Eng. & Technology, Vol.24, No.1, 2005

Back Mixing And Optimization Of Pulsed Plate Column Employing The Central Composite Rotatable Design

*Majid Saleem Radhaa**

Received on: 20/5/2004
Accepted on: 20/11/2004

Abstract

Study of the hydrodynamics of pulsed column is of vital importance in the scale up and understanding of such columns. The back mixing appears during the study of the hydrodynamic of the pulsed column, therefore the effect of the following variables on the back mixing, namely, frequency, amplitude, dispersed phase velocity and aqueous to organic phase flow rate ratio were studied. The central composite rotatable design was used in this study to conduct the effect of the studied variables. Accordingly, twenty-five experiments were needed with an extra 5 used for replication. Moreover Hooke & Jeeves pattern optimization method was adapted to determine the optimum conditions with respect to the studied variables. A column of 20 baffled 5 cm compartments was used, with an inside diameter of 5.08 cm. Gas – oil – water system was used under binary condition. The results shows that the optimum conditions at maximum back mixing are frequency (1.6 s^{-1}), amplitude (4.5 cm), dispersed phase velocity (0.508 cm/s) and aqueous to organic phase flow rate ratio (0.5).

الخلط العكسي و اختيار الأفضل في الأعمدة النبضية بتطبيق تقنية تصميم التجارب لبوكس – ولسن

الخلاصة

إن دراسة السلوك الهيدروديناميكي للأعمدة النبضية له أهمية حيوية في تصميم و فهم سلوك مثل هكذا أعمدة. يظهر الخلط العكسي خلال دراسة السلوك الهيدروديناميكي لهذه الأعمدة و عليه تم دراسة تأثير المتغيرات التالية على الخلط العكسي و هي: التردد، مدى التردد، سرعة الطور المشتت، و نسبة الطور العضوي إلى الطور المائي. لقد تم تطبيق تقنية بوكس – ولسن في تصميم التجارب في هذه الدراسة و عليه تم أجراء خمسة و عشرون تجربة بالإضافة إلى خمسة تجارب أخرى لأغراض إحصائية. كذلك تم تطبيق طريقة (hooke & Jeeves) الخاصة بأختيار الأفضل لغرض الحصول على القيم المثلى للمتغيرات. لقد تم في هذه الدراسة استخدام عمود قطره الداخلي (٥,٠٨ سم) مزود بعشرين صفيحة مثقبة كانت المسافة بين صفيحة و أخرى ٥ سم و كان النظام المستخدم هو زيت الغاز – الماء كنظام ثنائي.
اوضحت النتائج ان القيم المثلى للمتغيرات كانت كالآتي: التردد (١,٦ ثانية⁻¹)، مدى التردد (٤,٥ سم)، سرعة الطور المشتت (٠,٥٠٨ سم/ثانية)، و نسبة الطور العضوي إلى الطور المائي (٠,٥).

Keywords: *Central composite rotatable design, Back Mixing and Pulsed Plate Column*

* Chemical Engineering Department / University of Technology
Email: majid_radhaa@yahoo.com

https://doi.org/10.30684/etj.24.1B.5
2412-0758/University of Technology-Iraq, Baghdad, Iraq

INTRODUCTION:

The pulsed perforated – plate column is widely used for a diverse range of industrial processes. In this column, energy is added in the form of a cyclic pulsing motion, usually sinusoidal, superimposed on the counter – current flow of the liquid phase. The action disperses one of the liquids into drops as it is forced through small perforations in the fixed horizontal plates and agitates the continuous phase. This technique in liquid – liquid extraction in addition to its various merits has proved to be beneficial in saving energy requirements as compared with classical electrical driven rotating disc contactor (RDC) column [1].

Many authors [2 –5] have observed the existence of different flow in pulsed columns. Though these regions are ill defined, it is useful to distinguish between the principles regions of operation. Since the perforated plates usually permit the passage of the dispersed phase only during half of the pulsed cycle, it follows that at low agitation the drops will tend not always to be dispersed and the behavior of the column will approximate to that of a mixer – settler. At low agitation intensity, holdup is high and decreases as agitation intensity increases until a minimum is reached corresponding to the beginning of the transition (or dispersed) region. The dispersed region, occurring at intermediate agitation levels, is characterized by low droplet coalescence between successive pulses. The drops are large and uniform in size moving predominantly in the vertical direction. Moreover, the holdup increases slowly as the agitation intensity increases. Emulsion – type operation occurs at high agitation levels, is characterized by relatively small drops and high degree of turbulence, and thus provides good conditions for mass transfer. However, considerable back mixing may occur which lowers the driving potential for back mixing [1,6].

