Mass Transfer Studies in Three-phase Fluidized Bed Using Response Surface Method

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Mass transfer characteristics of co-current three-phase fluidization were determined in terms of mass transfer coefficient and Sherwood number using Box-Behnken method. The experiment was carried out in a 5.4 cm I.D, 6 cm O.D and 160 cm high vertical Perspex column. Gypsum particles of diameter 0.0842 cm, 0.1676 cm and 0.2818 cm, water, and air were used as solid, liquid and gaseous phase respectively. Initially, the superficial liquid velocity was maintained constant and superficial gas velocities varied. After attaining steady state, at a particular gas velocity, the fluidized bed height and manometer readings were recorded for pressure drop estimation. The above-mentioned procedure was repeated for four different liquid velocities in a fluidized bed. The effect of individual phase holdup and mass transfer coefficient for various particle sizes with the specific liquid flow rates and gas flow rates were studied. It was observed that the mass transfer coefficient and Sherwood number increased with increase in superficial gas velocity and particle size in cocurrent three-phase fluidized bed. A quadratic model for bed porosity, gas holdup, Sherwood number and mass transfer coefficient were developed using response surface method (RSM).

Key words:
Box-Behnken method, co-current three-phase fluidization, Sherwood number, mass transfer coefficient, response surface method

Introduction

Three-phase fluidization is a subject of fundamental research over the last three decades due to its industrial importance. Three-phase fluidized beds have been applied successfully to many industrial processes, such as Fischer-Tropsch process, biocatalysts fluidized bed reactors (FBR) for aerobic and anaerobic wastewater treatment, petrochemical and biochemical processes because of their higher heat and mass transfer rates, low pressure drop, simple operation, low operation costs, and good contact efficiency between different phases.[1–7] Co-current gas–liquid–solid fluidization is defined as an operation in which a bed of solid particles is suspended in gas and/or liquid upward flowing media due to the net gravitational force on particles. This enhances intimate contact among the gas, liquid and solid particles, and provides substantial advantages for applications in physical, chemical or biochemical processing involving gas, liquid and solid phases.[8] For design of three-phase fluidized bed, it is important to study the hydrodynamics and mass transfer characteristics. Design of FBR depends on bed height, porosity and bubble size. Mass transfer is influenced by gas holdup and liquid holdup.[15,16] The particles in the bed are usually porous or small, so that they increase the contact area with the continuous and dispersed phases.[9,11] In addition, the three-phase circulating fluidized bed, which can increase the heat and mass transfer coefficients at a higher range of superficial liquid velocity, can be useful for regenerating the deactivated

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catalyst, adsorbent, absorbent continuously, and minimizing the dead zone in the reacting or contacting system by means of the circulating fluidization mode.[10,12] In this present study, the solid-liquid mass transfer has been investigated in cocurrent three-phase fluidization using response surface method. The optimized experimental condition to achieve better mass transfer coefficient and Sherwood number are obtained by RSM, instead of performing enormous work, this software will direct us to conduct the minimum number of trial runs to achieve the better results where we can reduce the number of experiments.

Response surface method

The response surface method (RSM) is a statistical and mathematical technique used for modeling and optimization of processes in which a response of interest is influenced by several variables. The RSM has important application in the design, development and formulation of new products, as well as in the improvement of existing product designs. It defines the effect of the independent variables on the process, either individually or collectively. Further, the experimental methodology generates a mathematical model, which describes the chemical processes. The response surface method has been very popular for optimization studies in recent years. The design procedures of the response surface methodology are as follows (i) A mathematical model for the second order response surface with the best fit is developed, (ii) the optimal sets of experimental parameters that produce a maximum or minimum value of response are found, (iii) the response surface plot and contour plot of the response as a function of the independent parameters and optimal points are determined. Response surface method offers several designs depending on the number of design factors. Among them Box-Behnken is very convenient and easily applicable. RSM attempts to analyze the influence of the independent variables on a specific dependent variable (response). The independent variables, denoted by $x_1, x_2 \ldots x_k$, are presumed to be continuous and can be controlled with negligible error. The response (y) is postulated to be a random variable. The individual variables (x_1, $x_2 \ldots x_k$) and the response (y) can be related as follows:

$$y = \mathrm{f}(x_1, x_2 \ldots x_k) + \mathrm{e} \tag{1}$$

Where y is the response of the system, f is the unknown response function, $x_1, x_2 \ldots x_k$ are the independent variables, k is the number of independent variables, and e is the statistical error. The set of values of independent variables where no further increase in response is observed is known as the optimal region. In most cases, a second-order response surface model is used which can be given as:[13]

$$y_0 = \beta_0 \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{k-1} \sum_{j=2}^{k} \beta_{ij} x_i x_j + \varepsilon \tag{2}$$

