맥동 추출탑내에서의 축방향 분산

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AXIAL DISPERSION IN A PULSED EXTRACTION COLUMN

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요 약

맥동 추출탑 내경 10.8cm 내에서의 축 방향 분산계수에 관하여 고찰하였다. 계수의 측정은 산ー 알칼리 중화 반응을 이용하여 연속상의 지시약이 변화하는 과정을 관찰하는 방법을 사용하였다.

탑판으로서는 별모양의 판과 다공판 판의 두 종류를 사용 하였으며, 공기 맥동은 솔레노이드 밸브와 시간 조절 장치를 이용 하였다.

맥동의 진폭과 진동수의 변화 범위는 각각 0.5~8cm, 0.5~2.0. Hz 이다. 축 방향의 분산계수 는 진폭의 제곱과 진동수의 곱에 비례하였고, 판의 자유 행로 면적과 판간의 거리에 따라 감소 하였다.

판의 두께와 액상의 유속은 축 분산에 별로 영향을 미치지 않았다. 측정된 계수는 진폭, 진동수, 자유행로 면적과 판간의 거리의 변수들의 상관식을 갖는다.

Abstract

Axial dispersion coefficients have been measured in the continuous phase of a 10.8 cm diameter pulsed plate extraction column. The measurements were made by observing the course of an instantaneous chemical reaction between acid and base in the continuous phase, using phenolphthalein as an indicator.

Two different plates were employed, namely perforated and star-shaped plates. The air

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pulsing was generated by means of solenoid valves and a timing controller. The pulsation amplitude and freguency were varied from 0.5 to 8.0 cm and from 0.5 to 2.0 Hz, respectively.

Axial dispersion increased proportionally with the product of the square of amplitude and frequency, however, it decreased with free opening area of the plate and plate spacing. The flow rates of each phase and the plate thickness have insignificant effects on the axial dispersion.

The data have been correlated in terms of amplitude, frequency, plate spacing and free opening area of the plates.

1. INTRODUCTION

In recent years the usage of liquid-liquid extraction columns has been on up swing in the industry, especially in the area of nuclear fuel processing.

In the designing of continuous countercurrent interfacial mass transfer equipments, the main objectives are to achieve a high mass transfer effectiveness and a high throughput in relation to column capacity while maintaining minimum axial mixing.

The variation of the extraction efficiency as a function of the pulse velocity(amplitude x frequency) has been investigated in most of the previous studies. [1~3] As agitation is increased, the beneficial effect of increased interfacial area tends to be offset and eventually reversed by axial dispersion which has the effects of reducing the axial concentration gradients in each phase and the driving force in the column. Therefore, the axial distribution of flows in the column must be determined in order to improve the performance of extraction column.

The problem of axial dispersion has been extensively studied by Sleicher[4] and Vermeulen et. al.[5] Axial mixing in the continuous phase of an extraction column is the result of twe effects. The first it true turbulence

and molecular diffusion in the axial direction. Socond, axial mixing is caused by non-uniform velocity and subsequent radial mixing, which is sometimes called Tayler diffusion. Non-uniform distribution of velocities increases with column diameter.[6] This results from the fact that the probability of velocity fluctuations, i. e. of variation of flow distribution and even their partial separation, is proportional to the cross-sectional area of the column and increases with increasing height and decreasing flow rates.[7] Hence the scaling effect manifests itself as an argumentation of longitudinal mixing.[6] Failure to account for axial mixing may lead to serious errors in scaling up the equipments.

The published experimental results regarding on the axial dispersion coefficients are not sufficient for a broad generalization, and the data on the effect of various factors on the axial mixing, as these were obtained in the previous experiments, are often contradictory in nature.

The main objective of the present study is to measure the axial dispersion coefficients in a 10.8 cm diameter air-pulsed column under single and two phase flow conditions, and to determine the functional dependence of the mixing on the operation conditions and plate geometry.

