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Transient Response of a Distillation Column Plate. Part II. Model Evaluation from Flow and Mixing Data on a Plate without Two-Phase Mass Transfer

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Abstract

The operation of a distillation column plate was simulated by using air for the vapor phase and water for the liquid phase; the turbulent mixing effect in the liquid on the tray and in the downcomers was analyzed by following composition changes of salt (sodium chloride). Composition changes entering the plate and in the downcomer leaving the plate were monitored. The composition–time data was numerically Laplace transformed, and the frequency response form was fit to each of five models: (1) perfect mixing; (2) plug flow; (3) perfect mixing with time delay in the downcomer; (4) dispersion; and (5) dispersion with dead zones on the tray. The composition–time data was reconstructed from each of the best fitting parameters of each of the models, and this data is given for comparison with the experimental data.

The model utilizing dispersion was the best statistical model. The model of perfect mixing on the tray and delay in the downcomer gives a satisfactory fit and is much simpler.

INTRODUCTION

In order to check the five models of a distillation column plate previously presented for the simpler dynamics of liquid flow without liquid vapor mass transfer (distillation) and without frothing, a simulator was

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built and operated with the system air–water–salt, with salt solutions being used for pulse injections. A 4-plate simulator was used, with the salt solution being injected into the downcomer from the plate above the test plate and the concentration changes were measured in the downcomer from the test plate.

In the subsequent part of the paper the five models are compared for adequacy of fit along with the data on the models for distillation.

EXPERIMENTAL EQUIPMENT

The apparatus used to simulate liquid dynamics without mass transfer is shown schematically in Fig. 1. Tap water was fed through a calibrated rotameter onto the top plate. Compressed air entered through the bottom.

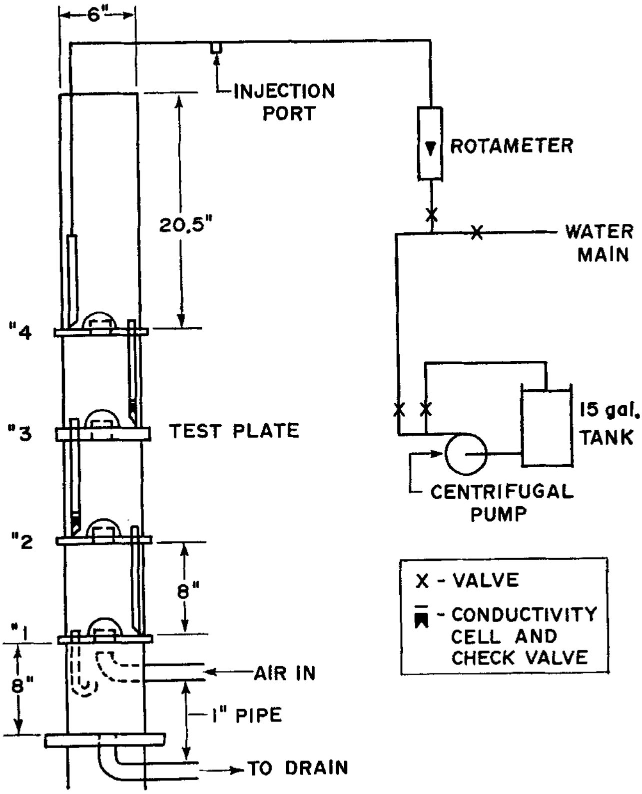

FIG. 1. Schematic of distillation simulator apparatus.

Air velocity was determined using a 0.402-in. orifice meter. The column was constructed of 8-in. long, 6-in. diameter Pyrex pipe. A 20½-in. glass section at the top was used to reduce liquid entrainment. The vapor distributor was a 3/4-in. pipe nipple brazed to an 8-in. long sheet steel can. Liquid drained from the simulator through a 1-in. diameter nipple brazed to the can bottom. A 36-in. section of Tygon tubing was attached to the drain nipple and suspended a height of 31 in. above the can. This leg pressurized the column and forced the air to flow through the column and not out the drain. The tubing diameter was sufficient to prevent siphoning.