Where x_i, x_j are coded independent variables, β_0, β_i, β_ii, β_ij (i = 0,1, 2… k; j = 0,1, 2… k) are the regression coefficients for the intercept and linear, quadratic and interaction terms, respectively, and ε is the statistical error. In the present study, the RSM has been used to determine the relation between mass transfer coefficient and operating parameters such as superficial gas velocity, superficial liquid velocity and particle size. The dimensional coded variables x_1, x_2, x_3, and x_4 vary between – 1 and +1, while the variables are designated as: –1, 0, and +1. The mathematical representation of the response Y and the variables is given as

$$\begin{aligned} Y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_3 x_3 + \beta_4 x_4 + \beta_{11} x_1^2 + \\ + \beta_{22} x_2^2 + \beta_{33} x_3^2 + \beta_{44} x_4^2 + \beta_{12} x_1 x_2 + \\ + \beta_{13} x_1 x_3 + \beta_{14} x_1 x_4 + \beta_{23} x_2 x_3 + \beta_{24} x_2 x_4 \end{aligned} \tag{3}$$

and

$$\beta_{\mathrm{ij}\,=}\ 0,\ 1,\ 2,\ 3,\ 4,\ \ldots,\ k$$

Where β and k are regression coefficients and variables.

Box-Behnken method

In the present work, the Box-Behnken experimental design has been chosen to find the relationship between the response functions and variables.[14] The Box-Behnken design is a rotatable second-order design based on three-level incomplete-factorial designs. The special arrangement of the Box-Behnken design levels allows the number of design points to increase at the same rate as the number of polynomial coefficients. Box-Behnken designs are experimental designs for response surface methodology, devised by George E. P. Box and Donald Behnken in 1960, to achieve the following goals: (i) Each factor, or independent variable, is placed at one of three equally spaced values, (ii) the design should be sufficient to fit a quadratic model, that is, one containing squared terms and products of two factors, (iii) the ratio of the number of experimental points to the number of coefficients in the quadratic model should be reasonable, (iv) the estimation variance should more or less depend only on the distance from the centre. This is achieved exactly for the designs with 4 and 7 factors, and should not vary too much inside the smallest (hyper)cube containing the experimental points.

Each design can be perceived as a combination of a two-level (full or fractional) factorial design with an incomplete block design. In each block, a certain number of factors are put through all combinations for the factorial design, while the other factors are kept at the central values. Most of the designs can be split into groups (blocks), for each of which the model will have a different constant term, in such a way that the block constants will be uncorrelated with the other coefficients. In conventional experimentation, the experiments were conducted keeping all the variables constant except the parameters whose influence is being studied. This type of experiment reveals the effect of the chosen parameters under set conditions, assuming that variables were independent and that effect will be same at other values of the remaining variables.

Experimental setup and procedure

A schematic diagram of experimental setup is shown in Fig. 1. A vertical Perspex column of 5.4 cm inner diameter and 6 cm outer diameter, and height of 160 cm was used for mass transfer studies in three-phase fluidization. The column consisted of three sections: 1. gas-liquid distributor section, 2. test section, 3. gas-liquid disengagement section. Test section was the main component of fluidizer, where fluidization took place.

The entrained particles were retained on the mesh attached to the outlet pipe of the column. The gas-liquid distributor was located at the bottom of the test section, and it was designed in such a manner that uniformly distributed liquid and gas mixture entered the test section. The circular gas distributor section made of copper was provided with four protrusions which was 2 cm inner diameter. The liquid inlet pipe was 2.5 cm inner diameter located centrally at the lower end of the test section. This was done in order to have less pressure drop at gas distributor and uniform flow of fluids. The outlet of the test section was at a height of 150 cm in the column and pressure tapings were provided at the walls of the column connected to mercury manometer for pressure drop measurement.

