Modeling and Optimization of Recovery Process of Glycolic Acid using Reactive Extraction

Dipaloy Datta and Sushil Kumar

***Abstract*—The equilibrium reactive extraction of glycolic acid (GA) from aqueous solution is studied using tri-*n*-octylamine (TOA) as extractant dissolved in organic solvents (cyclohexane and 1-decanol). Experimental studies are designed using central composite orthogonal design method to investigate the main and interaction effects of initial GA concentration in the aqueous phase (C_{in}, mol/L), initial TOA composition in the organic phase ($C_{TOA,o}$, %v/v) and modifier composition (M, %v/v) on the degree of extraction (Y). The process design parameters are optimized based on Y and using bio-inspired optimization algorithm, called differential evolution (DE). A quadratic response surface model is satisfactorily described with R^2 of 0.98. The optimum conditions using DE are obtained as C_{in} = 0.24 mol/L, $C_{TOA,o}$ = 16.1 (%v/v), and M = 80.38 (%v/v). At this optimum conditions, a Y of 73.18% can be obtained from the model. Experimental verification gives a Y of 69.25% with a model error of 5.7%. This indicates high reliability of the model.**

***Index Terms*—Reactive extraction, glycolic acid, modeling, optimization.**

I. INTRODUCTION

Glycolic acid (GA) has a broad spectrum of consumer and industrial applications. It is used in leather, oil, gas, laundry, textile industries and as a component in personal care products like skin care creams. Commercially, GA is produced from petroleum feed-stocks. The chemical industry has come under increasing pressure to make chemical production more eco-friendly due to its reliance on fossil resources, its environmentally damaging production processes and its toxic byproducts and waste. Within this framework, bio-based chemistry and biotechnologies offer great prospects. Therefore, it is essential to replace the petroleum based feed-stocks by renewable resources for the sustainable development of industry. Glycolic acid can be produced by the enzymatic conversion of glycolonitrile by nitrilase as an aqueous solution of ammonium glycolate [1].

Classical extractants (aliphatics, aromatics, ketones, alcohols etc.) have almost no ability to extract carboxylic acids from their aqueous solutions because of their low distribution coefficients (lower than 1) [2], [3]. Also hydrophilic nature of glycolic acid ($\log P$ = -1.097, where P is the distribution coefficient of solvent in a standard 1-octanol and water two-phase system, [4]) and low concentration found in the fermentation broth, makes it poorly extractable by common organic solvents. For an affordable recovery process comparatively a high distribution coefficient is a must. Reactive extraction with an extractant (organophosphoric and aminic extractants) has been gaining great attention to solve this problem [5]-[8]. In the reactive extraction, a reaction occurs between the extractant and the solute (acid) and forms an acid-extractant complex. This complex is solubilized into the organic phase using suitable extractant-diluent system, and then the acid is back-extracted from the organic phase [2]. The extractant is generally diluted with an organic (conventional) solvent to get appropriate physical properties such as viscosity, density, surface tension etc. of the organic phase [9].

Several studies on the reactive extraction of glycolic acid with different extractants (phosphoric and aminic) dissolved in several diluents (alkanes, ketones, alcohols etc.) are carried out to determine the effect of various parameters such as initial acid concentration, initial extractant concentration, type of extractant, effect of diluents, effect of temperature on the recovery of this acid [5, 10-13]. To maximize the recovery of glycolic acid from aqueous solution, the effective choice and optimum combinations of these design variables are essential. In this sense, response surface methodology (RSM) is a powerful mathematical method suitable for modeling and simulation of various processes in real applications [14]-[16]. RSM model approximates the functional relationships between input (design) variables and output variable (response) of the process using experimental data and regression analysis. The developed model can then be used to estimate the optimum process variables (parameters) to maximize or minimize the response [17].

Most of the traditional optimization algorithms based on gradient methods have the possibility of getting trapped at local optimum depending upon the degree of non-linearity and initial guess [18]. The non-traditional optimization techniques based on evolutionary computation and natural phenomenon such as genetic algorithm (GA), differential evolution (DE), etc. have been developed to overcome the above said problems and to obtain more suitable and exact optimum solution of the process [6, 18].

In this paper, the equilibrium experimental data for reactive extraction of glycolic acid are designed using central composite orthogonal design method and these data are modeled using response surface methodology. The critical and effective independent parameters (design variables) for the recovery of glycolic acid using reactive extraction are chosen as initial GA concentration in the aqueous phase (C_{in}, mol/L), initial TOA composition in the organic phase ($C_{TOA,o}$, %v/v) and modifier composition (M, %v/v) and the degree of extraction (Y) as the response. The process design parameters are optimized based on Y and using bio-inspired optimization algorithm, called differential evolution (DE).

