



Development of a model for the prediction of hydrodynamics of a liquid–solid circulating fluidized beds: A full factorial design approach

Ritesh Ramesh Palkar, Vidyasagar Shilapuram *

Department of Chemical Engineering, National Institute of Technology, Warangal, Telangana 506 004, India

ARTICLE INFO

Article history:

Received 8 December 2014

Received in revised form 17 March 2015

Accepted 20 April 2015

Available online 28 April 2015

Keywords:

Circulating fluidized bed

Statistical modeling

Design of experiments

Hydrodynamic behavior

Response

ABSTRACT

Hydrodynamics of a liquid–solid circulating fluidized bed (LSCFB) system has a significant impact on the reactor design. In the present study, statistical design approach was adopted to model the hydrodynamic behavior of an LSCFB in terms of average solids holdup and solids circulation rate. Primary liquid velocity, auxiliary liquid velocity, solids inventory and liquid viscosity are the input variables, called factors, which affects the system. Average solids holdup and solids circulation rate are called responses of the system. A full factorial design approach with four factors and three levels of the factors were considered. Various models such as linear, two factor interaction, quadratic, and cubic models were tested for the adequacy. Within the range of experiments conducted, for both responses, the quadratic regression model is suggested. The model shows that some of the interaction effects between the factors are dominant. The developed model was verified by using various statistical tests. Also, the model was validated using various experimental data sets chosen at different conditions. Results based on 'R' value, and deviations in parity plot were falling within agreement level. This suggests that the proposed model can be adopted for various processing applications in the LSCFB unit.

© 2015 Elsevier B.V. All rights reserved.

1. Introduction

Fluidized beds are the processing equipment in which particles are fluidized against gravity by the fluid. In general fluid may be liquid or gas. Variation in the forces acting on the particles leads in the variation of fluid velocity which results in dynamic behavior. Circulating fluidized beds (CFBs) are one such fluidized bed reactors, which essentially consists of two columns in the same unit, namely the riser and the downcomer with solids circulating in between these two columns. These reactors have a wide application range wherever the process requires temperature uniformity, plug flow conditions, avoidance of mass transfer limitations, favoring of fast reactions, good heat transfer between the processing materials, and continuous regeneration of catalyst [1]. Gas–solid circulating fluidized bed (GSCFB) is used for product purification and energy optimisation studies [2]. In this, continuous capture of CO_2 was studied, where this equipment was used with sorbent adsorption and regeneration phenomenon [3]. GSCFB also has potential all-round applications in many processing industries for fluid catalytic cracking, Fischer–Tropsch synthesis, combustion, environmental remediation, heterogeneous catalytic applications, solids processing i.e. calcinations, reduction of iron ore and gold roasting. This is due to higher efficiency, operational flexibility and overall profitability as compared to other reactors [4]. Recently, LSCFBs are gaining importance in various applications. To name a few, LSCFBs are used to purify the

municipal wastewater by means of simultaneous removal of carbon, nitrogen and phosphorous [5]. LSCFBs are also used for many green technological aspects such as extractive fermentation of lactic acid and removal of cesium from radioactive liquid waste [6,7]. Various experimental studies by different research groups for different reactor geometries, mode of operation and operating conditions are available for the hydrodynamic studies in the liquid–solids and gas–liquid–solid circulating fluidized bed reactor systems [8–20]. Average solids holdup and solids circulation rate are the most important hydrodynamic properties which decide the reactor performance and separation efficiency as a contactor in the LSCFB. In literature, various empirical approaches, modeling, simulation and optimisation strategies were adopted by different researchers to predict the hydrodynamics [21–24]. Core-annulus flow structure model used for GSCFBs were extended to LSCFB [21]. Computational fluid dynamic models were tried to predict the flow of solid and liquid phases [22]. Mathematical model based on homogeneous fluidization was developed and extended to predict the performance of an LSCFB for ion-exchange system for protein recovery [23]. Same group carried out multi-objective optimisation using the genetic algorithm approach and verified with experimentally obtained data for continuous protein recovery [24]. A correlation for liquid phase axial dispersion coefficient has been reported for liquid–solid circulating multistage fluidized bed in which liquid phase residence time distribution were studied along with CFD validation [25]. Recently an empirical correlation was developed from the exhaustive experimental data by considering various factors [19]. In addition, various empirical correlations are available for the average solids holdup and

* Corresponding author. Tel.: +91 83329 69463.

E-mail address: vidyasagars@nitw.ac.in (V. Shilapuram).

solids circulation rate with their respective applicability range [26,27]. Most of the approaches reported, as discussed above either show significant deviation or applicable within the specific experimental range studied.

Design of experiments (DOE) is one of the powerful data mining tools. It is considered as a black box of simulation with potential applications in various research areas. It can be implemented to get transparency in the developed model behavior with less time and lower cost for processing [28]. Conventionally, experiments give the output or response value for a given fixed set of input factors that are influencing the system. Upon comparison of responses by varying one particular input keeping the rest of the input factors constant gives an idea about the effect of that particular input factor on the response. However, in reality, combination of simultaneous variation in input factors may result in different responses because of interactions among the input factors. From the observed results, it is difficult to notice the effect of interactions among the input factors that are reflected in the observed experimental response. This cannot be visualized in conventional experiments. Therefore, one of the significant advantages of DOE is to come up with a model which gives the effect of each individual factor as alone as well as a combination with the other factor on the response. This model development also gives an idea for more accurate and realistic model.

So far the DOE technique has not been used very extensively in the field of process engineering. Abbas and Baker studied the influence of operating parameters, i.e., catalyst weight, decomposition temperature and methane partial pressure on rate of methane decomposition [29]. Jena et al. provided a statistical analysis for a gas–liquid–solid system for estimation of gas holdup and liquid holdup based on the factorial design [30]. Hydrodynamic characteristics of three-phase fluidization of a homogeneous ternary mixture of particles using factorial design has been carried out to study the effect of various operating parameters like superficial liquid velocity, liquid velocity, initial static bed height, average particle size and column diameter [31]. A detailed study based on the full factorial design has been carried out by Al-Hassani et al. in which various parameters such as temperature of the reaction, reaction relative time and types of the catalyst used are taken as the operating variables which are used to build a statistical model for methane decomposition rate [32].

In the previous work, experimental results showed that the observed responses (average solids holdup and solids circulation rate) were neither monotonically increasing nor decreasing nor constant for the systematic increase in all the input factors that are affecting the system [16–19]. This reflects that there is a relation between the input variables of the response. Further details related to the experimental setup, experimental procedure and physical properties and range of liquid velocities are described elsewhere [16–19]. In the present study, a full factorial approach is helpful to determine these effects efficiently and develop a model which can be extended for a particular application to develop an efficient process [33]. Therefore, an attempt has been made to adopt the DOE approach for modeling the chosen LSCFB system. As per the best of the authors' knowledge this type of study has not been reported in the literature for modeling the LSCFBs.

2. Experimental design – statistical method

In order to evaluate the hydrodynamics of an LSCFB, factorial design approach was used. Factorial design method is one of the more efficient statistical techniques in regard of time, resources and sample size. In addition to this, where the interaction plays a dominant role; this methodology is a key factor to get insight over a range of experimental data. Factorial design method facilitates the relationship between various operating parameters, where influence of the independent variables is collectively expressed in terms of various mathematical and statistical techniques, which ultimately leads to the analysis of the degree of influence on the dependent variables [34].