Measurements of flooding in a pulsed sieve plate extraction column were carried out using toluene – acetone – water system. The influence of plate spacing and initial acetone mass fraction in toluene (in the case of the three – component system) on flooding curves was studied and empirical correlations for maximum of these curves are presented [7].

The two basic principles of experimental design are replication and randomization. Replication means a repetition of the basic experiment. It allows the estimation of the experimental error and the determination of whether observed differences in data are really statistically different. Thus, replication permits the experimenter to obtain a more precise estimate of this effect. Randomization is the cornerstone underlying the use of statistical method in experimental design. Randomization means that both the allocations of the experiment material and the order in which the individual experimental runs to be performed are randomly determined. Proper randomization will assist in averaging out the effect of extraneous factors that may be present.

Three basic types of statistically designed experiments are most often used in chemical industry. These are:

1- Factorial design.
2- Fractional factorial design, and
3- Central composite rotatable design, CCD, (Box – Wilson composite rotatable design).

Central composite rotatable design is applicable to optimization analysis. In this approach a special series of tests are defined. This technique was used in the study of the important parameters of practical significance for the operation of pulsed columns. It has showed benefits in organized approach towards the collection and analysis of data and it gives maximum information per run. The experimental results of these tests then serve as the basic for developing a simplified mathematical function, usually a polynomial, representing the relationship between the variables and the response; in addition, the reliable prediction of the response data in areas not directly covered by experimenttation. The number of terms (P) variables in the mathematical model is therefore: [(P+1)(P+2)/2], where P is the number of variables. For the purpose of the experiments, the independent variables are specified into five levels. The specific value of the five levels depends on the number of variables included and the range over which they are to be studied [8].

EXPERIMENTAL WORK:

The experimental setup consisted of a test section of 0.05 m inside diameter and 1 m high, sectioned into 20 stages (with 10 sieved plates and 10 baffles). The plate perforation diameter equal to 3 mm with fractional free area of 20%. The dispersed phase (gas-oil) fed at the bottom to move upwards counter – currently to the continuous phase (water) [9]. The physical properties of the continuous and dispersed phases are shown in Table (1).

The experimental variables studied were; pulse frequency (0.4 – 1.6) s^{-1}, pulse amplitude (0.5 – 4.5) cm, dispersed phase superficial velocity (0.283 – 0.894) cm/s, and aqueous to organic flow rate (0.5 – 2.5). The dispersed phase holdup was obtained by measuring the relative volume of the phases at seven different positions along the length of column under steady state condition. Further details are presented elsewhere [9].

EXPERIMENTAL DESIGN:

The experimental program was defined as the study of important parameter for the operation of pulsed extraction column [10]. The continuous phase back mixing coefficient was studied as the response function. The preliminary step is to setup the relationship between the coded levels (X_j, where; j=1, 2, 3,4) and the corresponding real process variables according to the following equation [8]:

$$X_{coded} = \frac{X_{actual} - X_{center}}{\frac{X_{center} - X_{\min}}{\sqrt{P}}} \quad ...(1)$$

Hence the correlation would be as follow:

1- In case of frequency, f: $X_1 = \frac{f-1}{0.3}$

2- In case of amplitude, A: $X_2 = \frac{A-2.5}{1}$

3-In case of dispersed phase superficial velocity, V_d: $X_3 = \frac{V_d - 0.566}{0.1415}$

4-In case of aqueous to organic flow ratio, R: $X_4 = \frac{R-1.5}{0.5}$

The experiments needed were 25 runs and an additional 5 used as a checkup. The design of these runs is shown in Table (2). A model of a second order degree was used to approximate the response because of curvature in the true surface, which proved to be adequate approximation. The model is:

$$y = B_o + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} X_i^2 + \sum_i \sum_j B_{ij} X_i X_j \quad .....(2)$$

RESULTS AND DISCUSSION:

Study the parameters effecting back mixing is essential in the study and design of pulsed plate columns due to the influence imposed by back mixing in mass transfer. And according to Vassallo [4] the researchers could only qualify rather than quantify the effects of operating parameters on back mixing, thus optimization method and statistical analysis were conducted to quantify the effective variables. The effect of introducing baffles in the geometry of the column was also under concern. The attainment anticipated in reducing back mixing will be explicated as an improvement of the efficiency of pulsed – plate columns. Continuous phase back mixing coefficient (E_c) was taken as a measure of the extent of back mixing. (E_c) is calculated according to:

$$Pe_m = \frac{E_c}{L \, V_c} \quad .....(3)$$

The value of the modified Peclet number (Pe_m) denotes the degree of back mixing.