Table 1. Dimensions of Column

Inside Diameter of Column, D : 10.8 cm

Inside Diameter of Side Arm : 5.2 cm

Effective Height of Column : 180 cm

Plate Free opening Area, S : 0.378, 0.592,
dimensionless

Plate spacing, H : 2.56, 5.01, 7.48, 12.4, 19.7 cn

Plate Thickness : 1.3. 2.7, 5.4 mm

Ranges of operational Variables

Flow Rates : Water, U_c : 0—1 cm/sec
Kerosene, U_d : 0 —1 cm/sec

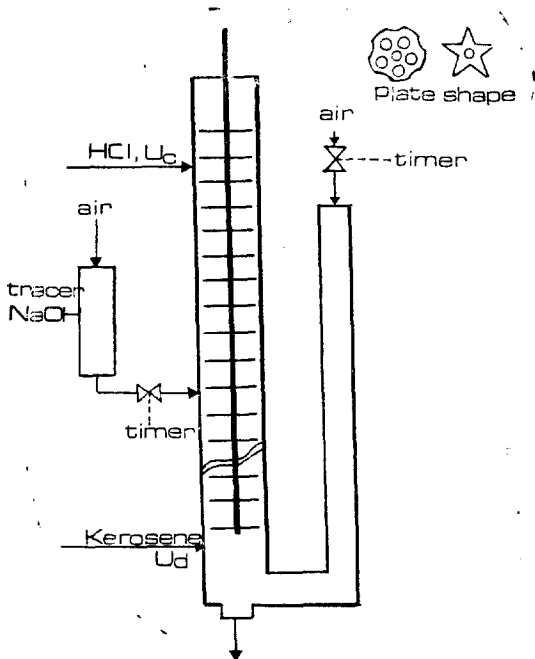Pulsation : Amplitude, A :0.5—8 cm
Frequency, f : 0.5-2.0 Hz

Fig. 1. Apparatus of Pulsed Column

2. EXPERIMENTAL

The air-pulsed extraction column shown schematically in *Figure 1* and the dimensions of the column are summarized in *Table 1*.

The column constructed of five flanged glass sections with a internal diameter of 10.8 cm and the engaging column height was 200 cm.

A side arm of 5.2 cm in diameter, 200 cm in height, was installed for air pulsation. The stainless steel bar of the plate stackwas attached to the rack and filled with plates and spacers.

Two types of plates were employed with 1.3 mm thick stainless steel perforated plate and starshaped plate which were staggered in order to avoid correspondence between edges direction in adjacent plates. The detailed dimensions of the plates are given in *Table 1*. Spacers were also made of stainless steel pipe with a outside diameter of 15.9 mm and 25.4 mm in length.

To pulsate the liquid in the column, compressed air was controlled by a pressure regulator and two solenoid valves which were controlled by a timing controller. The amplitude was controlled by altering time-ratio setting of the air supply and exhaust or changing the feeding air pressure.

The operation ranges of pulsation were varied between a maximum amplitude of 8 cm at 0.5 Hz and a minimum amplitude of 2 cm at 2 Hz. In most previous publications, the frequency was mainly varied at constant amplitude which may have the most significant effect on the axial dispersion. Therefore, in this study, wide variations of amplitude was employed.

Sodium hydroxide (0.95 N) as a tracer material was injected for 0.5 second through the 6.35 mm stainless steel tube ring which has 19 holes of 4 mm in diameter around the ring. The amount of the tracer injected was controlled by the pressure of air supply.
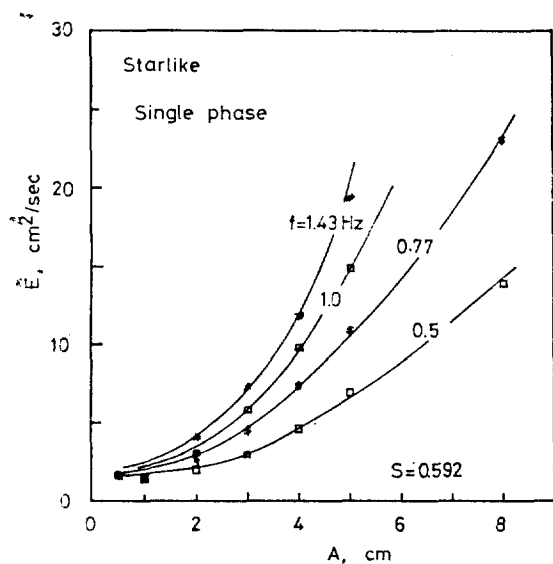
Fig. 2-1. Effect of amplitede on E for H=2.59cm, Uc=0.75cm/sec