The factorial design approach was used to determine the interaction effects of all independent variables affecting the response of the LSCFB. Four different independent variables (termed as factors) that are responsible for the hydrodynamic behavior/variation of the LSCFB are primary liquid velocity (U_{L1}) in m/s, auxiliary liquid velocity (U_{L2}) in m/s, solids inventory (L_0) in m and liquid viscosity (μ_L) in Pa·s. Typically there are two ways of presenting the solids inventory. One is in terms of the height and the other in terms of kgs of solids in the downcomer before start-up of operation. Both of these are inter-convertible. In the present method, similar to the reported literature solids inventory is presented in terms of height of the packed bed height in the downcomer in the cylindrical portion [11,26]. The average solids holdup (ε_S) and solids circulation rate (G_S) are the responses that depict the hydrodynamic behavior in the LSCFB. Therefore, in this study, effects of all four operating variables (factors) on the two dependent variables (responses) were studied simultaneously. As the number of factors was less (i.e. four), a full factorial design was adopted with four factors and three levels of these factors. Three levels of factors were chosen, as the experimental results showed that the observed responses were neither monotonically increasing nor decreasing nor constant for the systematic increase in all the input factors.

The list of independent variables with their actual and coded values used in the factorial design is shown in Table 1. The coded values are indicated by –1, 0 and +1. ‘–1’ indicates the lowest level, ‘0’ indicates center point level and ‘+1’ indicates the highest level of variables used for the modeling. This statistical modeling study consisted of the 3^4 number of experiments, where base value (3) represents the number of levels and exponent value (4) indicates the number of factors used in the modeling study. Only part of design matrix along with the experimental observations and simulated results are presented in Table 2. The percentage deviation of the simulated observations with respect to experimental is also reported to verify the developed model mathematically.

Design Expert® software, version 9.0.2, was used to determine the suitable regression from the design matrix created for chosen set of experimental data. In the subsequent step model is verified by using analysis of variance (ANOVA) which helps to understand effect of factors and its interactions. The criteria used for choosing the best suitable regression model is based on the higher order polynomial where maximum interacting terms are significant and model is not aliased. In this modeling work, several models like linear, two-factor interaction, quadratic and cubic polynomial were analyzed in order to fit the experimental data and simultaneously to test the best possible outcome in terms of the predicted response from the derived model. The summary of various models used for predicting average solids holdup and solids circulation rate are reported in Table 3. This table suggests that the quadratic model can be used for developing a regression model to predict the both average solids holdup and solids circulation rate as the F-test > p-test and p < 0.05.

3. Development of regression models

The regression model is used when more than two variables are significant or responsible for change in the response of the system.

Table 1

List of independent variables used in the factorial design.

Variables	Actual value (coded value)		
Primary liquid velocity (U_{L1}), m/s	0.149 (–1)	0.163 (0)	0.189 (+1)
Auxiliary liquid velocity (U_{L2}), m/s	0.03 (–1)	0.052 (0)	0.08 (+1)
Solids inventory (L_0), m	0.15 (–1)	0.25 (0)	0.35 (+1)
Liquid viscosity (μ_L), Pa·s	9.03×10^{-4} (–1)	1.55×10^{-3} (0)	3.22×10^{-3} (+1)

Table 2

Design matrix along with experimental and predicted responses.

Factors				Response – 1			Response – 2		
Variables				Average solids holdup (ε_s)			Solids circulation rate (G_s), kg/m ² s		
U_{L1} , m/s	U_{L2} , m/s	L_0 , m	μ_L ($\times 10^{-3}$) Pa·s	Experimental values	Predicted	% deviation	Experimental values	Predicted	% deviation
0.149	0.03	0.35	3.22	0.069	0.07	-1.45	14.214	14.27	-0.39
0.149	0.052	0.35	0.0903	0.102	0.1	1.96	8.783	9.99	-13.74
0.149	0.052	0.35	3.22	0.084	0.089	-5.95	23.326	22.38	4.06
0.149	0.08	0.35	1.55	0.126	0.13	-3.17	50.753	41.62	17.99
0.149	0.08	0.15	3.22	0.067	0.072	-7.46	19.26	19.36	-0.52
0.149	0.052	0.35	1.55	0.122	0.12	1.64	26.252	24.05	8.39
0.149	0.08	0.15	1.55	0.078	0.092	-17.95	22.601	25.73	-13.84
0.149	0.08	0.35	0.0903	0.105	0.11	-4.76	22.69	27.62	-21.73
0.149	0.08	0.25	3.22	0.127	0.095	25.20	41.093	31.29	23.86
0.149	0.03	0.25	1.55	0.089	0.09	-1.12	20.062	15.48	22.84
0.163	0.03	0.15	3.22	0.043	0.031	27.91	7.964	6.66	16.37
0.163	0.03	0.35	3.22	0.065	0.064	1.54	15.991	14.96	6.45
0.163	0.08	0.15	0.0903	0.077	0.065	15.58	16.717	15.28	8.60
0.163	0.052	0.35	0.0903	0.089	0.094	-5.62	10.731	11.46	-6.79
0.163	0.08	0.15	3.22	0.061	0.066	-8.20	19.229	20.34	-5.78
0.163	0.052	0.25	3.22	0.08	0.077	3.75	13.921	18.03	-29.52
0.163	0.052	0.35	3.22	0.077	0.083	-7.79	26.887	23.06	14.23
0.163	0.052	0.15	3.22	0.058	0.052	10.34	14.323	9.56	33.25
0.163	0.03	0.35	1.55	0.094	0.093	1.06	12.814	17.07	-33.21
0.163	0.08	0.35	1.55	0.122	0.12	1.64	48.215	42.84	11.15
0.189	0.03	0.35	3.22	0.054	0.061	-12.96	17.141	13.3	22.41
0.189	0.08	0.35	1.55	0.114	0.11	3.51	51.475	42.17	18.08
0.189	0.052	0.35	0.0903	0.082	0.084	-2.44	13.652	11.24	17.67
0.189	0.03	0.25	1.55	0.089	0.075	15.73	20.062	16.62	17.16
0.189	0.08	0.25	1.55	0.118	0.1	15.25	50.932	36.42	28.49
0.189	0.08	0.35	3.22	0.093	0.09	3.23	38.338	38.71	-0.97
0.189	0.08	0.35	0.0903	0.088	0.092	-4.55	26.062	28.81	-10.54
0.189	0.08	0.15	0.0903	0.068	0.056	17.65	17.664	15.63	11.51
0.189	0.08	0.25	3.22	0.088	0.087	1.14	25.803	30.68	-18.90
0.189	0.052	0.35	3.22	0.068	0.079	-16.18	25.058	21.36	14.76

ANOVA analysis is performed for the verification of the suggested regression model to establish the correlation between the experimental and predicted results. These ANOVA results are presented in terms of various statistical parameters. If the statistical parameters evaluated are within satisfactory limits, model developed shows the good relationship between these factors for the response.

In general, the regression model is classified as first-order model, second-order model, etc. A general form of the regression model is as shown in Eq. (1).

$$y = \beta_0 + \sum_{i=1}^n \beta_i x_i + \sum_{i=1}^{n-1} \sum_{j=i+1}^n \beta_{ij} x_i x_j + \sum_{i=1}^n \beta_{ii} x_i^2 \quad (1)$$

where, y is the value of the response predicted by the model; β_0 , β_i , β_{ij} , and β_{ii} are the coefficients of the model estimated by the ANOVA; x_i and

Table 3
Model summary used for the fitting average solids holdup and solids circulation rate.

Model	F-test	p-Value	R ²	Comment
<i>1. Average solids holdup</i>				
Linear	34.15	<0.0001	0.6425	
2-factor interaction	0.41	0.8725	0.6545	
Quadratic	14.57	<0.0001	0.8165	Suggested
Cubic	2.16	0.0200	0.8914	Aliased
<i>2. Solids circulation rate</i>				
Linear	34.78	<0.0001	0.6467	
2-factor interaction	0.65	0.6885	0.6654	
Quadratic	26.01	<0.0001	0.8701	Suggested
Cubic	2.50	0.0069	0.9279	Aliased

x_j are the factors. For the present study x_1 , x_2 , x_3 and x_4 are respectively U_{L1} , U_{L2} , L_0 , and μ_L as presented in Table 1.