If $Pe_m \rightarrow 0$ this indicates inconsequential dispersion, hence plug flow prevails. And when $Pe_m \rightarrow \infty$ then large dispersion will eventuate, hence back mixing is comprehensive [11].

The results of the experiments listed in table (2) was presented using a second order polynomial; equation (2); the coefficients of the polynomial are regressed nonlinearly by a programmable software namely statistica (the results are given in details by Mansour 1993).

The results of the regression are presented in Table (3) with a correlation coefficient of 0.856 and an average absolute error of 4.1974435. Moreover, optimum conditions at maximum back mixing were determined using Hooke and Jeeves pattern optimization method. The optimization conditions are given in Table (4).

The effect of each variable on Peclet number is shown in figs. (1 – 4), with keeping other variables at optimum values in each case.

Figure 1, shows the relation between the frequency of pulsation and Peclet number. It is clearly demonstrated that by increasing the frequency of pulsation, Peclet number increases which means increasing in back mixing (referring to equation 3) and this behavior can be attributed to that extensive back mixing occurred in the continuous phase caused by the additional droplets of the dispersed phase that were transported upward through the column by increasing the frequency. The transport of these droplets will increase the recycling of the continuous phase or by other words increasing the turbulence of the continuous phase, leading to the increment noticed in the back mixing. Swaft and Burger [12], Ingham [13], and Grag and Pratt [14] noticed this behavior also.

The effect of amplitude of pulsation on Peclet number is shown in fig. 2. It is clearly show that by increasing the amplitude of pulsation, the Peclet number increases leading to an increase in the back mixing. The explanation proposed in clarifying the effect of frequency is also useful to explain this behavior. As a final conclusion, increasing the pulsation intensity (frequency and amplitude) resulted in increases in back mixing coefficient.

Figure 3 represent the effect of dispersed phase superficial velocity on Peclet number and it is clearly shows an increase in Peclet number with increasing the superficial velocity to a certain point then starts to decrease with increasing velocity, which results a decrease in back mixing with this continuous increasing. This behavior can be attributed to, by applying a pulse to the column the pulsation force inside the column will cause the increase in back mixing as discussed above, and by increasing the velocity of the dispersed phase the back mixing increases for the same reason up to a limit, then the force gained by the droplet from the velocity increment is being larger than that gained by pulsation which will enforce the droplet to flow upwards in channels leading to decrease in the turbulency of the continuous phase which will finally cause a decrease in back mixing, this was also noticed by Kasipathi et al. [15]. They noticed that this channeling effect would diminish with increasing plate spacing.

While figure (4) shows the effect of aqueous to organic flow ratio on Peclet number, and it can be seen clearly that by increasing the flow ratio a decrease in Peclet number was resulted as a final result a decrease in the back mixing coefficient is observed. This can be explained as by decreasing the organic phase (dispersed phase) flow rate (increasing the ratio of aqueous to organic flow ratio) the turblency of the continuous phase will decrease. In other view of point, by increasing the continuous flow rate (increasing the ratio) the gravitational forces will be larger than the floating forces caused by the dispersed leading to a decrease in the turbulancy of the continuous phase which will finally cause a decrease in back mixing. This result is in accordance with those found by Thornton[16].