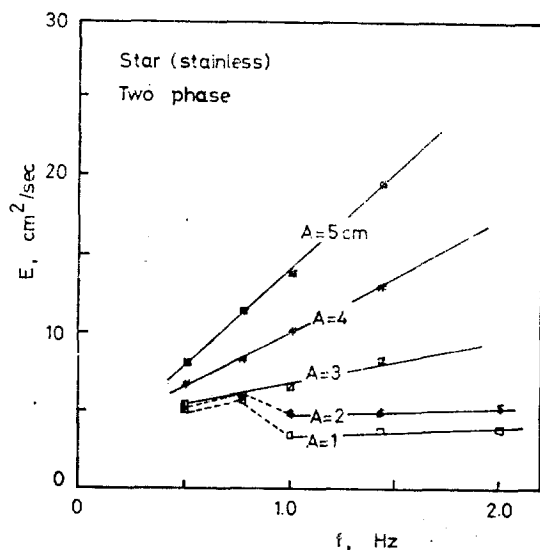Fig. 3. Effect of frequency on E for H=2.59 cm and Uc=0.75 cm/sec
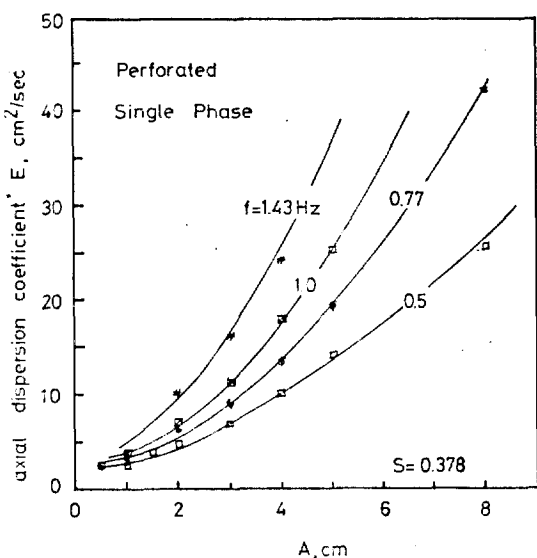

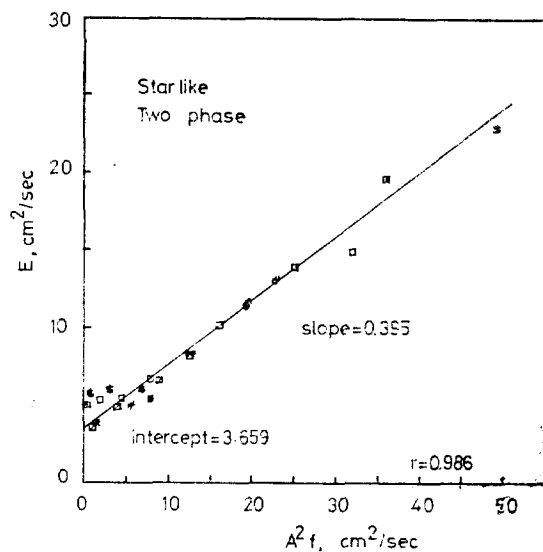Fig. 2-2. Effect of amplitude on E for H=2.56 cm

Fig. 4. Correlation of data (E vs A²f)

3. MEASUREMENTS

It has been reported that[8,23~25] axial mixing coefficients could be measured by using a fast chemical reaction occurring in the pr-

esence of an indicator. In the previous investigation a steady state method and a step change method were developed. Kim and Baird[9] extend the chemical reaction technique to the case where a single pulse of one reactant is injected into a flowing stream of the

other reactant. The reacting components in that case were sodium hydroxide and hydrochloric acid, respectively. In the present study, the above pulse injection method was employed in order to determine the axial dispersion coefficient. From the measurements of the length of color spread with time and completion of reaction time, the coefficient can be derived from the following three equations.[9]

$$t_r = (K^2 E)^{-1} \qquad (1)$$

where $K = 2\pi R^2 C_{10} \sqrt{\pi}/m^2$ in which m_2 refers to the molar amount of base injected.

$$E = x_{rm}^2/2t_r \qquad (2)$$

where x_{rm} is maximum reaction distance and t_r is reaction time.

$$x_r^2/t = -2E \, lnt - 4E ln(K \sqrt{E}) \qquad (3)$$

In equation (3), a plot of x_r^2/t versus the logarithm of time should give a straight line with a slope of $-2E$. This latter method of determining E is not only independent of K but involves an average of many data points in x_r and t, so it is thought to be inherently more accurate than the methods based on Equations (1) and (2).