The minimum total liquid velocity in the riser at which there will be a circulation of solids from the riser to the downcomer and simultaneously recirculation of solids from downcomer to riser to establish continuous circulation between riser and downcomer is called critical transition to circulating fluidized bed regime (U_{cr}). The total liquid velocity in the riser is the sum of the primary and auxiliary liquid velocities. Therefore, for all the liquid velocities above U_{cr} , there always exists a liquid solid circulating fluidized bed. However, the value of U_{cr} (i.e. the combination of primary and auxiliary liquid velocity) depends on the fluid and particle properties chosen as well as structure of solids feeding system. The critical transitional liquid velocities for circulating fluidized bed regime are different for different viscosities of liquids [19]. Similarly, the final total liquid velocity (primary as well as auxiliary liquid velocity) to complete circulating fluidized bed regime or to reach unstable operating condition depends on the fluid and particle properties chosen as well as on the solids feeding system. Hence, operating ranges of primary and auxiliary liquid velocities for the circulating fluidization regime were different for different viscosities of liquids. Usually with low viscous liquids, high window of circulating fluidized bed (CFB) regime is expected in terms of primary and auxiliary liquid velocity. With increasing viscosity operating window under the circulating fluidized bed regime becomes narrower [19]. Therefore, common ranges of primary and auxiliary velocities under circulating fluidized bed regime are identified for various viscosities of fluidizing media as shown in Table 1. Lowest velocities of primary and auxiliary liquid velocities are assigned ‘-1’ as coded value whereas highest values of primary as well as auxiliary velocities are assigned ‘+1’ as coded value and center values were assigned ‘0’ as coded value. Experimental data considered for this study is from Vidyasagar et al. [16,18] and Shilapuram et al. [17,19].

The multiple linear regression models for developing the statistical relationship to predict hydrodynamic behavior of LSCFB in terms of coded factors are represented in Eqs. (2) and (3).

$$\begin{aligned} \varepsilon_S = & 0.10 - (6.652 \times 10^{-3} \times x_1) + (0.015 \times x_2) + (0.017 \times x_3) \\ & - (2.543 \times 10^{-3} \times x_4) - (1.823 \times 10^{-4} \times x_1 \times x_2) \\ & - (4.369 \times 10^{-4} \times x_1 \times x_3) \\ & + (2.414 \times 10^{-3} \times x_1 \times x_4) \\ & - (1.387 \times 10^{-3} \times x_2 \times x_3) \\ & + (1.681 \times 10^{-3} \times x_2 \times x_4) \\ & + (2.193 \times 10^{-3} \times x_3 \times x_4) + (3 \times 10^{-3} \times x_1^2) \\ & - (5.571 \times 10^{-3} \times x_2^2) - (9.648 \times 10^{-3} \times x_3^2) \\ & - (0.023 \times x_4^2) \end{aligned} \quad (2)$$

$$\begin{aligned} G_S = & 30.20 + (0.78 \times x_1) + (10.30 \times x_2) + (6.45 \times x_3) \\ & + (4.5 \times x_4) - (0.24 \times x_1 \times x_2) + (0.50 \times x_1 \times x_3) \\ & - (0.93 \times x_1 \times x_4) + (2.13 \times x_2 \times x_3) \\ & - (0.69 \times x_2 \times x_4) + (0.72 \times x_3 \times x_4) + (0.45 \times x_1^2) \\ & + (1.44 \times x_2^2) - (1.69 \times x_3^2) - (15.48 \times x_4^2) \end{aligned} \quad (3)$$

3.1. Verification of developed model

Various statistical parameters like correlation coefficient (R^2), adjusted R^2 , predicted R^2 , adequate precision etc. are employed to get the degree of model fitness. These performance measures adopted in this modeling work for both hydrodynamic properties are presented in Table 4. The correlation coefficient is used to determine quantitative relationship between the interacting variables to observe the resemblance of an observed and predicted response. Correlation coefficient was calculated from the ratio of the regression sum of squares to the total sum of squares. For developed model of average solids holdup (Eq. (2)) and solids circulation rate (Eq. (3)) estimated R^2 values are 0.8165 and 0.8701 respectively. These values are adequate for selection of the model. In addition, adjusted R^2 and predicted R^2 are also used to determine the suitability of the developed model. Adjusted R^2 is the correction of the R^2 based on the number of terms and the sample size. Adjusted R^2 values show good agreement for statistical model development if the difference between adjusted R^2 and predicted R^2 is less than 0.2 [34]. As observed in Table 4 the differences between adjusted and predicted are below 0.2. Hence, the quadratic model is suggested. Standard deviation and mean noticed in the case of the average solids holdup were very low. In the case of solids circulation rate the standard deviation and mean noticed were 5.11 and 17.85. Adequate precision is another statistical term used to compare the range of predicted values. This term relates the signal to noise ratio of the terms used. Values of this adequate precision are found to be 19.409 and 23.408 for the average solids holdup and solids circulation rate respectively. Generally, values above four indicate adequate model discrimination. Since the values observed in Table 4 are well above four, the model further confirms the adequacy and this is strongly supports the use of quadratic model.

3.2. Model term verification by ANOVA

Model verification was carried out by using ANOVA test. This is a method based on the Fisher test which considers the null hypothesis of no treatment effects. This is an important statistical tool which subdivides the total variation of a set of data into component parts associated with specific sources of variation. This test mainly used to determine the significance of model terms, effect of individual factors and its interaction effects, ultimately leads to the precision of the model developed. Fisher's test is used to determine the regression, standard error and significance of each term used in the regression analysis. The value of 'F' is calculated by dividing the mean square by the residual mean square. Along with F-test, 'p-value' also has significance in ANOVA test. This usually relates to the risk of falsely rejecting a given hypothesis. Generally p-value less than 0.05 is considered as significant, whereas values more than 0.1 have less significance in the model prediction. However, in some of the cases depending on the knowledge of the process the terms having a p-value from 0.05 to 0.1 were also considered as significant [34].

The significance of each term and various interaction terms are evaluated within 95% confidence interval. For the design matrix shown in Table 2, ANOVA results obtained are summarized for the average solids holdup in Table 5. Constant F value for the average solids holdup is 20.98. In addition, the p-value of the constant is having value less than 0.0001. The table also shows that model operating parameters such as primary liquid velocity, auxiliary liquid velocity, solids inventory and square effects of solids inventory and liquid viscosity are the most significant terms.

The same procedure as discussed above was followed for the regression model for solids circulation rate. These results are presented in Table 6. From the table it may be observed that constant F-value for developed model is 31.58 and p-value reported is less than 0.0001 showing significance of the developed model. In addition, auxiliary liquid velocity, solids inventory, liquid viscosity; interaction of auxiliary liquid velocity and solids inventory, and square effect terms of solids inventory and liquid viscosity have a significant effect which is clearly observed from this ANOVA test.

Percentage contributions by the terms involved in the model developed for the both average solids holdup and solids circulation rate are shown in Table 7. Upon comparison of Tables 5 and 6 with Table 7 it may be observed that terms significant in Tables 5 and 6 are showing its significant percentage contribution for the predicted response.

4. Interaction effect of factors

Effects of various factors with their interaction in the hydrodynamic variation are discussed in the following sections separately. Though an empirical correlation was developed recently which could help to determine the hydrodynamic properties but it was unable to convey interaction effects of the hydrodynamic variables [18]. To overcome this deficiency in the present modeling work a new approach along with a statistical design has been adopted.

4.1. Average solids holdup

The regression model developed for the current study in terms of actual factors is as follows:

$$\begin{aligned} \varepsilon_S = & 0.155 - (3.007 \times U_{L1}) + (1.647 \times U_{L2}) + (0.762 \times L_0) + (52.116 \times \mu_L) \\ & - (0.36459 \times U_{L1} \times U_{L2}) \\ & - (0.218 \times U_{L1} \times L_0) + (104.207 \times U_{L1} \times \mu_L) - (0.554 \times U_{L2} \times L_0) \\ & + (58.043 \times U_{L2} \times \mu_L) - (18.931 \times L_0 \times \mu_L) + (7.498 \times U_{L1}^2) \\ & - (8.912 \times U_{L2}^2) - (0.964 \times L_0^2) - (17070.548 \times \mu_L^2) \end{aligned} \quad (4)$$

Table 4

Statistical parameters obtained by ANOVA study for hydrodynamic properties.