Moreover, a statistical study was made to demonstrate the interaction parameters effect on the observed equation, and the results show that only the terms (x_1x_2, x_1x_4) has a significant effect on the observed equation. This was also clarified in figs. (5 – 16). Table 5 shows the results of this study. Therefore, the proposed equation can be written as in equation 4, which describes the effect of the studied variables on the back mixing coefficient.

$$y = 0.2122 + 0.0348X_1 + 0.0514X_2 - 0.0191X_3 - 0.0671X_4 + 0.022X_1X_2 - 0.026X_1X_4 + 0.011\ X_1^2 - 0.0171\ X_2^2 - 0.0317\ X_2^3 + 0.015X_2^4 \quad \ldots\ldots\ldots 4$$

Nomenclature:

A	amplitude of pulsation (cm)
B	equation coefficient
E	back mixing coefficient (cm^2/s)
f	frequency of pulsation (1/s)
L	spacing (cm)
P	number of variables
Pe	modified Peclet number
R	continuous to organic phase ratio (-)
V	superficial velocity (cm/s)
X	variable

Subscript:

m	mixture
c	continuous

References

1- Prabhakar A., sriniketam G. and Varma Y. B. G., "Dispersed Phase Holdup and Size distribution in pulsed Plate Columns", Can J. Chem. Eng., 66, 232-240 (1988).

2- Bell R. L. and Babb A. L., "Holdup and Axial Distribution of Holdup in Pulsed Sieve – Plate Extraction Column", I&EC Proc. Des. Dev., 8 (3), 392-400 (1969).

3- Kumar A. and Hartland S., "Prediction of Dispersed Phase holdup in Pulsed perforated extraction Columns", chem.. Eng. Process, 23, 41-59 (1988).

4- Vassallo G., "The hydrodynamic Behavior of a Pulsed Column", EUR 8597 – Series: Nuclear Science and Technology, 1983.

5- Rvcic L. M., Pratt H. R. C. and Stevens G. W., "Axial Dispersion in Pulsed Perforated – Plate extraction Columns", AICHE j., 35, No. 11, Nov., 1845 – 1855 (1989).

6- Tung L. S., and Luecke R. H., "Mass Transfer and Drop Size in Pulsed – Plate Extraction Columns" I&EC Proc. Des. Dev., 25, 664 – 673 (1986).

7- Tribess A. and Brunello G. "Flooding in Pulsed Plate Extraction Column with Mass Transfer Effects", Braz. J. Chem. Eng., Vol. 15, No. 1, March (1998).

8- Montgimery D. C., "Design and Analysis of Experiments", John Wiley & sons, New York, (1976).

9- Mansour E. F., "Hydrodynamic Behavior of Pulsating Liquid – Liquid Extraction Columns", Ph. D. Thesis, University of Baghdad (1993).

10- Porta J. J. and Merz A., "Experimental Design, Deconvolution of RTD Data and Response Surface Analysis in solvent Extraction", ISEC (1986), Munchen, Vol. 3, 207 – 214.

11- Levenspiel O. and Fitzgerald T. J., " A Warning on The Misuse of The Dispersed Model", Chem. Eng. Sci., 38, No. 3, 489 (1982).

12- Swaft W. H., and Burger L. L., " Back Mixing in Pulsed Column. Part II: experimental Values and Effect of Solvent Variables", HW – 29010, Aug. 1953.

13- Ingham J. "The Study of Longitudinal Mixing in Liquid – Liquid Extractor", sited in Hanson C., "recent Advances in Liquid – Liquid Extraction", Pergamon Press, Oxford (1971).

14- Garg M. O. and Pratt H. R. C., "Effect of Column Diameter on Back Mixing in Pulsed plate Column", I&EC Proc. Des. Dev., 20, 492 (1981).

15- Kasipathi Rao K. V., Jeelani S. A. k., and Balasurbramanian G. R., "Back Mixing in Pulsed Perforated Column", Can. J. Chem. Eng., Vol. 56, 120, Feb. (1978).

16- Thornton J. D., "The Extraction Efficiency of Mixer – settler Contractors", Chem. Eng. And fuel Sci., Newcastle Univ., 25, Nov. (1957).

Table (1) Physical properties of the dispersed and continuous phases

Continuous Phase		Dispersed Phase	
Viscosity	$1x10^{-3}$ N.s/m	Viscosity	$2.125x10^{-3}$ N.s/m
Density	1000 kg/m	Density	810.7 kg/m
Interfacial Tension (σ) = $43.24x10^{-3}$ N/m			