3-1. Procedures

The column was filled with dilute hydrochloric acid (0.005 N) containing phenolphthalein indicator and the air pulsing system was started. The flow rates of continuous phase (acid) and dispersed phase (Kerosene) were regulated at the desired superficial velocity by means of needle valves and measured by calibrated rotameters. A jeckleg to maintain the interfacial level was also set at the desired optimum height. The pulse amplitude, defined as the linear distance between two extreme positions of the liquid pulse in the main column could be determined by observing the liquid displacement in the side arm.

As soon as the column operating conditions reached steady state the tracer was injected into the column. The amount of tracer injected was measured by observing the displacement of the tracer level in the cylinder. The concentrations of tracer and aqueous phase were predetermined before each experiment.

The resulting red color tended to spread initially and then contract, while travelling down the column at average velocity of contineous phase flow.

The amount of the tracer injections was carefully chosen by experiences of preparative operations so that the red color persists long enough for its length to be measured at 20 seconds interval and that the color disappeared well before it could reach the bottom of the plate stack.

The color length was usually measured about ten times during the reaction time which was 3-4 minutes.

The two phase operations, the dispersed phase holdup was measured by the way of measuring the volume increase of dispersed phase after cutting-off the pulsation and closing the organic and aqueous phases feed valves simultaneously.

Kerosene was used as an organic phase with density of 0.8 gr/cc.

4. RESULTS AND DISCUSSION

The objective of the present study is to provide more additional information on the effects of operating variables and plate geometries on axial dispersion characteristics which has been presented with controversy in the previous publications.

Visual observation indicated that the pulse amplitude gives greater contribution to the axial dispersion than those of any other variables such as frequency, flow rates of each phases and plate spacing.

The reason of employing starlike plates in the column lies in the fact that starlike shape plate has more specific shape and larger free area than those of other plates invetigated previously. The perforated plate was also employed in this study since the plate has been commonly used in a pulsed and reciprocating extraction columns.

EFFECT OF AMPLITUDE AND FREQUENCY

The axial dispersion data for starlike plates showed that the coefficients increased nonlinearly with amplitude, however, it increased linearly with frequency as shown in *Figures 2 and 3.*

The effects of the pulsation were interpreted differently in many previous articles, most of which contended that the axial dispersion coefficient, E has linear relationship. with pulse velocity Af. However, E varied approximately with the product of the square of amplitude and frequency in these experiments.

A correlation of E with A^2f whose dimension are the same as those of E itself is satisfactory as can be seen in *Figure 4.* The intercept in the figure corresponds to dispersion coefficient without pulsation. Similar trends were also reported by Rao, et al.[10]

It was also noted that channeling and circulation phenomena were observed at lower pulsation intensities. The irregular tilting of the color zone verifies the phenomena, which fact reflect as the irregularities in the Figure. Similar trends were also observed in the column of perforated plates.

Effects of continuous and Dispersed phase Flows.

The contin eous phase flow rates had no significant effect on E as can been seen in *Figure 5.* However, E was increased slightly with increasing flowrate of U_c at lowest pulsation.

In the absence of pulsation, axial dispersion tend to increase with U_c was observed. Unfortunately, however, the E values could not be measured experimentally because of severe tilting phenomenon of the color change zones. Veinstein et al.[11] showed that the value of E increases proportionally with the flow rate without pulsations and the effect of U_c found to be negligible at considerably high pulsation intensities.

The effects of the dispersed phase flow on E found to be not appreciable as shown in *Figure 6.* Since the pulsation intensities were reasonably high, the effects of flow rates of two phases on E had minor contribution.

Holdup of dispersed phase at different amplitude and frequency were shown in *Figures 7 and 8.* The dependency and magnitude of holdups for starlike plates on pulsation was smaller than those of perforated plates. This may be due to the difference of free opening area and flow paths through the plates. However, both cases showed that a minimum holdup exists at a certain pulsation intensity which is sometimes called transition frequency[11] at the corresponding amplitude, which is reached faster for perforated plates of small free opening area.

EFFECTS OF PLATE GEOMETRY

Since there are many contradictory arguments,[8,9,12,13] on the effect of plate geometry on axial dispersien, it will be discussed in more depth.