Hydrodynamic property	SD	Mean	CV (%)	R^2	R^2_{adj}	R^2_{pred}	Adequate precision
ε_S	0.012	0.078	15.04	0.8165	0.776	0.7281	19.409
G_S	5.11	17.85	28.63	0.8701	0.8426	0.8080	23.408

Table 5

Statistical ANOVA data for average solids holdup model.

Term	SS	df	MS	F-value	p-Value	Comment
Constant	0.041	14	2.915E–003	20.98	<0.0001	Significant
U_{L1}	2.315E–003	1	2.315E–003	16.66	0.0001	Significant
U_{L2}	0.011	1	0.011	79.88	<0.0001	Significant
L_0	0.015	1	0.015	111.04	<0.0001	Significant
μ_L	3.438E–004	1	3.438E–003	2.47	0.1205	
$U_{L1} * U_{L2}$	1.238E–006	1	1.238E–006	8.91E–003	0.9251	
$U_{L1} * L_0$	7.078E–006	1	7.078E–006	0.051	0.8221	
$U_{L1} * \mu_L$	2.302E–004	1	2.302E–004	1.66	0.2025	
$U_{L2} * L_0$	6.956E–005	1	6.956E–005	0.50	0.4818	
$U_{L2} * \mu_L$	1.089E–004	1	1.089E–004	0.78	0.3793	
$L_0 * \mu_L$	1.844E–004	1	1.844E–004	1.33	0.2535	
$(U_{L1})^2$	1.302E–004	1	1.302E–004	0.94	0.3366	
$(U_{L2})^2$	5.400E–004	1	5.400E–004	3.89	0.0529	
$(L_0)^2$	1.676E–003	1	1.676E–003	12.06	0.0009	Significant
$(\mu_L)^2$	5.750E–003	1	5.750E–003	41.38	<0.0001	Significant
Residual	9.171E–003	66	1.390E–004			
Total	0.050	80				

In this modeling work, it can be clearly seen that, individual terms like auxiliary liquid velocity, solids inventory and liquid's viscosity; interaction terms like primary liquid velocity – liquid viscosity, auxiliary liquid velocity – liquid viscosity; and square effect term of primary liquid velocity have a synergistic effect which positively helps to increase the average solids holdup. Furthermore, the remaining terms like primary liquid velocity, liquid viscosity; interaction effect of primary liquid velocity–auxiliary liquid velocity, primary liquid velocity–solids inventory, auxiliary liquid velocity–solids inventory, solids inventory–liquid viscosity; and square effects of auxiliary liquid velocity have very less antagonistic effect.

4.1.1. Interaction effect of primary and auxiliary liquid velocity on average solids holdup

The combined effect of primary liquid velocity and auxiliary liquid velocity is studied in the developed model. 3-D surface plot for variation of velocities is shown in Fig. 1. This effect is observed at constant solids inventory in the downcomer and liquid viscosity. From this figure it can be observed that simultaneous increase in the primary and auxiliary liquid velocities tend to reduction in the average solids holdup. This is due to the increase in liquid velocity in the reactor exerts more drag on solid material in the riser section, therefore solids tends to go out from the top of the riser. If the individual effect on the constant auxiliary liquid velocity is considered, the average solids holdup decreases with increase in primary liquid velocity whereas at constant primary velocity increase in auxiliary velocity increases the average solids holdup. This firmly indicates the synergistic effect induced by the introduction of the auxiliary liquid velocity and antagonistic effect due to the presence

of the primary liquid velocity in LSCFB at constant solids inventory and liquid viscosity. Thus, within the circulation fluidized bed regime at lower primary liquid velocity and higher auxiliary liquid velocity this hydrodynamic property shows a peak point.

4.1.2. Interaction effect of primary liquid velocity and solids inventory on average solids holdup

In this study, solids inventory of 0.15 m, 0.25 m, and 0.35 m was considered. A combined simultaneous effect of primary liquid velocity and solids inventory at constant auxiliary liquid velocity and liquid viscosity is shown in Fig. 2. The observations in this case are similar to the combined effect of primary and auxiliary liquid velocity on average solids holdup. However, quantitatively it may be different. The predicted average solids holdup region as shown by the color code on the right side of the figure suggests that at lower primary liquid velocity and a higher range of solids quantity in the downcomer contributes to an increased average solids holdup. A confirmation of the positive effect laden by the solids inventory on the prediction of the average solids holdup can be observed from this figure.

4.1.3. Interaction effect of primary liquid velocity and liquid viscosity on average solids holdup

Fig. 3 shows the effect of primary liquid velocity and liquid viscosity on the average solids holdup. This study was carried out at constant auxiliary liquid velocity and solids inventory in the downcomer. This combined effect shows that, at a constant auxiliary liquid velocity and solids inventory, average solids holdup increases at lower value of primary liquid velocity (i.e. 0.15 m/s) and liquid viscosity (i.e. up to a viscosity range of approximately 0.002 Pa·s to 0.00225 Pa·s); afterward

Table 6

Statistical ANOVA data for solids circulation rate.

Term	SS	df	MS	F-value	p-Value	Remark
Constant	11,542.62	14	824.47	31.58	<0.0001	Significant
U_{L1}	31.86	1	31.86	1.22	0.2733	
U_{L2}	5483.77	1	5483.77	210.05	<0.0001	Significant
L_0	2142.25	1	2142.25	82.06	<0.0001	Significant
μ_L	1078.58	1	1078.58	41.31	<0.0001	Significant
$U_{L1} * U_{L2}$	2.23	1	2.23	0.085	0.7712	
$U_{L1} * L_0$	9.23	1	9.23	0.35	0.5541	
$U_{L1} * \mu_L$	34.52	1	34.52	1.32	0.2544	
$U_{L2} * L_0$	164.20	1	164.20	6.29	0.0146	Significant
$U_{L2} * \mu_L$	18.16	1	18.16	0.70	0.4073	
$L_0 * \mu_L$	19.66	1	19.66	0.75	0.3887	
$(U_{L1})^2$	2.90	1	2.90	0.11	0.7400	
$(U_{L2})^2$	35.95	1	35.95	1.38	0.2448	
$(L_0)^2$	51.36	1	51.36	1.97	0.1654	Significant
$(\mu_L)^2$	2625.74	1	2625.74	100.58	<0.0001	Significant
Residual	1723.06	66	26.11			
Total	13,265.68	80				

Table 7

Percentage contribution of terms used for statistical modeling.

Term	Average solids holdup	Solids circulation rate, kg/m ² s
	% contribution	
U_{L1}	6.20	0.27
U_{L2}	29.45	46.87
L_0	40.15	18.31
μ_L	0.92	9.22
$U_{L1} * U_{L2}$	0.00	0.02
$U_{L1} * L_0$	0.02	0.08
$U_{L1} * \mu_L$	0.62	0.30
$U_{L2} * L_0$	0.19	1.40
$U_{L2} * \mu_L$	0.29	0.16
$L_0 * \mu_L$	0.49	0.17
$(U_{L1})^2$	0.35	0.02
$(U_{L2})^2$	1.45	0.31
$(L_0)^2$	4.49	0.44
$(\mu_L)^2$	15.39	22.44

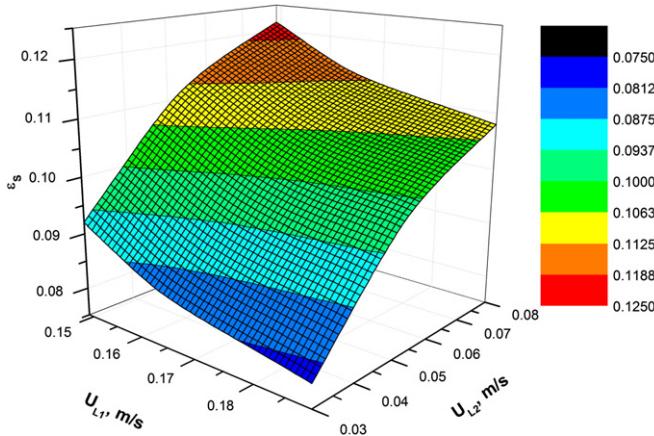


Fig. 1. Effect of primary liquid velocity and auxiliary liquid velocity on average solids holdup.

it decreases significantly. In the previous study, it was noticed that the average solids holdup was increased up to a certain liquid viscosity followed by a decrease in average solids holdup with further increase in viscosity. The value of viscosity for maximum solids holdup was dependent also on the value of the solids inventory in the downcomer [18]. The same phenomenon is also reflected in terms of interaction effects as shown in Fig. 3.