The three phases involved in G-L-S fluidization were air, water and gypsum. The solid particles are supported on a perforated stainless steel mesh containing 300 spaced holes of 0.5 mm each. The

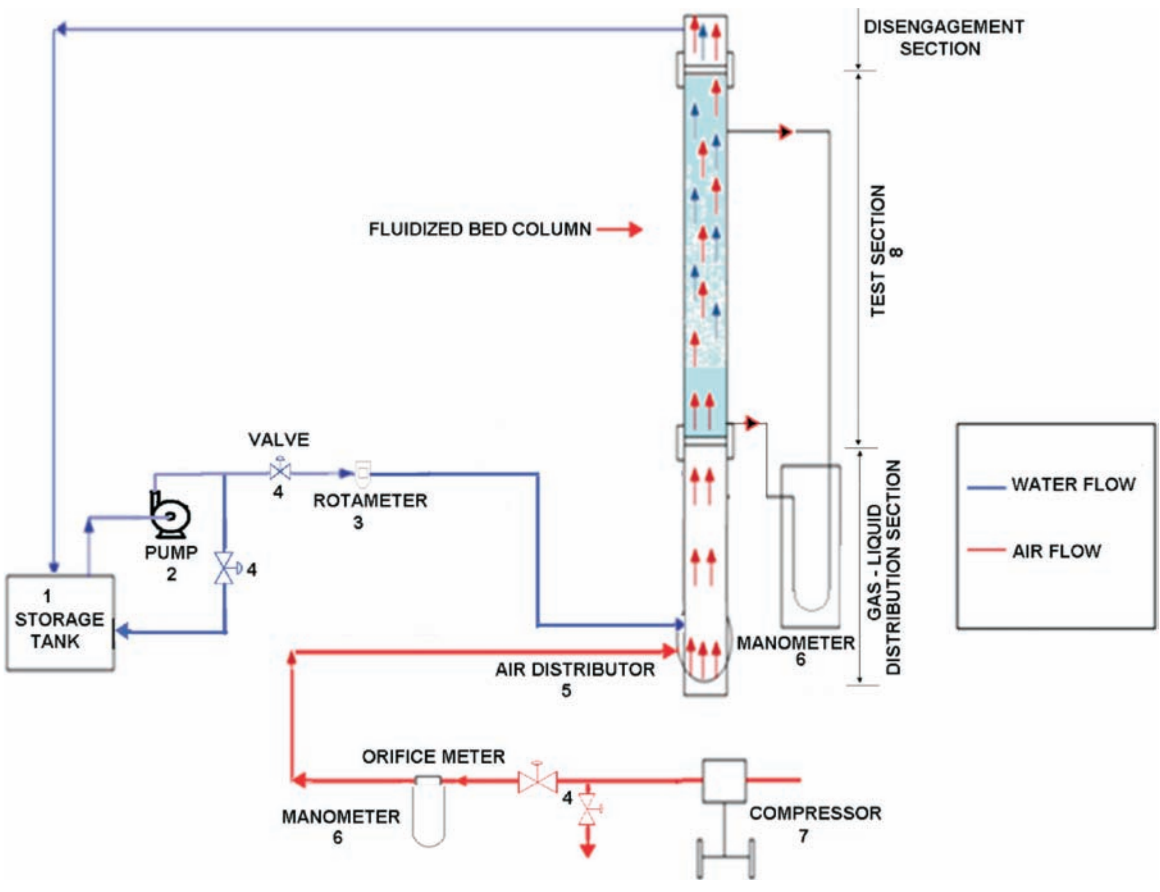


Fig. 1 – *Experimental setup schematic diagram: 1 – Storage tank, 2 – Pump, 3 – Rotameter, 4 – Valves, 5 – Air distributor, 6 – Manometer, 7 – Compressor, 8 – Test section*

