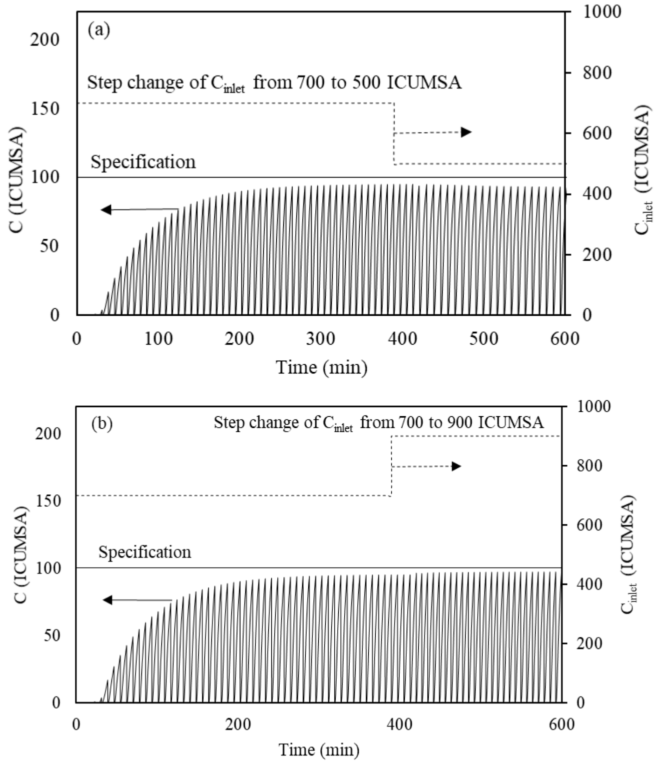

Fig. 10. The predicted performance of pulsed bed model for step change at flow rate 6 ml/min (a) cycle time adjusted with the delay of 5 cycles for a step change of inlet concentration from 700 to 500 ICUMSA and (b) cycle time adjusted with the delay of 5 cycles for a step change of inlet concentration from 700 to 900 ICUMSA.

In order to remedy this negative effect, the delay of 5 cycles was added to the manipulated variable. When this delay was added, the performance of pulsed bed adsorber were much better as presented in Fig. 10a and 10b. There was no temporally undesirable effect and the color of sugar syrup leaving the pulsed bed adsorption column was within its specification of 100 ICUMSA for all cycles.

The performance of feedforward control of pulsed bed column with the delay of manipulated variable was further investigated with the periodical disturbance of colorant concentration in feed syrup using cosine function which should represented the fluctuation of colorant concentrations in feed syrup observed in real sugar refining processes and the results were shown in Fig. 11.

From Fig. 11, it was obvious that the feedforward control of pulsed bed column was able to produce the product sugar syrup than met its specification with the efficient utilization of GAC. On the other hand, the pulsed bed adsorption column without feedforward control did not produce the sugar syrup that met its specification.

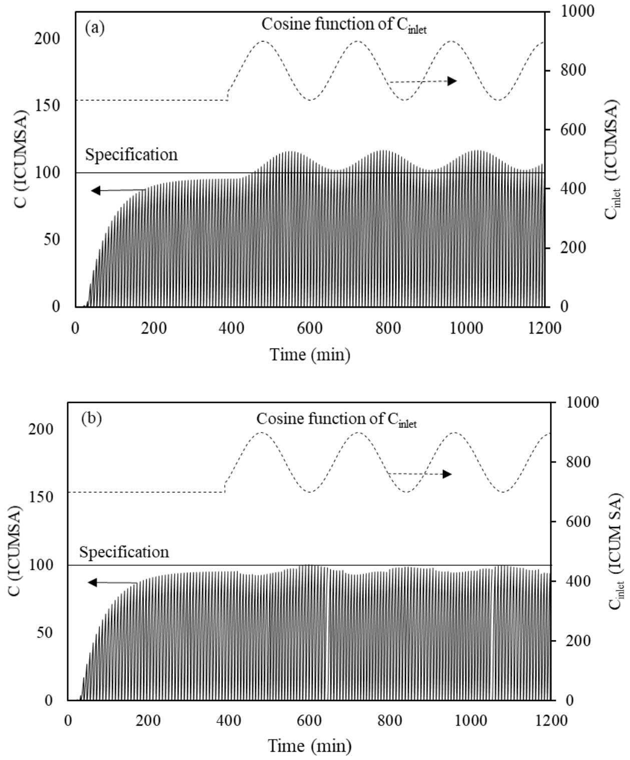

Fig. 11. The predicted performance of pulsed bed column of inlet concentration changes of 700 ICUMSA to cosine function (a) without feedforward control and (b) cycle time adjusted with the delay of 5 cycles.

As a result, the concept of feedforward control when applying to pulsed bed adsorption columns in sugar refining process would enhance the operation of pulsed bed adsorption column. However, in order to efficiently control pulsed bed adsorption column, further studies on feedforward are required, especially on the effect of other disturbances as well as the online parameter estimation on equilibrium constant of adsorption – the disturbance that comes from the reactivation of spent activated carbon. Furthermore, the more advanced process control which could yield the acceptable syrup when there are several disturbances; for example, feed flow rates and colorant concentrations in sugar syrup at the same time, should be investigated.

4. Conclusion

In conclusion, the model of pulsed bed adsorption column in sugar refining process was developed and solved by using the parameters obtained from the breakthrough experiment. The feedforward control using the cycle time as the manipulated variable, was able to compensate the disturbance of colorants concentration in feed sugar syrup. Further investigation on the effect of axial dispersion is currently undergoing and is going to be published subsequently.

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