The increased plate spacing H, center-to-center spacing distance between the plates, resulted in a decrease in E as illustrated in
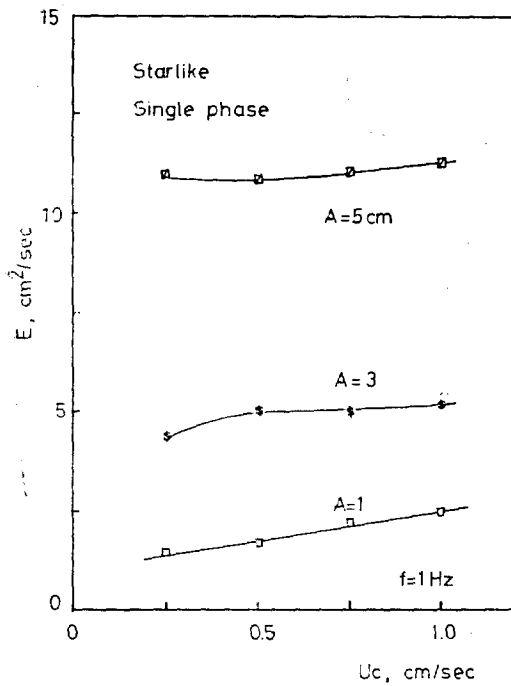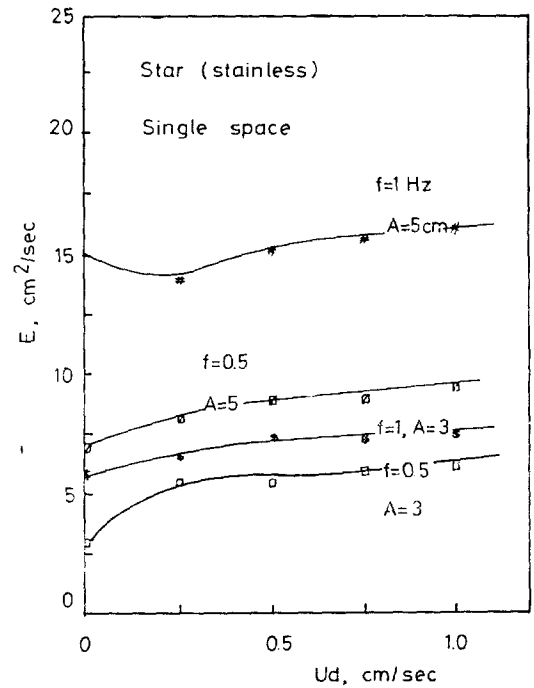
Fig. 5. Effect of Continuous Flow

Fig. 6. Effect of dispersed phase volocity on E.
$U_c = 0.75$ cm/sec

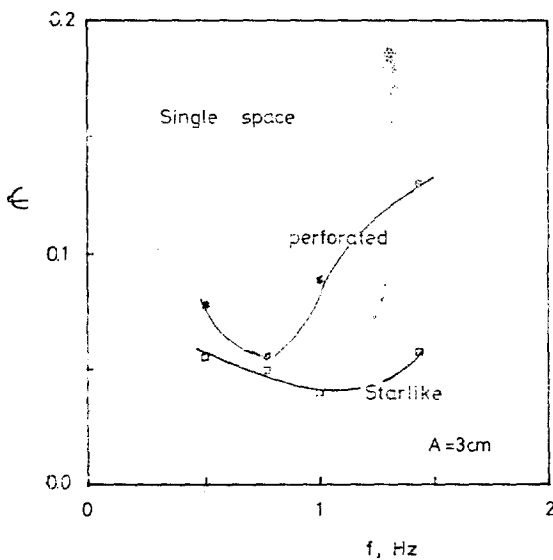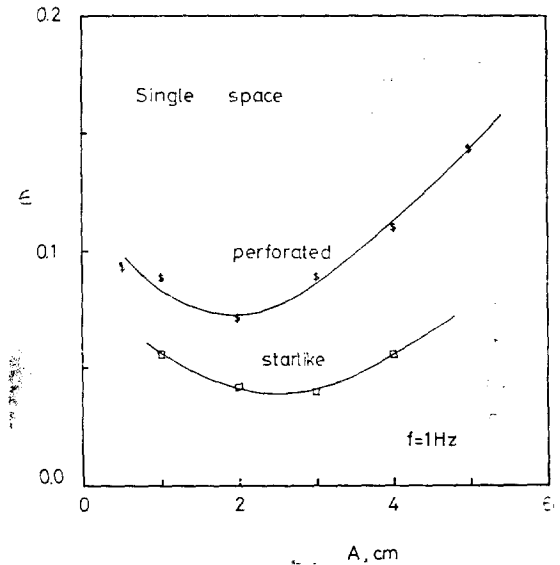Fig. 7. Holdup of dispersed phase vs Frequency
Uc = 0.75 cm/sec   Ud = 0.25 cm/sec