The plates were 8¼ in. apart, 6 in. in diameter with one 3½-in. diameter bubble cap in the center. Four plates were used in the apparatus, two below and one above the test plate. These plates were constructed of ½-in. Lucite sheet and were used to stabilize the flow. The test plate was a Corning Glass Vicor plate. The downcomers were ¼-in. PVC pipe. The pipe was machined to a 5/8-in. o.d. and a ½-in. i.d. Check valves were located at the base of the test downcomers to prevent tracer backflow. The check valves were 1-in. long, ½-in. o.d. machined Teflon. The center of the rod was reamed to a 3/8-in. taper, and a ¼-in. polyethylene ball was placed into this space and secured by a pin. Conductivity cells were placed 1½ in. from the downcomer bases. Cells were placed in the downcomers of plate #4 and the test plate, #3, to measure the input and output pulses.

A 15-gal tank, connected to the water system, held tracer solutions used in cell calibrations. The tracer was injected with a hypodermic needle through a septum in the inlet water line. The amount of tracer injected was between 0.1 and 0.3 cc. A salt solution was the tracer because of its linear relationship between conductivity and concentration. The conductivity cells were platinum black-coated platinum wires inserted in the downcomer bases.

EXPERIMENTAL TECHNIQUES

The electronic system for the mixing apparatus was "warmed up" for ½ hr and water was run through the apparatus to remove impurities from the lines. This was done so that the base conductivity would remain constant during a set of runs. The air lines were purged at an orifice pressure drop of 0.2 in. of water.

After the "warm up" period the recorder system was tested by injecting a 0.1-cc. saturated salt solution pulse. The system sensitivity was adjusted until a 0.1-cc. pulse gave approximately 80% full-scale deflection. The conductivity cells were calibrated by feeding water through the centrifugal pump system. Saturated salt was added to the 15-gal bucket in 3 to 4 cc increments, and steady state conductivity traces were recorded from zero to full scale. An additional calibration was performed by substituting a decade resistance box for each cell while the other cell remained in place. Resistance versus scale deflection readings were recorded over the full scale range.

Water flow rates were examined from 0.10 to 0.22 gal/min in increments of 0.02 gal/min. Air line orifice pressure drops investigated were 0, $\frac{1}{2}$, 1, 2, 3, 4, 5, $7\frac{1}{2}$, 10, and 15 in. of water. Duplicate runs were made at pressure drops of 0, 3, and $7\frac{1}{2}$ in. of water. Due to column flooding, not all air flow rate combinations were possible. For each flow rate the system was pulsed, and the conductivity traces were recorded. The air flow rate was changed and the water flow rate was checked and adjusted. The system was allowed to stabilize at least 5 min between each run. The water temperature was recorded during the middle of the set of runs. The resistance-scale deflection calibration was rechecked at the conclusion of a set of runs. The system was then shut down.

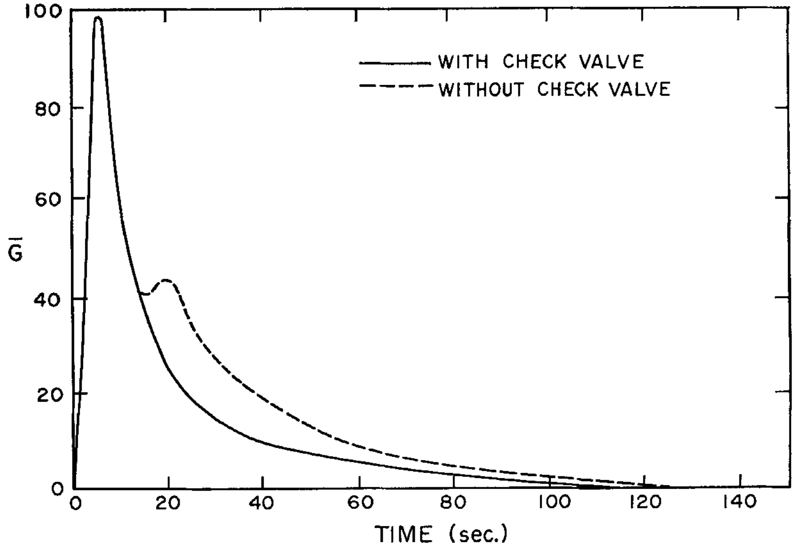

FIG. 2. Effect of back flow on recorded input pulse.