4.1.4. Interaction effect of auxiliary liquid velocity and solids inventory on average solids holdup

Fig. 4 shows the interaction effect of auxiliary liquid velocity and solids inventory at constant primary liquid velocity and liquid viscosity. From this figure, it is evident that the average solids holdup increases with an increase in the interaction of auxiliary liquid velocity and solids inventory in the downcomer of the LSCFB. It is also observed that, average solids holdup increases individually with an increase in auxiliary liquid velocity or solids inventory when the rest of the factors are held constant.

4.1.5. Interaction effect of auxiliary liquid velocity and liquid viscosity on average solids holdup

Fig. 5 shows a surface plot for variation of average solids holdup with auxiliary liquid velocity and liquid viscosity. This result shows that there is a significant interaction between U_{L2} and μ_L which showed a saddle point. At a certain value of liquid viscosity (which is somewhere between the range of viscosities chosen) maximum value of the average solids holdup is noticed. This maximum value increases at increasing

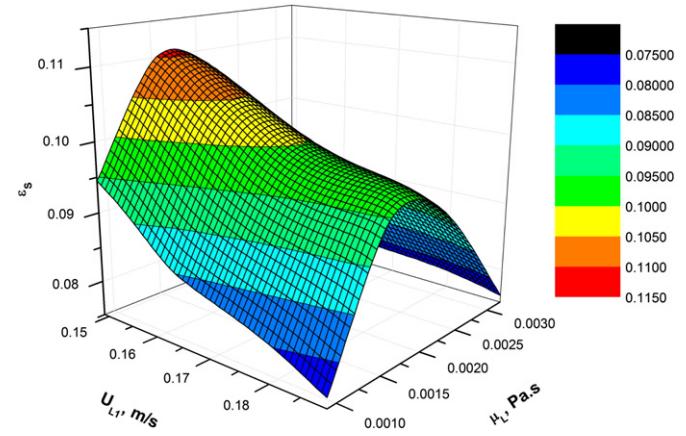


Fig. 3. Effect of primary liquid velocity and liquid viscosity on average solids holdup.

auxiliary liquid velocity. From this study, it is observed that variation of the liquid viscosity plays an important role.

4.1.6. Interaction effect of solids inventory and liquid viscosity on average solids holdup

The interaction effect of solids inventory and liquid viscosity studied for the system is illustrated in Fig. 6. A dome shaped orientation of the surface plot signifies the same effect as discussed above.

4.2. Solids circulation rate

Solids circulation rate is an important hydrodynamic variable which decides the intimate contact between the solids and fluid for an adsorption and desorption as well as reaction and regeneration. Also gives an idea about the residence time of solids in the riser. Statistical model developed for the prediction of the solids circulation rate in terms of active factors is as follows:

$$G_S = -39.695 - (291.575 \times U_{L1}) + (77.489 \times U_{L2}) + (47.146 \times L_0) + (58027.211 \times \mu_L) - (488.211 \times U_{L1} \times U_{L2}) + (249.472 \times U_{L1} \times L_0) - (40350.737 \times U_{L1} \times \mu_L) + (852.220 \times U_{L2} \times L_0) - (23703.612 \times U_{L2} \times \mu_L) + (6181.080 \times L_0 \times \mu_L) + (1119.210 \times U_{L1}^2) + (2299.472 \times U_{L2}^2) - (168.918 \times L_0^2) - (1.153 \times 10^7 \times \mu_L^2). \quad (5)$$

In this developed statistical model, for the prediction of the solids circulation rate, individual effects like auxiliary liquid velocity, solids inventory and liquid viscosity as well as in interaction effects like

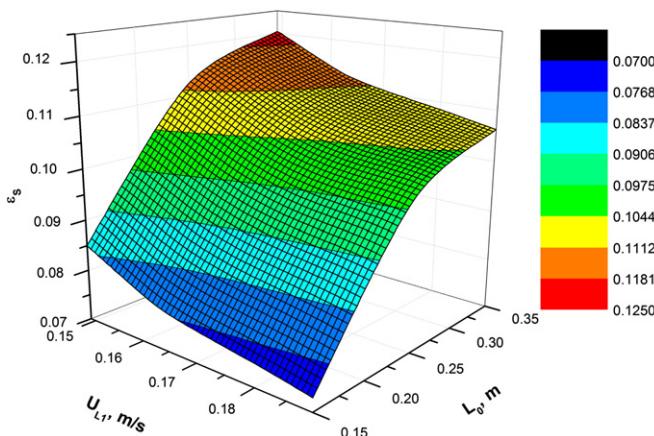


Fig. 2. Effect of primary liquid velocity and solids inventory on average solids holdup.

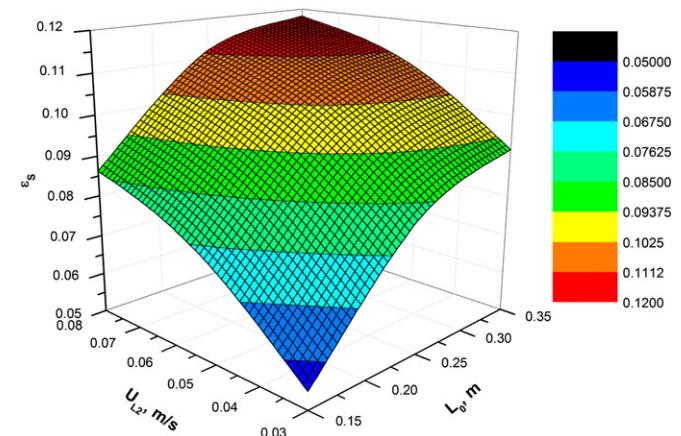


Fig. 4. Effect of auxiliary liquid velocity and solids inventory on average solids holdup.

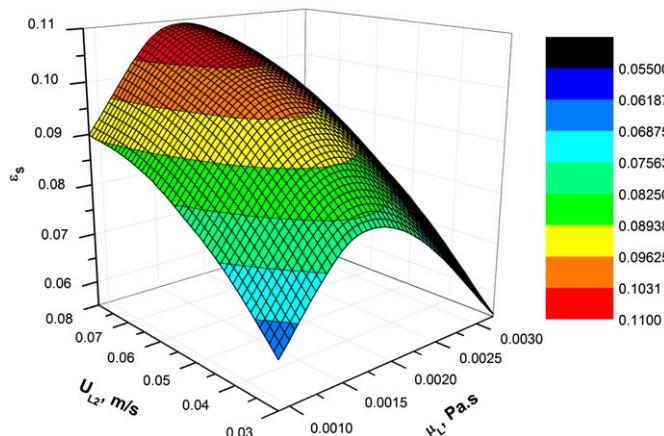


Fig. 5. Effect of auxiliary liquid velocity and liquid viscosity on average solids holdup.

primary liquid velocity–solids inventory, auxiliary liquid velocity–solids inventory, solids inventory–liquid viscosity; and square effect terms of liquid velocities are shows positive effect.