Manuscript received February 22, 2012; revised March 26, 2012.

The authors are with the Chemical Engineering Department, Birla Institute of Technology & Science (BITS) PILANI, Pilani, Rajasthan 333031, INDIA (e-mail: dipaloy@gmail.com; sushilk2006@gmail.com).

II. EXPERIMENTAL SECTION

A. Materials

Glycolic acid of purity 99.5 %w/w is purchased from Spectrochem, India. Tri-*n*-octylamine (density = 809 kg/m^3; purity = 98 %w/w; molar mass 353.68 kg/kmol) procured from Spectrochem, India, is used as an extractant in this study. Diluents such as cyclohexane (density = 779 kg/m^3; purity = 99 %w/w; S. D. Fine-Chem, India) as an inert diluent and 1-decanol (density = 830 kg/m^3; purity = 98 %w/w; Spectrochem Pvt. Ltd., India) as a modifier are used to prepare the organic solution. Sodium hydroxide (Merck, Germany) is used for titration and phenolphthalein solution (CDH, India; pH range of 8.2 to 10.0) is used as an indicator in the titration.

B. Method

The equilibrium extraction experiments are carried out in a temperature controlled reciprocating water shaker bath (REMI Labs, HS 250, India) using conical flasks of 100 ml with equal volumes of aqueous and organic phase (20 ml). The phase mixture is then shaken at 100 rpm for 6 hrs at 298 K. After reaching equilibrium, aqueous and organic phases is kept for 2 hrs for separation in separating funnel (125 ml) at constant temperature (298 K). The concentration of the residual glycolic acid in the aqueous phase is determined by titration with NaOH solution of 0.05 N and phenolphthalein as an indicator. The acid concentration in the organic phase at equilibrium is calculated by mass balance. The repeatability for few data points is checked and found within ±5% of error.

The efficiency of the equilibrium reactive extraction process is analyzed by calculating the degree of extraction (*Y*) which is defined as a ratio of acid concentration in the organic phase at equilibrium ($\overline{C}$) to initial acid concentration in the aqueous phase (C_{in}).

$$Y = \frac{\overline{C}}{C_{\text{in}}} \times 100 \tag{1}$$

III. RESULTS AND DISCUSSION

A. Reactive Extraction

The equilibrium reactive extraction mechanism can be described as a set of reactions between *m* molecules of acid (HA), and *n* molecules of extractant (T) to form various (*m*:*n*) acid-extractant complexes and is represented as:

$$m\text{HA} + n\overline{\text{T}} \Leftrightarrow \overline{(\text{HA})_m(\text{T})_n} \tag{2}$$

where, $\overline{(\text{HA})_m(\text{T})_n}$ is the formed acid-extractant complex in the organic phase at equilibrium. Depending on the molar ratios between the reactants (glycolic acid and TOA), different types of chemical interactions are possible [7]. The use of inert diluent (e.g. cyclohexane) in the case of high initial glycolic acid concentration in the aqueous solutions, may lead to the formation of a stable emulsion at the interface of aqueous and organic phases. Therefore, to avoid emulsion formation, a phase modifier (e.g. 1-decanol) is added to the organic solvent which also assures a higher solubility of formed acid-extractant complex in the organic phase by solvation.

B. Response Surface Methodology Approach and Experimental Design

The development of an industrial process requires study of the various process parameters, which can be achieved by exhaustive experimentation. The experiments are carried out by varying numerous experimental units to evaluate the performance of the system in terms of single/many output variable (s). It also requires comprehensive observation and gathering of information about the process and the system. These experimental data are useful to draw many valuable results and inferences about the system and the process. Therefore, in order to obtain already set objective conclusion, an experimenter needs to plan and design the experiments, and analyze the results. The approximation of the response function in terms of input variables is called Response Surface Methodology (RSM). RSM is a mathematical and a statistical technique that is applied for the construction of empirical models based on experimental data and experimental design [19, 20].