RESULTS AND DISCUSSION OF RESULTS

System Checks

The dynamics of the recording apparatus and column modifications were checked for their contributions to the system response.

Tracer was observed flowing from the plate back into the downcomer feeding the plate. This was unacceptable since the measured plate input function for the models was the concentration leaving the preceding plate, and this backflow interfered with the concentration measurements in the downcomer. Check valves installed between the conductivity cell and the downcomer base eliminated this problem. Figure 2 shows the

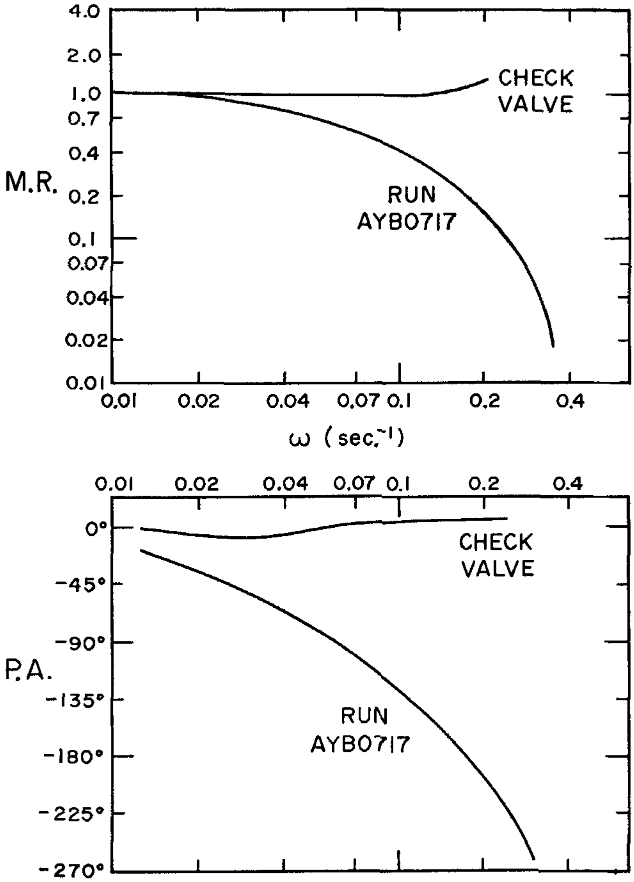

FIG. 3. Check valve response.

effect of backflow on the pulses. The effect of the check valve on the system frequency response was investigated for any contribution to the column dynamics. Figure 3 illustrates that the check valve had a negligible effect on the measured column dynamics.

When the apparatus used in the simulator flooded, bubbles would clog the downcomer. The bubbles surrounded the conductivity cells and the recorded base line would go off scale and could not be returned. The cells would clog before flooding was observed on the manometer across the apparatus and long before flooding was visually observed. This phenomena appears to be a criterion for sensing incipient flooding.

The Moseley 680 recorder dynamics were investigated. The response was observed to be flat to about 12 rad/sec. The maximum system response was less than 0.4 rad/sec, so the recorders contributed no significant dynamics to the total system response.

Simulator Operation

Figures 4–8 demonstrate the results of fitting the data from run AYB 0717 for the simulator with the perfectly mixed, plug flow, perfectly mixed plate with time lag, dispersion, and dead zone models (*1*). Run AYB