TABLE (2) Coded and experimental design Variable

Run No.	f	A	R	V_d	Run No.	f	A	R	V_d
1	-1	-1	-1	1	16	1	-1	1	-1
2	-1	-1	1	-1	17	1	1	-1	-1
3	-1	1	-1	-1	18	1	1	1	1
4	-1	1	1	1	19	0	0	0	0
5	1	-1	-1	-1	20	0	0	0	0
6	1	-1	1	1	21	2	0	0	0
7	1	1	-1	1	22	-2	0	0	0
8	1	1	1	-1	23	0	2	0	0
9	0	0	0	0	24	0	-2	0	0
10	0	0	0	0	25	0	0	2	0
11	-1	-1	-1	-1	26	0	0	-2	0
12	-1	-1	1	1	27	0	0	0	2
13	-1	1	-1	1	28	0	0	0	-2
14	-1	1	1	-1	29	0	0	0	0
15	1	-1	-1	1	30	0	0	0	0

Table (3) Constants of the Second Order Model

Parameter	*Value*	Parameter	Value
B_0	0.2122	B_{14}	-0.0269
B_1	0.0348	B_{22}	-0.0171
B_2	0.0514	B_{23}	-0.0086
B_3	-0.0191	B_{24}	-0.014
B_4	-0.0671	B_{33}	-0.0317
B_{11}	0.0109	B_{34}	0.0029
B_{12}	0.0221	B_{44}	0.0159
B_{13}	0.0800		

Table (4) The results of Optimization

Variable	Optimum value (Coded)	Optimum value (Coded)
X_1	2	1.6
X_2	2	4.5
X_3	-0.4104	0.508
X_4	-2	0.5

Table (5) variance analysis of variable effects on Peclet number

	X_1	X_2	X_3	X_4	X_1X_2	X_1X_3	X_1X_4
B_V	0.0348	0.05142	-0.0191	-0.0671	0.0221	0.008	-0.0269
$(B_V)^2$	0.00121	0.00264	0.00036	0.0045	0.00049	6.4E-05	0.00072
$\Sigma(V_i)^2$	24	24	24	24	16	16	16
Sb^2	3.652E-05	3.653E-05	3.653E-5	3.653E-5	5.48E-05	5.48E-05	5.48E-05
F-value $(B_v)^2/Sb^2$	33.213701	72.363477	9.9692248	123.1818	8.935266	1.168696	13.17673
F (1,15)=4.45 0.95 Confidence	S	S	S	S	S	NS	S

	$X_2 X_3$	$X_2 X_4$	$X_3 X_4$	X_1^2	X_2^2	X_3^2	X_4^2
B_V	-0.0086	-0.014	0.00288	0.01092	-0.0171	-0.0317	0.0159
$(B_V)^2$	7.44E-05	0.000196	8.27E-06	0.00012	0.0003	0.001	0.00025
$\Sigma(V_i)^2$	16	16	16	48	48	48	48
Sb^2	5.48E-05	5.48E-05	5.48E-05	1.83E-05	1.83E-05	1.83E-05	1.83E-05
F-value $(B_v)^2/Sb^2$	1.357068	3.575707	0.15099	6.53001	15.96458	55.01668	13.88014
F (1,15)=4.45 0.95 Confidence	NS	NS	NS	S	S	S	S

S: Significant, NS: not significant

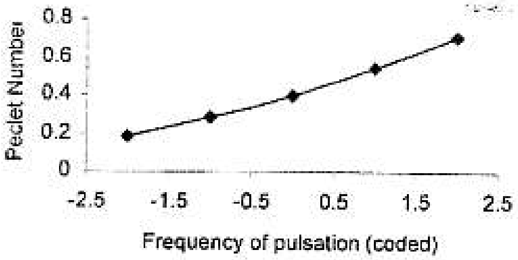

fig.1 The effect of frecuency of pulsation on Peclet number keeping other variables at optimum values

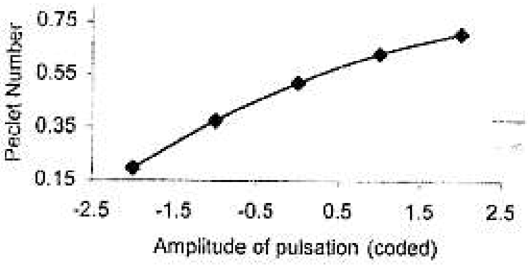

fig.2 The effect of amlitude of pulsation on Peclet number keeping other variables at optimum values