4.2.1. Effect of interaction of primary and auxiliary liquid velocity on solids circulation rate

Surface plot for the combined effect of primary and auxiliary liquid velocity on solids circulation rate is shown in Fig. 7. For an individual effect in the same study, it is observed that the solids circulation rate increases with an increase in the primary liquid velocity and auxiliary liquid velocity when the rest of the factors are held constant. As this liquid velocity increases it will try to push the solids into the reactor and form a closed loop. As a result of this interaction effect also showing positive influence results in increase in the solids circulation rate.

4.2.2. Interaction effect of primary liquid velocity and solids inventory on solids circulation rate

The interaction effect of primary liquid velocity and solids inventory is shown in Fig. 8. Effect of interaction effect is monotonous. For an individual effect, the solids circulation rate shows nonlinear increasing behavior with respect to the primary liquid velocity at a constant auxiliary liquid velocity and liquid viscosity. However, range of primary liquid velocity was chosen from 0.15 m/s to 0.19 m/s. This shows a lower deviation in the solids circulation rate because the primary and auxiliary liquid velocities were chosen such that this range would be common for all the range of viscosities studies. A linear change in the

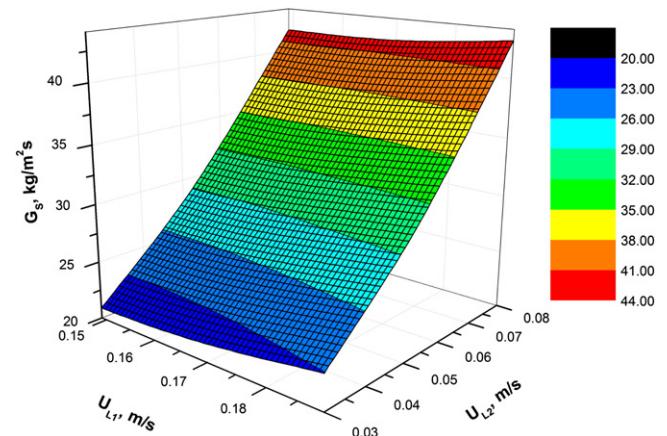


Fig. 7. Effect of primary liquid velocity and auxiliary liquid velocity on solids circulation.

solids circulation rate is seen up to 0.25 m of solids inventory after which a sluggish nature of the solids circulation rate is clearly visualized.

4.2.3. Interaction effect of primary liquid velocity and liquid viscosity on solids circulation rate

An interaction effect of primary liquid velocity and liquid viscosity is shown in Fig. 9. In this study this effect has been observed at constant auxiliary liquid velocity and solids inventory. In this study, it is observed that as the primary liquid velocity and liquid viscosity are increased simultaneously solids circulation rate shows a nonlinear behavior in solids circulation rate. With increasing combined effect, the solids circulation rate increases and reaches maximum at a certain point and after that point it tends to decrease as the viscosity effect is dominant. This shows that the dominance of first factor is higher compared to the second when the second factor is at a lower value and vice versa.

4.2.4. Interaction effect of auxiliary liquid velocity and solids inventory on solids circulation rate

The interaction effect of auxiliary liquid velocity and solids inventory in the downcomer is shown in Fig. 10. This interaction effect has been carried out at constant auxiliary liquid velocity and liquid viscosity. It is observed that, increasing auxiliary liquid velocity and solids inventory results in a monotonic increase in solids circulation rate. This trend is the same as that in the case of averages solids holdup (Fig. 4). The main reason for this effect is not only individual but also interaction effects are having positive significance on the solids circulation rate.

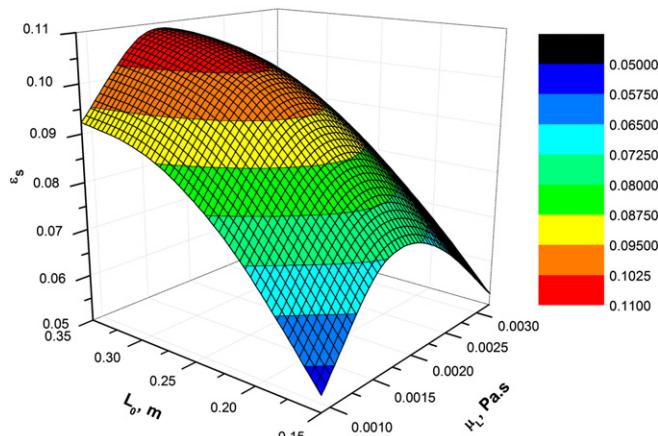


Fig. 6. Effect of solids inventory and liquid viscosity on average solids holdup.

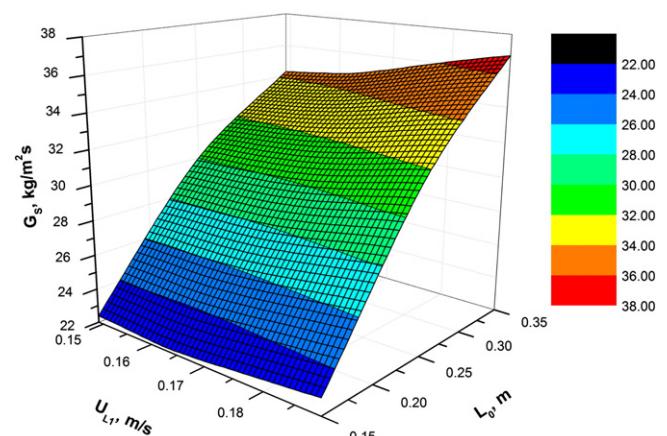


Fig. 8. Effect of primary liquid velocity and solids inventory on solids circulation rate.

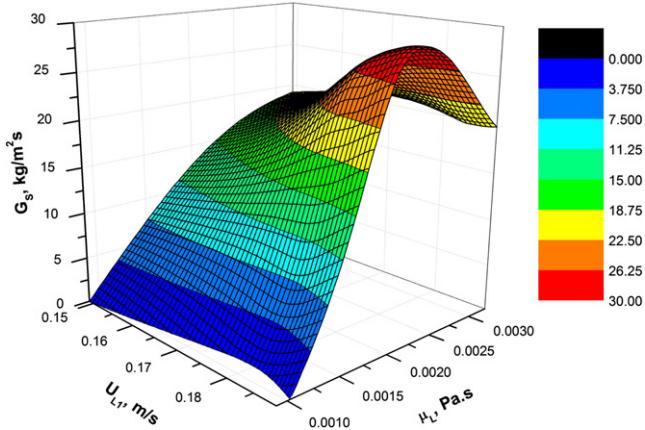


Fig. 9. Effect of primary liquid velocity and liquid viscosity on solids circulation rate.

4.2.5. Interaction effect of auxiliary liquid velocity and liquid viscosity on solids circulation rate

An interaction effect between auxiliary liquid velocity and liquid viscosity studied at constant primary liquid velocity and solids inventory is shown in Fig. 11. The combined effect of these properties was studied at constant primary liquid velocity and solids inventory. From this study, it is evident that the solids circulation rate demonstrates simultaneous increasing and decreasing trends. This shows a nonlinear behavior of the system with respect to operating parameters studied. This result is similar to that noticed in case of average solids holdup (Fig. 5). However, the response of solids circulation rate was faster compared to solids holdup. This shows that the significant interaction is between these variables as well as viscosity effect's dominance on the prediction of the solids circulation rate.

4.2.6. Interaction effect of solids inventory and liquid viscosity on solids circulation rate

The combined effect of solids inventory in the downcomer and liquid viscosity is shown in Fig. 12. This interaction effect is observed at constant liquid velocities. From this it is observed that, simultaneous increase in the solids inventory and liquid viscosity increases solids circulation rate to a maximum value followed by a decrease. These results are similar to Fig. 6 and same reason holds good in this case as well.