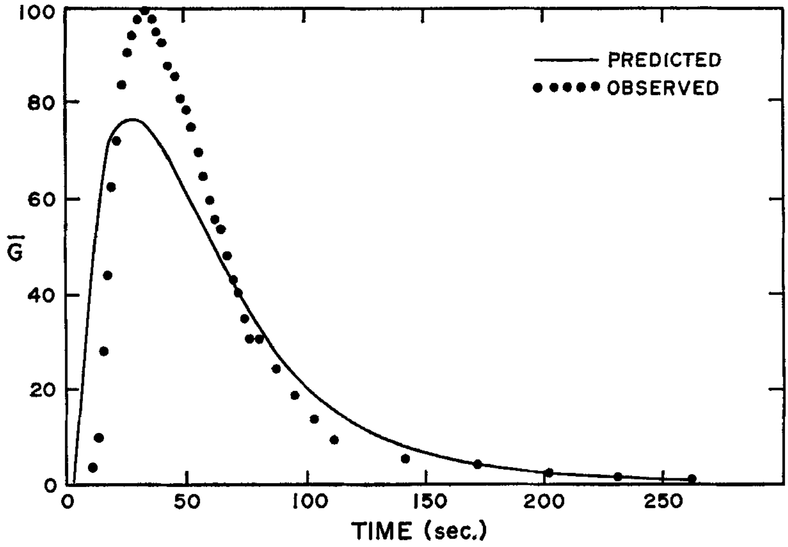

Fig. 4. Fitting of the data from run AYB 0717 for the simulator with the perfectly mixed model.

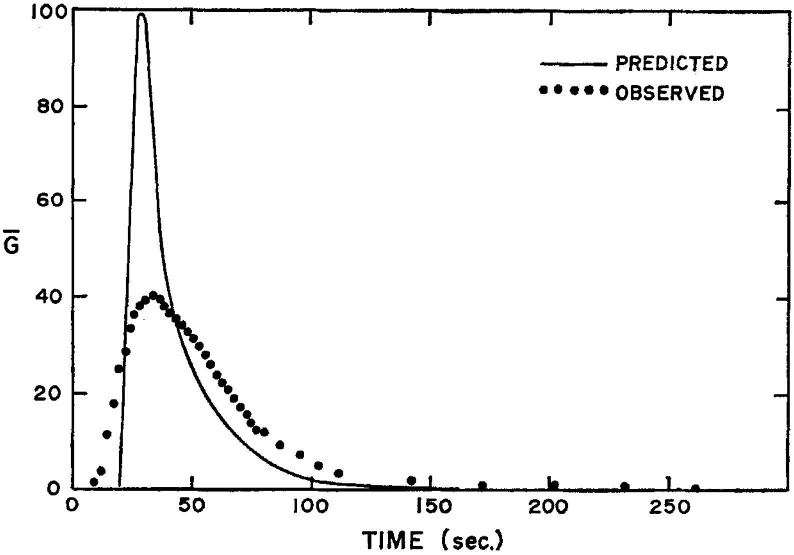

FIG. 5. Fitting of the data from run AYB 0717 for the simulator with the plug flow model.

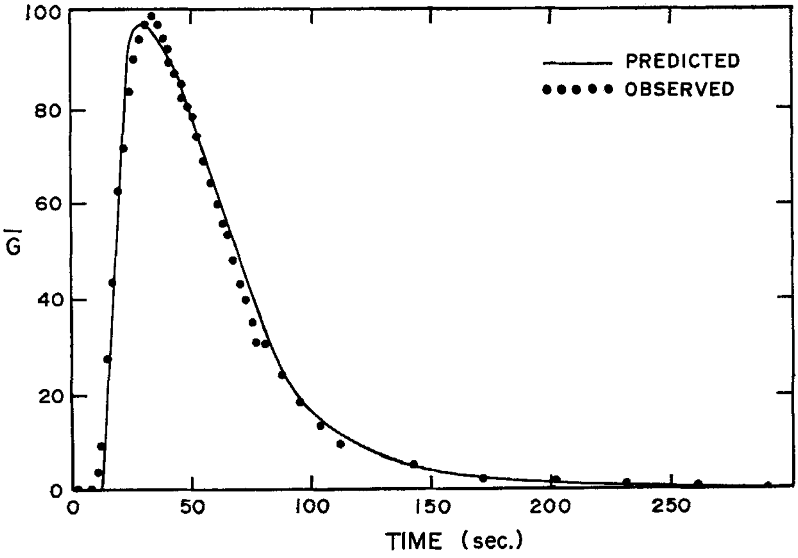

FIG. 6. Fitting of the data from run AYB 0717 for the simulator with the perfectly mixed plate with time lag model.