Fig. 8. Holdup of dispersed phase vs Amplitude
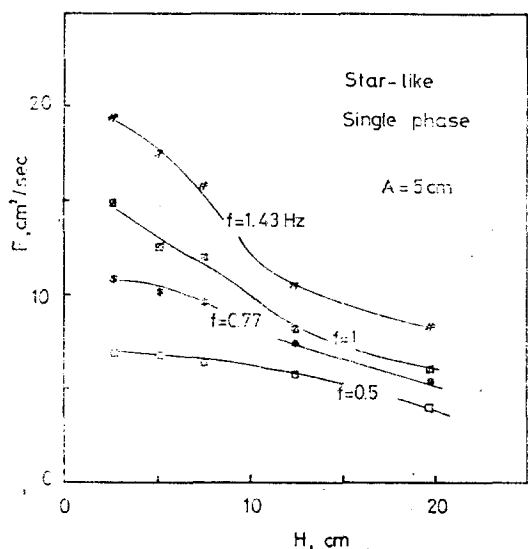Uc = 0.75 cm/sec   Ud = 0.25 cm/sec

Fig. 9. Effects of Plate Spacing
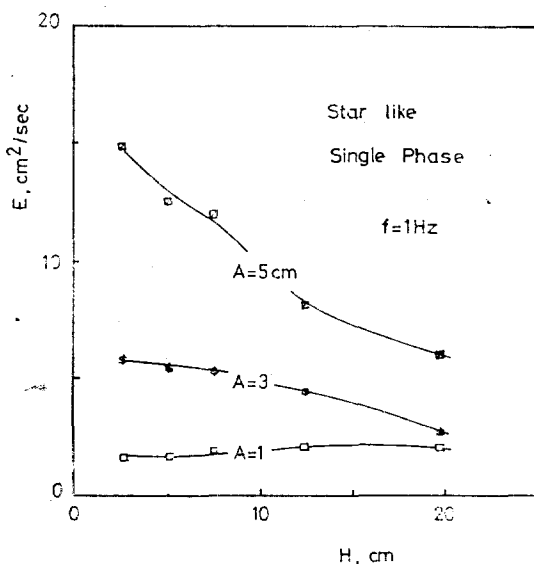Uc=0.75 cm/sec

Fig. 10. Effects of plate Spacing
Uc=0.75 cm/sec

Figures 9 and *10.* The ideal mixed stage model predicts that E would increase linearly with H and this has been confirmed by oldshue—Rushton[14] columns, but in pulsed columns the turbulence level tends to decline with an increase in H so that the interplate regions are no longer well mixed as in the ideal stage model.[15-17]

It is true that the eddy produced at the plate is smaller than the plate spacing in plate columns. Thus the diffusion scale is primarily proportional to this eddy size which is in turn proportional to pulsation degree for a given plate geometry. An increase in plate spacing allows the eddies to disappear appreciably, so the dispersion coefficients decreased with an increase in H. This type of flow regime is called free eddy regime probably confined to plate column.[8]

Negative effect of free area of the plates on the longitudinal mixing is found by comparing *Figures 2-1* and *2-2*, which is consistent with the previous works,[8,12] The free area is difined as the opening fraction of the cross sectional area of the column which is left free for flow. However, free area alone can not give satisfactory illustration of the plate effect in that the plates of different shapes may result in different effects.

Hence, a shaping factor (Q) should be considered in describing the plate geometry. Simply shaping factor can be substituted for the hydraulic radius of the plate, i. e., the ratio of the area of the plate to the wetted perimeter of the plate. Scaling up the column diameter alone results in increasing the ratio, since the size of the plate is accompanied with the column size. Thus the shaping factor Q includes the effects of the column diameter as well as the effects of the plate shape.

Physically a larger Q indicates that the plate is closer to disk-type plate or the column size is relatively large. And a smaller value of Q has a meaning that the plate is similar to mesh-like plate or the column is small in diameter.