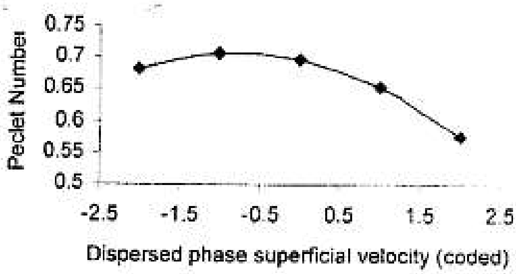

fig.3 The effect of dispersed phase superficial velocity on Peclet number keeping other variables at optimum values

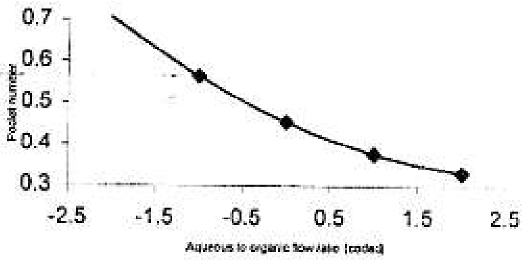

fig.4 The effect of aqueous to organic flow ratio on peclet number keeping other variables at optimum values

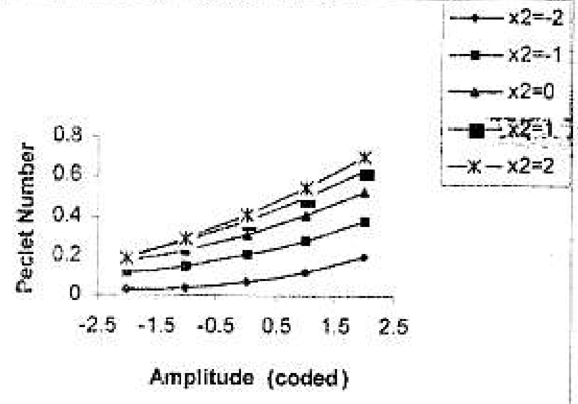

fig.5 the effect of amplitude andfrequency of pulsation on Peclet No.

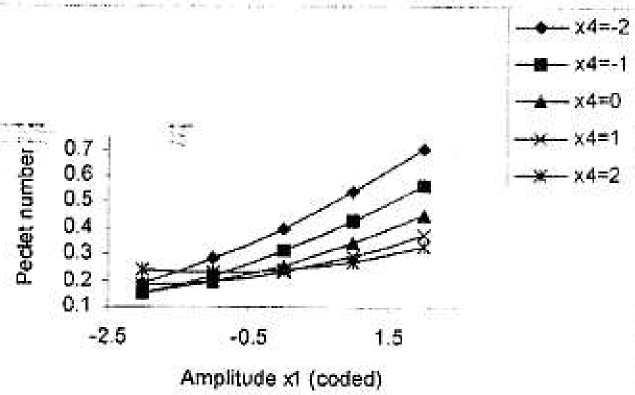

fig.7 The effect of amplitude of pulsation and aqueous to organic flow ratio on Peclet no.

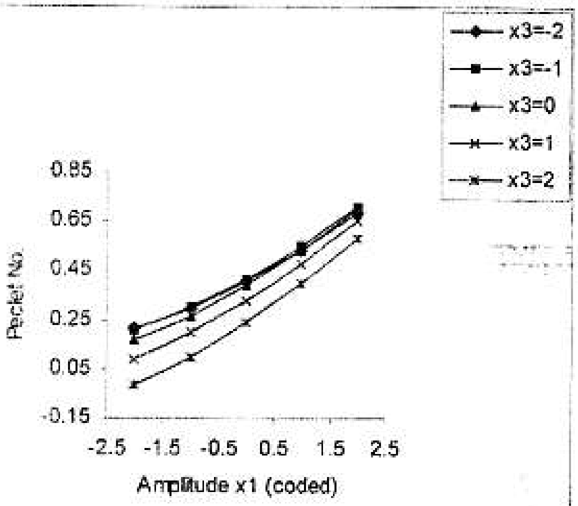

fig.6 The effect of amplitude of pulsation and the dispersed phase superficial velocity on Peclet No.