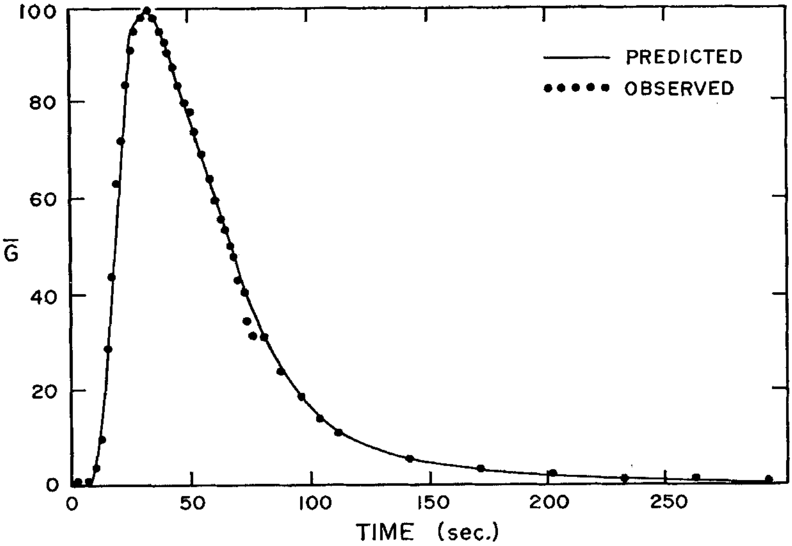

FIG. 7. Fitting of the data from run AYB 0717 for the simulator with the dispersion model.

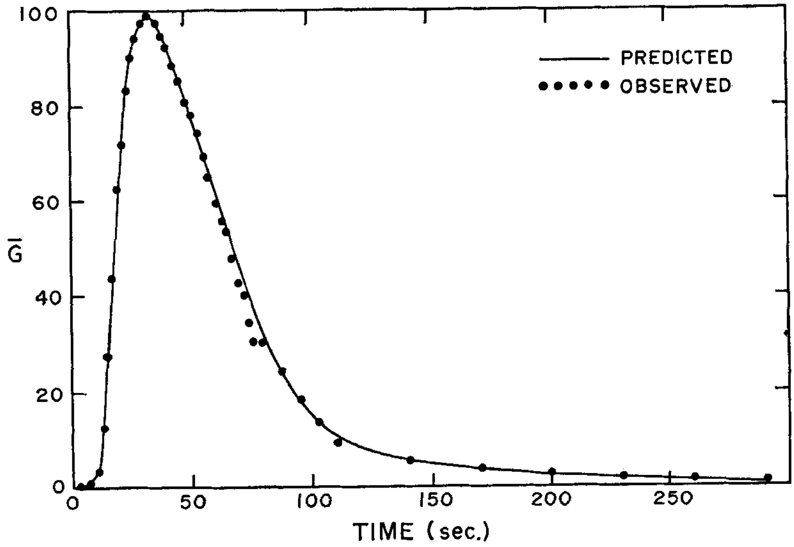

FIG. 8. Fitting of the data from run AYB 0717 for the simulator with the dead zone model.

0717 was made in the mixing apparatus. The ordinate, $\bar{G}$, is the reduced normalized concentration

$$\bar{G} = \frac{G_e}{G_{\max}} * 100 \tag{1}$$

where $G_{\max}$ is the maximum value of either the predicted or observed curve.

Table 1 gives the parameter values in fitting run AYB 0717 along with the standard error of estimate, S.E.:

$$\text{S.E.} = (y_o - y_p)/\text{D.F.} \tag{2}$$

where D.F. is the degrees of freedom.

Examination of Figures 4–8 and Table 1 show that, in order of increasing ability to fit the data, the models were the plug flow model, perfectly mixed model, perfectly mixed plate with time lag, dispersion model, and the dead zone model. However, it was observed that there was not a noticeable difference in the ability of the dispersion and dead zone models in fitting the data.