particles of different sizes of gypsum of density 2.14, 2.24, 2.19 g cm^{-3} were used as solid phase, fresh water was used as liquid phase with the superficial liquid velocity varied as 2.42, 3.63, 4.85, 6.06 and 7.2 cm s^{-1} and air was used as gaseous phase with the superficial gas velocity varying from 0.2 to 2.2 cm s^{-1}. The experiment was carried out for various superficial gas velocities corresponding to different constant superficial liquid velocities. Mixing section and grid zone ensured that gas and liquid were well mixed into the bed. Liquid from a storage tank was pumped by a centrifugal pump and its flow rate was measured by a calibrated rotameter, and the gas flow rate was measured by an orifice meter. A set of control valves were provided for regulating both liquid and gas flow rates. Gypsum of various sizes was used as bed material in co-current three-phase fluidization. Fresh water was allowed to pass through the bed and the flow rate was adjusted by control valve. Gas was used as mass transfer aid. Liquid flow rate, manometer readings and bed height were noted. After space time elapsed three times, a sample was collected from the top of the fluidized bed and analyzed for solid concentration by volumetric titration method. The same procedure was repeated for different sized particles. Mass transfer coefficient, Sherwood number and bed porosity were calculated for different superficial gas velocities. In three-phase fluidization, gas flow started after the bed had been expanded by liquid flow to about 10 % initial bed height. This was to avoid slug formation in bed when gas passed through the compact bed of settled solids. Stabilization of bed was measured by pressure difference in manometer since bed interface was not visually defined.

Analysis of variance (ANOVA)

ANOVA is a statistical technique that subdivides the total variation in a set of data into component parts associated with specific sources of variation of the purpose of testing hypotheses on the parameters of the model. The analysis of variance (ANOVA) on the statistical significance of the ratio of mean square variation due to regression and mean square residual error was performed where m is the total number of the experiments, and η_i is the S/N ratio at the i^{th} *test.* The sum of squares form the tested factors, SS_p, can be calculated as:

$$SS_{\mathrm{p}} = \sum_{j=1}^{t} \frac{(S_{n_j})}{t} - \frac{1}{m}\left(\sum_{i=1}^{m} n_i\right) \qquad (4)$$

Where p represents one of the tested factors, j the level number of this specific factor p, t the repe-

Table 1 – *Actual design of experiments and responses for mass transfer studies in co-current three-phase fluidization*

Run	A	B	C	Bed porosity	Gas holdup	Sherwood number	Mass transfer coefficient
1	0.41	2.42	2	0.6692	0.4156	64.22	0.0053
2	0.41	7.28	2	0.7765	0.6053	168.69	0.012
3	2.48	2.42	2	0.8476	0.7308	118.57	0.0077
4	2.48	7.28	2	0.8874	0.8012	245.77	0.0153
5	1.45	2.42	1	0.7614	0.5545	37.1	0.00534
6	1.45	7.28	1	0.8642	0.7465	98.09	0.01245
7	1.45	2.42	3	0.7976	0.6428	187.51	0.0077
8	1.45	7.28	3	0.8527	0.7399	430.87	0.0166
9	0.41	4.85	1	0.7746	0.5791	35.39	0.005
10	0.28	4.85	1	0.8041	0.6342	47.71	0.0065
11	0.41	4.85	3	0.6307	0.3481	124	0.00644
12	2.48	4.85	3	0.8759	0.7809	271.31	0.01015
13	1.45	4.85	2	0.8075	0.66	98.05	0.0067
14	1.45	4.85	2	0.8075	0.66	98.05	0.0067
15	1.45	4.85	2	0.8075	0.66	98.05	0.0067
16	1.45	4.85	2	0.8075	0.66	98.05	0.0067
17	1.45	4.85	2	0.8075	0.66	98.05	0.0067

tition of each level of the factor *p,* and S_{nj} the sum of the S/N ratio involving this factor and level *j.*

Degree of freedom (D). D denotes the number of independent variables. The degree of freedom for each factor (D_p) is the number of its levels minus one. The total degrees of freedom (D_r) are total number of the result data points minus one, i.e., the total number of trials multiplied by the number of repetition minus one. The degree of freedom for error (D_e) is the number of the total degrees of freedom minus the total degree of freedom for each factor.

Variance (V). Variance is defined as the sum of squares of each trial sum result involving the factor, divided by the degrees of freedom of the factor:

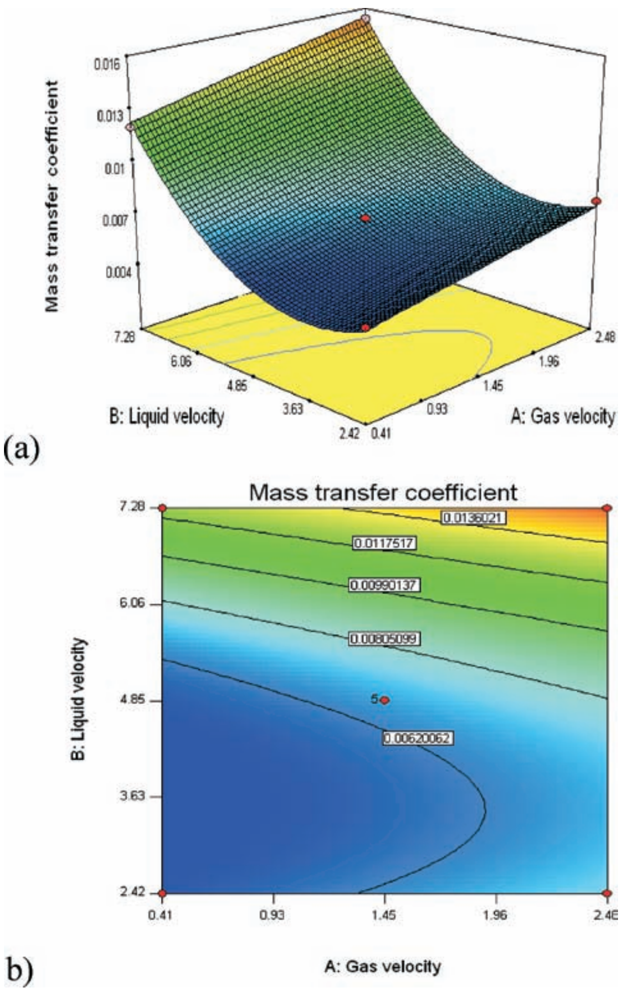
$$V_p\,(\%) = \frac{SS_p}{D_p} \cdot 100 \tag{5}$$

The corrected sum of squares (SS'_p), SS'_p is defined as the sum of squares of factors minus the error variance multiplied by the degree of freedom for each factor:

$$SS'_p = SS_p - D_p V_p \tag{6}$$

Percentage of the contribution to the total variation (P_p), P_p denotes the percentage of the total variance of each individual factor.

$$P_p\,(\%) = \frac{SS'_p}{D_p} \cdot 100 \tag{7}$$

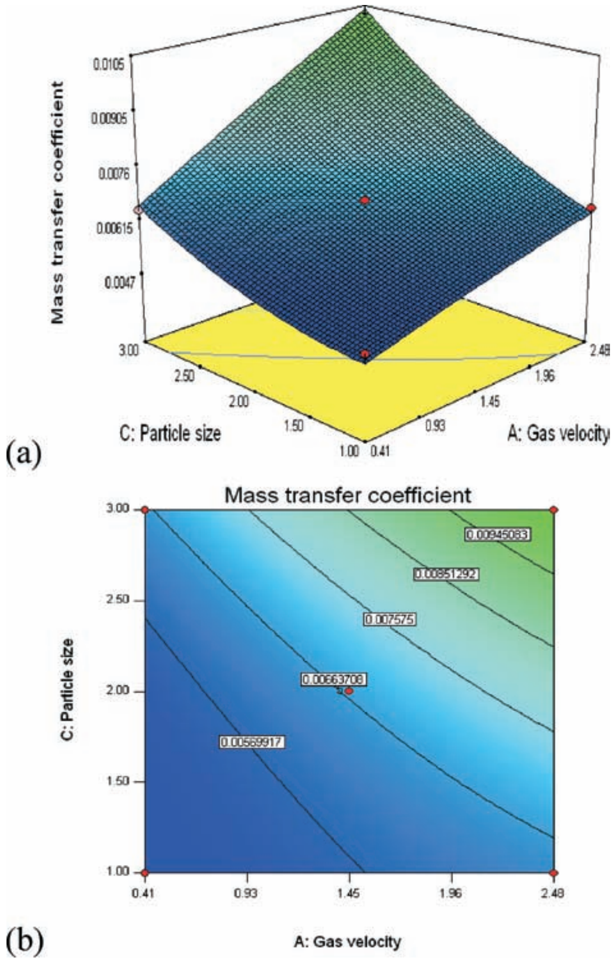


F i g . 2 – *The combined effects of gas and liquid velocity on mass transfer coefficient, (a) Response surface, (b) contour plot, A – 0.2 cm* s^{-1}; *B – 2.42 cm* s^{-1}