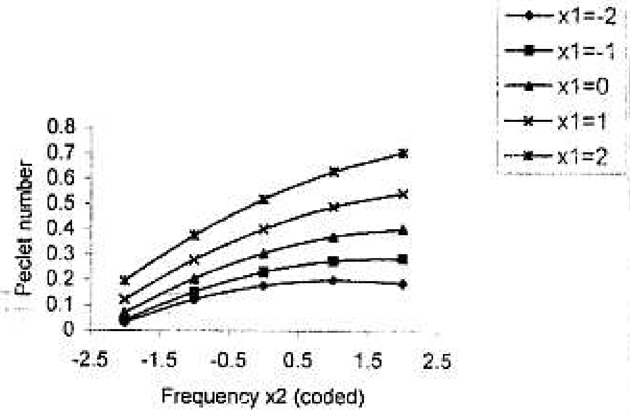

fig.8 The effect of frequency and amplitude of pulsation on Peclet number

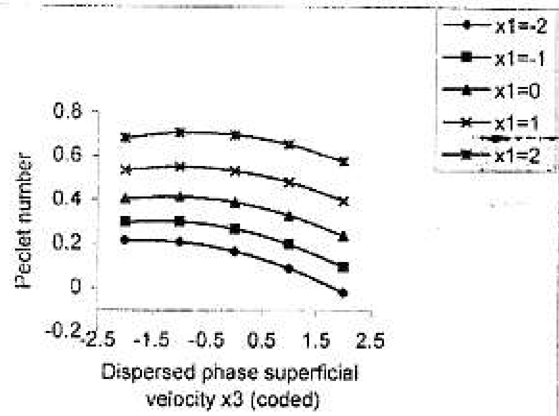

fig.9 The effect of dispersed phase superficial velocity and amplitude of pulsation on peclet no.

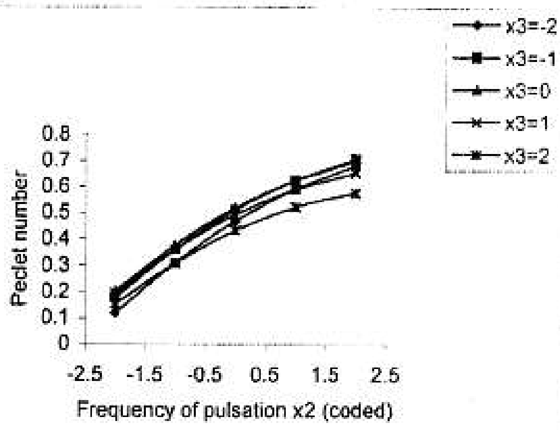

fig.11 The effect of frequency of pulsation and dispersed superficial velocity on Peclet no.

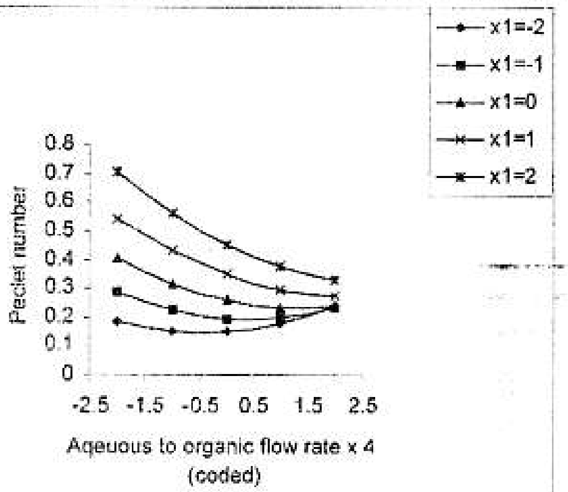

fig.10 The effect of aqeuous to organic flow ratio and amplitude of pulsation on Peclet no

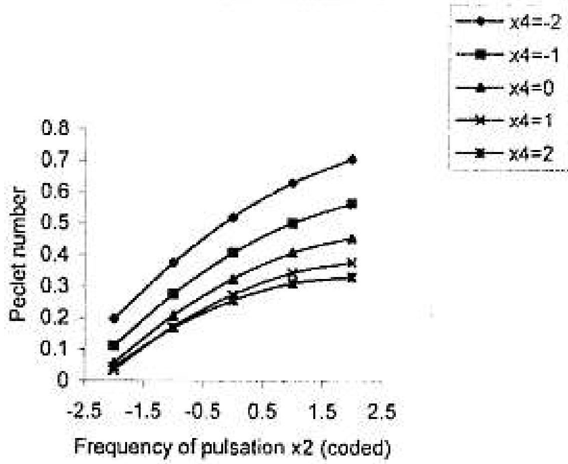

fig.12 The effect of frequency of pulsation and aqeuous to organic flow ratio on Peclet no.