The dead zone model parameters θ and τ were superfluous in fitting since they effectively canceled each other. To illustrate this, 27 data points were generated from the equation

$$y = x + 1 \qquad x = 1, 2, \ldots, 27 \tag{3}$$

and were fit to the model

$$y = a_1 + a_2 x\left(1.0 + \frac{a_3}{a_4 x + 1}\right) \tag{4}$$

where a_1, a_2, a_3, and a_4 were the unknown parameters.

TABLE 1

Typical Parameters for the Simulator

	α (sec)	η (sec)	τ_d (sec)	θ	τ	$\sqrt{\pi}$S.E.
Perfect mixed	34.2					0.08423
Plug flow	20.5					0.2278
Perfectly mixed plate with time lag	23.0		9.02			0.01555
Dispersion	31.4	118.5				0.00939
Dead zone	30.5	127.6		0.233	158.1	0.00734

The parameters were fit with the nonlinear least squares program of Marquardt (*2*) and gave the expression

$$y = 1.0000000 + 1.0000000x\left(1.0 + \frac{780.54126}{490{,}168{,}830x + 1}\right) \tag{5}$$

It is obvious that the parameters a_3 and a_4 have no real value, but they are finite valued and the sum of the squared error is not different from Eq. (4) due to round off errors and the number of significant digits to which a number may be expressed on a digital computer. Equation (5) is similar to the radical in the transform of the dead zone model (*1*). It is apparent that if the dispersion model accurately represents the data, then the dead zone model would also fit the data but the parameters θ and τ would have no significance (*1*). From Table 1 the ratio of θ to τ is 1470, which is similar to the result of Eq. (5) (i.e., the dead zone model has essentially converged to the dispersion model). The convergence was found in the majority of cases for the mixing apparatus and also for the distillation column.

The concentration-time profile as a function of plate distance was examined. Four conductivity cells were placed on a plate at the base of the inlet downcomer, half way between the bubble cap and the inlet downcomer, half way between the bubble cap and the outlet downcomer, and at the base of the outlet downcomer. Pulse tests were made at high and low liquid and air flow rates. A time lag was observed between the recorded pulses, implying that the perfectly mixed plate concept was physically invalid. The tracer pulse spread with position was also observed, causing the plug flow model to be physically inaccurate. These results aid in understanding why the dispersion model fit the data better. The plate unit in this study consisted of the bubble cap plate and its associated downcomer, since these column internals must be considered in any attempt at modeling distillation columns.

The mixing apparatus mass balance error was 7.1% and the parameters from duplicate runs differed by an average of 2.9%. For the mixing apparatus the residence time could be estimated to ±25% and for the column and the mixing apparatus the estimation was within ±30%. This error is not excessive since the clear liquid holdup had to be visually estimated just as the vapor flow ceased. The best estimate of the clear holdup height was $\frac{1}{4}$-in., which was an error of ±30%.

The dispersion function $(D/L)^{\frac{1}{2}} = (D_L/X^2)^{\frac{1}{2}}$ from the simulator (mixing only apparatus) runs was fit to an equation form suggested by Gerster et al. (*3*) to give

$$(D_L/X^2)^{\frac{1}{2}} = 0.1783 - 0.01517u + 0.001016L - 0.1390W \qquad (6)$$

for a range of variables; u from 0.0 to 1.05 ft^3/(sec) (ft^2 tray bubbling area); L from 0.355 to 0.755 gal/(min) (average tray width, ft), and W from 7/8 to 1 in.

SYMBOLS

D_L	longitudinal dispersion coefficient
G_e	effective deviation in measured quantity
$G_{\max}$	the maximum value of G_e occurring in any one run
$\bar{G}$	$G_e/G_{\max}$, a normalized variable
L	L factor, liquid flow rate [gal/min (average feet tray width)]
u	u factor, linear gas velocity [ft^3/sec (square feet of tray bubbling area)]
W	outlet weir height (in.)
x	liquid mole fraction
y	vapor mole fraction
θ	ratio of dead zone cross sectional area to active zone and for plate n
τ	inverse of dead zone mass transfer coefficient times the ratio of dead zone cross sectional area to volume on plate n (sec)

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