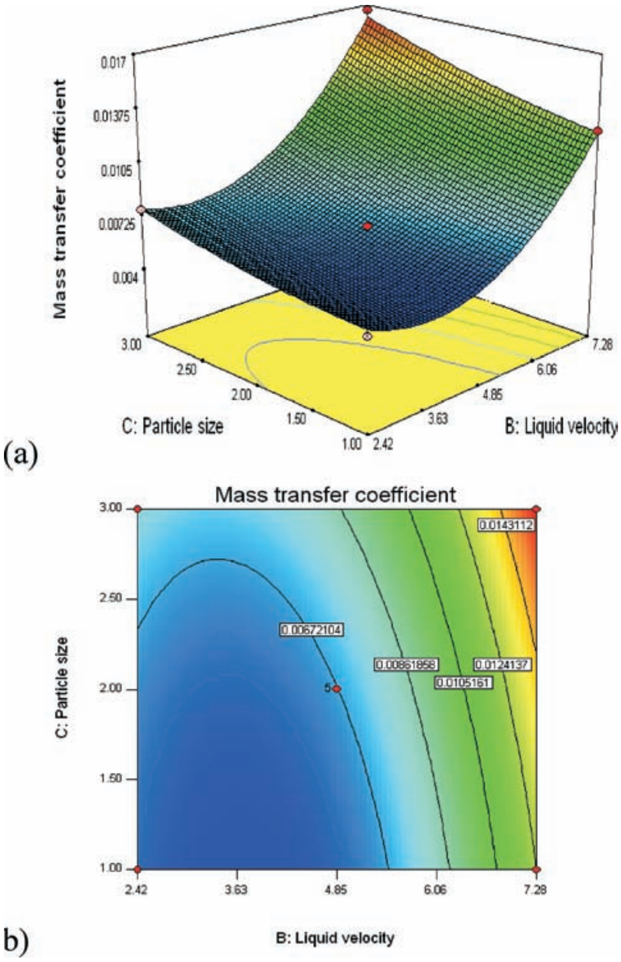
Results and discussion

The response surface methodology was applied to mass transfer studies in three-phase fluidization and the results were presented in both surface and contour plots. This study was carried out to check the influence of various operating parameters on mass transfer and the optimization was determined based on the influence of individual parameters. The effects of variables on the mass transfer from gypsum are given in Figs. 2 to 4. The interaction between varying gas and liquid velocity along with particle diameter is given in 3-dimensional surface plots (Fig. 2(a)) and the contour plot (Fig. 2(b)). It can be ascertained from the surface plot that Sherwood number is a function of mass transfer coefficient which increases with increase in superificial gas and liquid velocity along with increase in particle size. Increase in superficial gas velocity speeds up the rising velocity of bubbles in the bed and also enhances the turbulence of liquid phase in higher particle size (Fig. 3(a) and 3(b)) which promotes the Sherwood number and mass transfer coefficient. The mass transfer coefficient depends on the extent of mixing. The mixing was high when the gas velocity increased. Mass transfer coefficient increases with increase in superficial gas and liquid velocity. The increase in particle size with increase in superficial liquid velocity (Fig. 4(a) and 4(b)) increases the mass transfer coefficient in the bed more sharply and it may be due to increase in the mixing in the bed leading to transfer of calcium sulfate from gypsum present inside the fluidized bed column. Bed porosity increases with increase in superficial gas and liquid velocity. It can be observed that the bed porosity decreases with increase in particle size. The gas holdup increases with increase in superficial liquid velocity and gas velocity whereas it slightly decreases with increase in particle size.

The mathematical relationship between mass transfer coefficient and variables such as superficial liquid velocity, superficial gas velocity and particle size was determined as



F i g . 3 – *The combined effects of gas velocity and particle size on mass transfer coefficient, (a) Response surface, (b) contour plot, A – 0.2 cm s^{-1} C – 0.0842 cm*



F i g . 4 – *The combined effects of liquid velocity and particle size on mass transfer coefficient, (a) Response surface, (b) contour plot, B – 2.42 cm s^{-1}; C – 0.0842 cm*

$$\textbf{Mass transfer coefficient} = 6.700\text{E-}003 + 1.364\text{E-}003 \cdot A + 3.789\text{E-}003 \cdot B + 1.450\text{E-}003 \cdot C + 2.250\text{E-}004 \cdot A \cdot B + 5.525\text{E-}004 \cdot A \cdot C + 0.00045 \cdot B \cdot C - 6.250\text{E-}005 \cdot A^2 + 0.00344 \cdot B^2 + 0.000385 \cdot C^2 \quad (8)$$