There is a discrepancy on whether E increases or decreases with column diameter.[13,18] The free area S was found to have a positive effect on E or a negative one.

Generally speaking, however, the longitudinal dispersion coefficient is increased with decreasing free opening area S, and with increasing shaping factor Q; Which were well shown the effects in the previous studies.[8,18] The present study confirms that S has negative effects on E. The free areas of the starlike plate(Q=1.07) and the perforated plate (Q=1.09) were 0.592 and 0.378, respectively. With respect to the shaping factor, Baird's[9] experiments showed that the effect of semicircular baffle (Q=2.51, s=0.55) is many times greater than that of the meshlike perforated plates (Q=0.69, S=0.61).

The flow fluctutations around the plates were observed to be more intense with the hydraulic radius namely the shaping factor
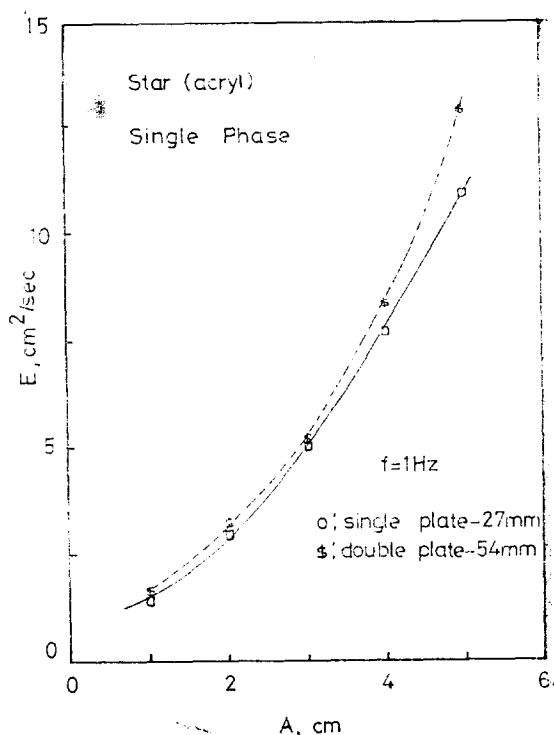

Fig. 11. Axial dispersion coefficient versus amplitude with different plate thickness Uc=0.75 cm/sec

Table 2. Published Data for Correlation of E

Reference	Experimental Details			Correlations of E
	D(cm)	H(cm)	S(—)	
Novotny[16]	5.16	2.5 to 15	0.029 to 0.16	$E=\dfrac{0.15HdAf}{(H-4.5)S^{3/2}}$ where H>4.5
Miyauchi[18]	3.2 5.4	1—10 (9-values)	0.095 0.19	$E=\dfrac{u_cH}{2B-1/n}+AfH(1-\varepsilon)/B$ $B=0.57(HD^2)^{1/3}S/d$
Rosen[13]	10	3.75 7.5 15.3	0.095 0.23 0.32	$E=3zl^{2/3}d1^{/3}(u_c+0.5u_d+Af)$ $1=H/(H+H/D)$
Kagan[20]	5.6	5	0.087	$E=0.49H^{0.76}(Af+u_c)$
Aerov[21]	5.6	5	0.082	$E=1.2\times10^5\dfrac{A^{1.2}f^{1.35}}{u_c^{1.4}}$
Navatov[22]	12.0	4.5	0.83	$E=0.5u_cH+0.402\dfrac{A^2f}{H^{1.2}S^{1.9}}$
*Kim[9]	5.0	2.8-59	0.57	$E=1.98A^{1.7}f^{0.95}H^{-0.69}$

*reciprocating plate column[4]

Table 3. Intercepts and Slopes of the plot E vs A²f

Free Area S	spacing H(cm)	u_d (cm/sec)	K_1 (cm²/sec)	K_2
0.378	2.56	0.	3.574	0.811
0.378	5.01	0.	3.58	0.807
0.378	2.56	0.25	3.596	0.880
0.378	5.01	0.25	2.756	0.849
0.592	2.83	0.	1.454	0.374
0.592	2.59	0.	1.383	0.463
0.592	2.59	0.25	3.659	0.395
0.592	5.05	0.	1.697	0.405
0.592	7.48	0.	2.164	0.352
0.592	12.4	0.	2.80	0.196
0.592	19.7	0,	3,04	0.111

is larger owing to vortex shedding and circulation effects. This claims that E is increased with shaping factor, Q.