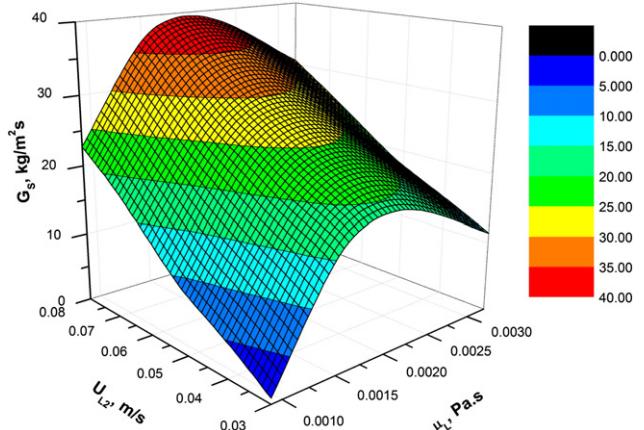


Fig. 11. Effect of auxiliary liquid velocity and liquid viscosity on solids circulation rate.

5. Statistical model validation

The performance of the developed statistical model is tested by comparing the predicted response with the experimental response noticed. The experimental data points chosen were other than considered in the design matrix for model development. Eqs. (4) and (5) are used for the prediction of statistical response. Numbers of datasets used for the model validation chosen were 447 and 333 for ε_S and G_S respectively. Figs. 13 and 14 are the parity plots obtained for the validation of developed statistical model in terms of average solids holdup and solids circulation rate respectively. The correlation coefficient (R) for this comparison is found to be 0.89 and 0.92 for average solids holdup and solids circulation rate respectively. From this statistics it very clear that predicted responses are very close to the respected observed experimental as the R values closely approaches to 1. Another method employed to find out the accuracy of the developed model is the % deviation of the model with the experimental observations. The percentage deviation is defined as the error between experimental and predicted response over experimental response multiplied by 100. % deviation is found to be $\pm 25\%$ and $\pm 35\%$ for ε_S and G_S respectively. However, larger deviations were noticed in the case of solids circulation rate because of the nature of solids circulation rate change noticed with the liquid velocity. That is at lower velocities changes in solids circulation were rapid and after over particular liquid velocities changes in solids circulation rates were insignificant. Since the developed model has to capture both the variations hence resulted in more deviation. Therefore, these observations are in good agreement with the developed statistical model and strongly evident that the developed

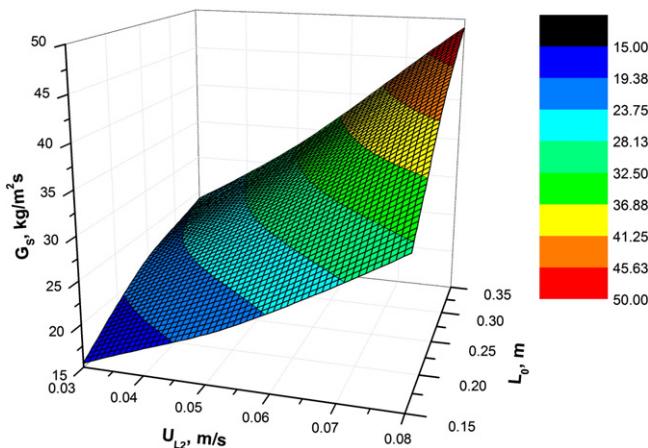


Fig. 10. Effect of auxiliary liquid velocity and solids inventory on solids circulation rate.

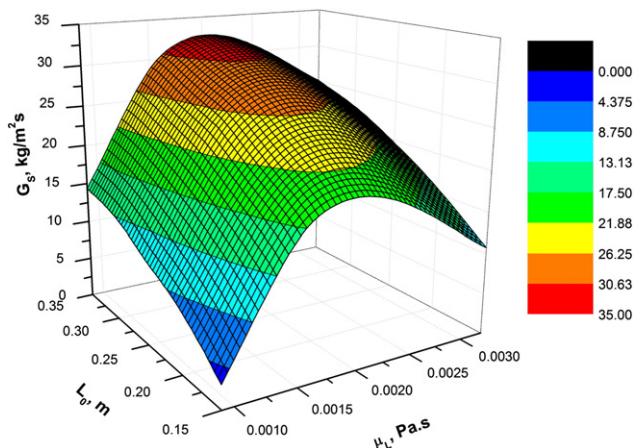


Fig. 12. Effect of solids inventory and liquid viscosity on solids circulation rate.

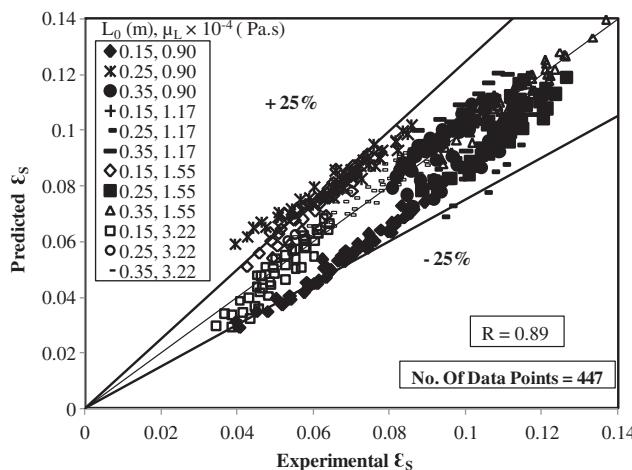


Fig. 13. Model validation parity diagram for average solids holdup.

statistical model gives the best predictions for the hydrodynamics of the LSCFB.

6. Conclusions

The statistical regression model has been developed and subsequently model suitability is confirmed by the ANOVA for the hydrodynamic prediction of the LSCFB. Investigations of the present study are summarized as follows:

1. This study supports the use of the statistical design approach for the model development in terms of interacting effects contributing to the suitability of the desired changes in the system under consideration. This is the major advantage of this technique which is hardly observed in the conventional methods.
2. The full factorial design with three levels of four factors has been employed to formulate the hydrodynamic variation along with the statistical performance. This regression analysis suggests the quadratic model for the prediction of hydrodynamic properties. Various statistical parameters confirm the suitability of the developed model.
3. The various interacting effects from the developed statistical model are examined and these show the close resemblance with the experimental observations.
4. A comparison between predictions from developed statistical model and experimental work has been investigated. This suggests the developed quadratic model is in ease of the predictions.

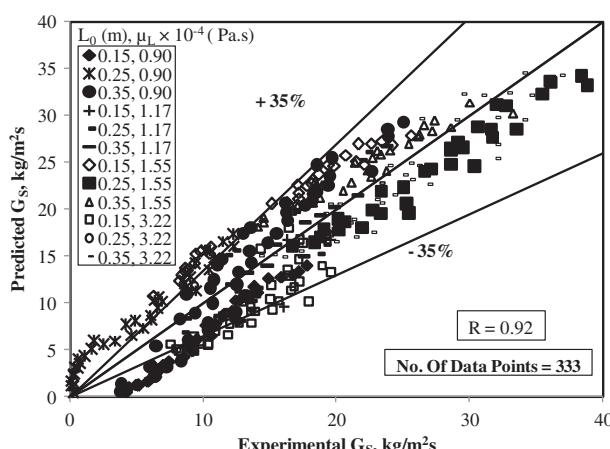


Fig. 14. Model validation parity plot for solids circulation rate.

Nomenclature

ANOVA	analysis of variance
CV	coefficient of variation
df	degrees of freedom
F-test	fishers test value
G_s	solids circulation rate ($\text{kg}/\text{m}^2 \text{s}$)
LSCFB	liquid solid circulating fluidized bed
L_0	solids inventory in the downcomer (m)
p-Value	probability value
R	correlation coefficient
SD	standard deviation
SS	sum of squares
MS	mean squares
U_{L1}	primary liquid velocity (m/s)
U_{L2}	auxiliary liquid velocity (m/s)
x_i, x_j	$U_{L1}, U_{L2}, L_0, \mu_L$ corresponding factors in the general model equation

Greek letters

β_{ij}	coefficients of the polynomial equation
ε_s	average solids holdup
μ_L	viscosity of the liquid (Pa·s)

Acknowledgments

Help rendered by Mr. Bishwadeep Bagchi, Department of Chemical Engineering, National Institute of Technology, Warangal, in refining the language of the manuscript in the final stage is thankfully acknowledged.