The prediction of mass transfer coefficient using the above equation has been compared with the experimental values given in Table 1 and Fig. 5. It can be ascertained from the figure that the model equation predictions adequately match the experimental values within 5 % error. Similarly, the bed porosity, gas holdup, and Sherwood number is related to the variables such as superficial gas velocity [A], superficial liquid velocity [B] and particle size [C] was determined as

$$\textbf{Gas holdup} = 0.66 + 0.12 \cdot A + 0.069 \cdot B - 3.250\text{E-}004 \cdot C - 0.030 \cdot A \cdot B + 0.094 \cdot A \cdot C - 0.024 \cdot B \cdot C - 0.054 \cdot A^2 + 0.032 \cdot B^2 - 0.021 \cdot C^2 \quad (9)$$

$$\textbf{Sherwood number} = 98.05 + 36.38 \cdot A + 67.00 \cdot B + 99.43 \cdot C + 5.68 \cdot A \cdot B + 33.75 \cdot A \cdot C + 45.59 \cdot B \cdot C - 8.76 \cdot A^2 + 60.03 \cdot B^2 + 30.32 \cdot C^2 \quad (10)$$

$$\textbf{Bed porosity} = 0.81 + 0.071 \cdot A + 0.038 \cdot B - 5.925\text{E-}003 \cdot C - 0.017 \cdot A \cdot B + 0.054 \cdot A \cdot C - 0.012 \cdot B \cdot C - 0.030 \cdot A^2 + 0.018 \cdot B^2 - 0.00618\ C^2. \quad (11)$$

The predicted gas holdup, Sherwood number, mass transfer coefficient using the above three equations 9–11 was compared with experimental values given in Table 1 and Fig. 6. It can be noticed from the figure that the equation predictions adequately match the experimental values within 5 % error. The parameters in eqs. 8–11 are optimized for maximum mass transfer coefficient and Sherwood number and the optimized values are given in Table 2. It can be noticed from Table 2 that the optimum values of parameters correspond to maximum mass transfer coefficient of 0.1796.

Table 2 – *Optimized parameters of higher mass transfer coefficient and Sherwood number*

Factor	A	B	C	Sherwood number	Mass transfer coefficient/cm s^{-1}
Parameter	Superficial gas velocity/cm s^{-1}	Superficial liquid velocity/cm s^{-1}	Particle size/cm	487.7503	0.017962077
Optimized value	2.48	7.27	2.818	487.7503	0.017962077

Table 3 – *Estimated regression coefficient and corresponding t and p values for mass transfer coefficient*

Factor	Coefficient	Standard error	*t*	*p*
Intercept	0.0067	0.000136	0.006378	0.007022
A	0.001364	0.000108	0.001109	0.001618
B	0.003789	0.000108	0.003534	0.004043
C	0.00145	0.000108	0.001196	0.001704
AB	0.000225	0.000152	–0.00013	0.000585
AC	0.000553	0.000152	0.000193	0.000912
BC	0.000448	0.000152	8.79E-05	0.000807
A^2	–6.2E-05	0.000148	–0.00041	0.000288
B^2	0.003438	0.000148	0.003087	0.003788
C^2	0.000385	0.000148	3.45E-05	0.000736

Table 4 – *Estimated regression coefficient and corresponding t and p values for Sherwood number*

Factor	Coefficient	Standard error	*t*	*p*
Intercept	98.05	11.23003	71.49521	124.6048
A	36.3825	8.878116	15.38909	57.37591
B	67.0025	8.878116	46.00909	87.99591
C	99.425	8.878116	78.43159	120.4184
AB	5.6825	12.55555	–24.0067	35.37166
AC	33.7475	12.55555	4.058338	63.43666
BC	45.5925	12.55555	15.90334	75.28166
A^2	–8.76375	12.23764	–37.7012	20.17367
B^2	60.02625	12.23764	31.08883	88.96367
C^2	30.31625	12.23764	1.378834	59.25367

The significance of the regression coefficients were analysed using the *p* test and *t* test. The *p* values were used to check the consequences of interactions among the variables and in turn indicated the patterns of the interactions among the variables. In general, the larger the magnitude of the *t* value and smaller the *p* value, the greater is the significance of the corresponding coefficient term. It can be noticed from Table 3 that the coefficients for the linear effect of superficial gas velocity, superficial liquid velocity and particle size are significant when compared to the other linear effect. Whereas, the coefficients in the quadratic term for both gas and liquid velocity are significant when compared to the coefficients in the quadratic term for particle size.