The thicknness of the plate may be a parameter to consist of the plate geometry, however, the effect of plate thickness is not susceptible. A slight increase in E resulted from doubling the plate thickness may be seen in *Figure 11*. This effect was also found by Mar and Babb.[19]
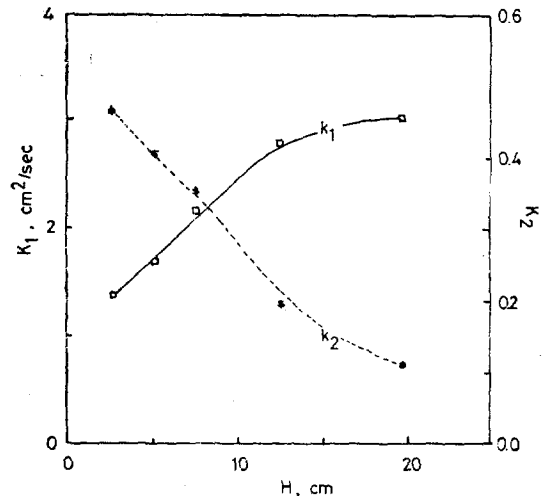
5. CORRELATION

Several correlations of E were presented by previous publications, which is summarized in *Table 2*. The linear character of the first three correlations with respect to pulse velocity, Af, suggested that the starting theoretical concepts have been oversimplified. The attention of the cited papers was concentrated on the pulsation effect.

The comparison of the E in the cited papers with that of present study gives a suggestion that E has a proportional relationship with A²f whose dimensions are the same as those of E. Therefore, the following form of correlation may be suggested;

$$E = K_1 + K_2 A^2 f \qquad (4)$$

where K_1 and K_2 can be found from the intercept and the slope of the fitting line in the plot of E vs A² f. The resulting values of K_1 and K_2 are shown in *Table 3*.

We may assume K_1 and K_2 are functions of the plate geometry alone at constant flow conditions. Increasing the free opening area results in decreasing both K_1 and K_2 (*Table 3*), whereas increasing plate spacing results in increasing K_1 and decreasing K_2 as shown in *Figure 12*.

Fig. 12. Plate Spacing versus K_1 and K_2

A regression analysis gives the following correlations;

$$K_1 = 0.382 \ S^{-1.78} \ H^{0.38} \qquad (r = 0.978)$$
$$K_2 = 0.364 \ S^{-1.58} \ H^{-0.58} \qquad (r = 0.945)$$

Although K^1 and K^2 have been correlated with plate free area and plate spacing alone, considerable further research is needed on the effect of plate geometry with respect to not only S and H but also shaping factor which may be defined as the hydraulic radius of the plate.

CONCLUSIONS

1. The longitudinal dispersion coefficients for a continuous phase are mainly affected by the pulsation intensity and has a proportional relationship with $A^2 f$ rather than Af. It is also noted that circulation or channeling phenomena is observed at lowest pulsation.

2. The flow rates of each phase have no significant effects on axial mixing whereas free opening fraction of the plate and plate spacing have negative effect on E.

3. A correlation of E in terms of pulsation intensity and plate geometry is presented as,

$$E = 0.38 \ S^{-1.78} \ H^{0.38} + 0.36 \ S^{-1.58} \ H^{-0.58} A^2 f$$

Nomenclature

A : amplitude of pulsation, cm

C_{10} : initial acid concentration, mole cm^{-3}

D : diameter of column, cm

E : axial dispersion coefficient, cm^2/sec

f : frequency of pulsation, Hz

f_a : transition frequency, Hz

H : plate specing distance, cm

K_1 : correlation constant in the absence of pulsation, cm$_2$/sec

K_2 : correlation constant in the presence of pulsation, dimensionless

K : constant defined as $2\pi R^2 C_{10} \sqrt{\pi}/m_2$

m_2 : amount of tracer, mole

Q : shaping factor, cm

r : correlation coefficient

R : radius of column, cm

S : free opening fraction of the plate, dimensionless

t : time, sec

t_r : reaction time, sec

U_c : superficial velocity of continuous phase, cm/sec

U_d : superficial velocity of dispersed phase, cm/sec

X_r : reaction axial distance, cm

ρ : density, g/cc

ϵ : holdup of organic phase

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