References

- [1] C.J. Pereira, T.M. Leib, Perry's Chemical Engineers' Handbook, 8th ed. McGraw-Hill Companies, 2007.
- [2] Z. Chen, Y. Yan, S.E.H.S. Elnashaie, Novel circulating fast fluidized-bed membrane reformer for production of hydrogen from steam reforming of methane, *Chem. Eng. Sci.* 58 (19) (2003) 4335–4349.
- [3] R. Veneman, Z.S. Li, J.A. Hogendoorn, S.R.A. Kersten, D.W.F. Brilman, Continuous CO_2 capture in a circulating fluidized bed using supported amine sorbents, *Chem. Eng. J.* 207–208 (2012) 18–26.
- [4] F. Berruti, J. Chauki, L. Godfroy, T.S. Pugsley, G.S. Patience, Hydrodynamics of circulating fluidized bed risers: a review, *Can. J. Chem. Eng.* 73 (1995) 579–602.
- [5] M. Andalib, G. Nakhla, J. Zhu, High rate biological nutrient removal from high strength wastewater using anaerobic-circulating fluidized bed bioreactor (A-CFBBR), *Bioprocess Technol.* 118 (2012) 526–535.
- [6] M. Patel, A.S. Bassi, J.-X. Zhu, H. Gomaa, Investigation of a dual-particle liquid-solid circulating fluidized bed bioreactor for extractive fermentation of lactic acid, *Biotechnol. Prog.* 24 (2008) 821–831.
- [7] X. Feng, S. Jing, Q. Wu, J. Chen, C. Song, The hydrodynamic behavior of the liquid-solid circulating fluidized bed ion exchange system for cesium removal, *Powder Technol.* 134 (2003) 235–245.
- [8] W. Liang, S. Zhang, J.-X. Zhu, Y. Jin, Z. Yu, Z. Wang, Flow characteristics of the liquid-solid circulating fluidized bed, *Powder Technol.* 90 (1997) 95–102.
- [9] Y. Zheng, J.-X. Zhu, The onset velocity of a liquid-solid circulating fluidized bed, *Powder Technol.* 114 (2001) 244–251.
- [10] S. Roy, A. Kemoun, M. Al-Dahan, M.P. Dudukovic, A method for estimating the solids circulation rate in a closed-loop circulating fluidized bed, *Powder Technol.* 121 (2001) 213–222.
- [11] Y. Zheng, J.-X. Zhu, N.S. Marwaha, A.S. Bassi, Radial solids flow structure in a liquid-solids circulating fluidized bed, *Chem. Eng. J.* 88 (2002) 141–150.
- [12] T. Wang, J. Wang, W. Yang, Y. Jin, Experimental study on bubble behavior in gas-liquid-solid three phase circulating fluidized beds, *Powder Technol.* 137 (2003) 83–90.
- [13] A. Atta, S.A. Razzak, K.D.P. Nigam, J.-X. Zhu, (Gas)-liquid-solid circulating fluidized bed reactors: characteristics and applications, *Ind. Eng. Chem. Res.* 48 (2009) 7876–7892.
- [14] P.V. Chavan, D.V. Kalaga, J.B. Joshi, Solid-liquid circulating multistage fluidized bed: hydrodynamic study, *Ind. Eng. Chem. Res.* 48 (2009) 4592–4602.
- [15] S.A. Razzak, S. Barghi, J.-X. Zhu, Axial hydrodynamic studies in a gas-liquid-solid circulating fluidized bed riser, *Powder Technol.* 199 (2010) 77–86.
- [16] V. Vidyasagar, K. Krishnaiah, P.S.T. Sai, Hydrodynamics of liquid-solid circulating fluidized bed: effect of dynamic leak, *Chem. Eng. J.* 138 (2008) 425–435.
- [17] V. Shilapuram, K. Krishnaiah, P.S.T. Sai, Comparison of macroscopic flow properties obtained by three methods of operation in a liquid-solid circulating fluidized bed, *Chem. Eng. Process.* 48 (2009) 259–267.

- [18] S. Vidyasagar, K. Krishnaiah, P.S.T. Sai, Macroscopic properties of liquid–solid circulating fluidized bed with viscous liquid medium, *Chem. Eng. Process.* 50 (2011) 42–52.
- [19] V. Shilapuram, P.S.T. Sai, Axial solids holdup distribution in a liquid–solid circulating fluidized bed: effect of liquid distributor, method of operation, and viscosity of the fluidized media, *Ind. Eng. Chem. Res.* 51 (2012) 16242–16250.
- [20] D.V. Kalaga, A. Dhar, S.V. Dalvi, J.B. Joshi, Particle liquid mass transfer in solid–liquid fluidized beds, *Chem. Eng. J.* 245 (2014) 323–341.
- [21] W. Liang, J.-X. Zhu, A core-annulus model for the radial flow structure in a liquid–solid circulating fluidized bed (LSCFB), *Chem. Eng. J.* 68 (1997) 51–62.
- [22] S. Roy, M.P. Dudukovic, Flow mapping and modeling of liquid–solid risers, *Ind. Eng. Chem. Res.* 40 (2001) 5440–5454.
- [23] J. Mazumder, J. Zhu, A.S. Bassi, A.K. Ray, Modeling and simulation of liquid–solid circulating fluidized bed ion exchange system for continuous protein recovery, *Biotechnol. Bioeng.* 104 (1) (2009) 111–126.
- [24] J. Mazumder, J. Zhu, A.S. Bassi, A.K. Ray, Multiobjective optimization of the operation a liquid–solid circulating fluidized bed ion-exchange system for continuous protein recovery, *Biotechnol. Bioeng.* 103 (5) (2009) 873–890.
- [25] D.V. Kalaga, R.K. Reddy, J.B. Joshi, S.V. Dalvi, K. Nandkumar, Liquid phase axial mixing in solid–liquid circulating multistage fluidized bed: CFD modeling and RTD measurements, *Chem. Eng. J.* 191 (2012) 475–490.
- [26] Y. Zheng, J.-X. Zhu, Overall pressure balance and system stability in a liquid–solid circulating fluidized bed, *Chem. Eng. J.* 79 (2000) 145–153.
- [27] V.V. Basava Rao, Ch. Sailu, D.K. Sandilya, An experimental study of liquid solid particle flow in circulating fluidized bed, *Chem. Eng. Commun.* 194 (3) (2007) 353–367.
- [28] I. Lorscheid, B.-O. Heine, M. Meyer, Opening the ‘black box’ of simulations: increased transparency and effective communication through the systematic design of experiments, *Comput. Math. Organ. Theory* 18 (2012) 22–62.
- [29] H.F. Abbas, I.F. Baker, Thermocatalytic decomposition of methane using activated carbon: studying the influence of process parameters using factorial design, *Int. J. Hydrol. Energy* 36 (2011) 8985–8993.
- [30] H.M. Jena, B.K. Sahoo, G.K. Roy, B.C. Meikap, Statistical analysis of the phase holdup characteristics of a gas–liquid–solid fluidized bed, *Can. J. Chem. Eng.* 87 (2009) 1–10.
- [31] D.T.K. Dora, Y.K. Mohanty, G.K. Roy, Hydrodynamics of three-phase fluidization of a homogeneous ternary mixture of regular particles experimental and statistical analysis, *Powder Technol.* 237 (2013) 594–601.
- [32] A.A. Al-Hassani, H.F. Abbas, W.M.A. Wan Daud, Hydrogen production via decomposition of methane over activated carbons as catalysts: full factorial design, *Int. J. Hydrol. Energy* 39 (2014) 7004–7014.
- [33] D.C. Montgomery, *Design and Analysis of Experiments*, Fifth ed. John Wiley and Sons, Inc, 2008.
- [34] Stat-ease Inc, *Design-Expert software*, 9.0.2. USA, Computer Software, 2013.