Finally the coefficients in the interaction terms for the superficial gas velocity and particle size are more significant than the other interactive terms (superficial gas-liquid velocity, superficial liquid velocity-particle size). Similar analyses performed for the Sherwood number are presented in Table 4. An analysis of variance to determine the significant

Table 5 – *ANOVA results – Sherwood number*

Source	Degree of freedom	Sum of squares	Mean square	*F*- value	*P*
Regression	9	1.585 E005	1.761 E004	27.94	0.0001
Linear	3	1.256 E005	1.256 E005	199.17	0.004
Square	3	1.936 E004	1.936 E004	30.71	0.5412
Interaction	3	1.299 E004	1.299 E004	20.69	0.7041
Residual error	7	4.414 E003	630.6		
Lack of fit	3	4.414 E003	1.471 E003		
Pure error	4	0	0		
Total	16	1.630 E+005			

$R^2 = 0.9729$, $R_{adj}{}^2 = 0.938$

Table 6 – *ANOVA results- mass transfer coefficient*

Source	Degree of freedom	Sum of squares	Mean square	*F*- value	*P*
Regression	9	2.0 E-004	2.222 E-005	2.40 E002	0.0000
Linear	3	0.0424	0.0424	1.583 E003	0.0000
Square	3	0.0398	0.0398	5.44 E002	0.7215
Interaction	3	0.0122	0.0122	24.05	0.2125
Residual error	7	6.476 E-007	9.251 E-008		
Lack of fit	3	6.476 E-007	2.159 E-007		
Pure error	4	0	0		
Total	16	2.006 E-004			

$R^2 = 0.9968$, $R_{adj}{}^2 = 0.9926$

effects of process variables was conducted and the results presented in Tables 5 and 6. It can be noticed from Tables 5 and 6 Sherwood and mass transfer coefficient output responses, that the F-statistics values regressions are higher.

The large F values indicate that most of the variation in the response can be explained by the regression model equation. The associated P value is used to estimate whether the F statistics are large enough to indicate the statistical significance. The lower p value indicates that the model is considered to be statistically significant. The model adequacies were checked by $\boldsymbol{R}^2$ and adj- $\boldsymbol{R}^2$. A higher value of $\boldsymbol{R}^2$ shows that the model can explain the response successfully. The model adequacy has also been verified by the adjacent- $\boldsymbol{R}^2$ value. The ANOVA indicates that the second-order polynomial model (see eqs. 8, 9, 10 and 11) is significant and adequate to represent the actual relationship between the response and the (transfer efficiency) variables, with a small p value (0.0000) and a high value of R^2(0.9968) for bed porosity, gas holdup, Sherwood number and mass transfer coefficient with an R^2 value of (0.9830, 0.9839, 0.9968, 0.9729).

Conclusion

Experiments were carried out using gypsum as solid phase for mass transfer studies in three-phase fluidization. It was observed from the investigation that the mass transfer coefficient, Sherwood number, gas holdup, bed porosity were significantly influenced by particle size, superficial gas and liquid velocity. The experimental data was analysed using response surface methodology and the individual and combined parameter effects on mass transfer coefficient and Sherwood number were analysed. The 3-level, 3-factor Box-Behnken method was applied. Regression equations were developed for all responses.

It was proved that the model predictions of mass transfer coefficient, Sherwood number, gas holdup and bed porosity were in good agreement with the experimental observations. Further, the parameters were optimized for effective mass transfer from gypsum using the Response Surface Method. The optimized values for maximum mass transfer coefficient through three-phase fluidization were: particle size-0.2818 cm, superficial gas velocity –

2.48 cm s^{-1} and superficial liquid velocity – 7.28 cm s^{-1} so that the value of mass transfer coefficient – 0.017962077, Sherwood number – 487.7503, gas holdup – 0.805453, bed porosity – 0.88979.

Symbols

A – superficial gas velocity, cm s^{-1}
B – superficial liquid velocity, cm s^{-1}
C – particle size, cm
β_0 – intercept, –
β_i – linear effect, –
β_{ii} – squared effect, –
β_{ij} – interaction effect, –
Sh – Sherwood number
x_1 x_2 – variables, –
y – response, –

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