In RSM modeling the input variables are normalized or scaled to coded levels which usually vary from a minimum level (−*α*) up to a maximum level (+*α*). Generally, in the case of insufficient knowledge of the true response surface, a first-order model is helpful to approximate the shape of the response. When the first order model is unable to describe the behavior of the response function, it is then upgraded to a second order model [21]-[23]. An expression describing a second order RSM model can be written as:

$$Y = A_0 + \sum_{i=1}^{k} A_i x_i + \sum_{i=1}^{k} A_{ii} x_i^2 + \sum_{i<j}^{k} A_{ij} x_i x_j \tag{3}$$

where *Y* denotes the predicted response; x_{i} refers to the input design variables in terms of coded levels; A_{o}, A_{i}, A_{ii}, A_{ij} are the regression coefficients; and *k* is the total number of factors in the experimental design. The regression coefficients are determined using ordinary least squares (OLS) method. OLS estimator can be written as follows [24]:

$$A_{\text{OLS}} = (X^T X)^{-1} X^T Y \tag{4}$$

where A_{OLS} is a vector of regression coefficients; *X* is an extended designed matrix of the coded levels of the input variables; and *Y* is a column vector of responses determined experimentally.

The actual values of design variables (C_{in}, $C_{\text{TOA,o}}$ and *M*) are coded as x_{i} (dimensionless) and presented in Table I. The coding of the design parameters is done according to the following equations:

TABLE I: INDEPENDENT VARIABLES (THEIR CODED AND ACTUAL VALUES)

Design variables	Coded variables	Coded levels				
		-*α*	-1	0	1	+*α**
C_{in} (mol/L)	x_1	0.24	0.3	0.6	0.9	0.96
$C_{\text{TOA,o}}$ (%v/v)	x_2	3.93	5	10	15	16.1
M (%v/v)	x_3	19.63	25	50	75	80.38

*α = 1.215 (star point for CCOD) for *k* = 3

$$x_1 = \frac{C_{\text{in}} - 0.6}{0.3} \tag{5}$$

$$x_2 = \frac{C_{\mathrm{TOA,o}} - 10}{5} \quad (6)$$

$$x_3 = \frac{M - 50}{25} \quad (7)$$

In the present study, the experiments are designed considering (i) 2^k factorial CCOD points; (ii) n_c central points (coded as zero value); (iii) two axial points from the central design point at a distance of $\pm\alpha$; and (iv) $2k$ star points. Hence, the total number of experimental design points becomes as, $n = 2^k + 2k + n_c$. Therefore, with $k = 3$, $n_c = 1$ (two replicates at the center point) and $\alpha = \pm 1.215$ [23] for CCOD, a total of 16 batch experiments are carried out. Each experimental run represents a unique combination of factor's level. The degree of extraction (Y) is determined for each experimental run using Eq. 1 and the values are shown in Table II.

TABLE II: EXPERIMENTAL DESIGN POINTS AND RESPONSE

Run No.	Run type	Coded variables						Response (Y)
		C_{in}	x_1	$C_{TOA,o}$	x_2	M	x_3	
1	O1	0.9	1	15	1	75	1	31.63
2	O2	0.3	-1	15	1	75	1	66.38
3	O3	0.9	1	5	-1	75	1	15.10
4	O4	0.3	-1	5	-1	75	1	31.07
5	O5	0.9	1	15	1	25	-1	18.18
6	O6	0.3	-1	15	1	25	-1	25.19
7	O7	0.9	1	5	-1	25	-1	7.54
8	O8	0.3	-1	5	-1	25	-1	10.90
9	S1	0.96	α	10	0	50	0	20.41
10	S2	0.24	$-\alpha$	10	0	50	0	34.85
11	S3	0.6	0	16.1	α	50	0	42.00
12	S4	0.6	0	3.93	$-\alpha$	50	0	15.10
13	S5	0.6	0	10	0	80.38	α	32.75
14	S6	0.6	0	10	0	19.63	$-\alpha$	11.74
15	C1	0.6	0	10	0	50	0	29.39
16	C2	0.6	0	10	0	50	0	25.19

O = orthogonal design points, C = center points, S = star points, −1 = low value, 0 = center value, +1 = high value, +/− α = star point value

These experimental data (Table II) are regressed to obtain regression coefficient of the RSM model. The significance of each regression coefficient is determined by Student's t-test (a null hypothesis test) and Fischer distribution test (F-test). Only the significant contribution of each design variable on the response function is considered and approximate RSM model equation of second order polynomial describing Y of reactive extraction as a function of coded design variables is represented as:

$$\begin{aligned} Y = 28.069 - 7.18x_1 + 9.993x_2 \\ + 9.851x_3 - 2.894x_3^2 - 2.804x_1x_2 \\ - 5.044x_1x_3 + 3.364x_2x_3 \end{aligned} \quad (8)$$

Subjected to: $-\alpha \le x_i \le +\alpha$ and $0 \le Y \le 100$ (9)

Statistical significance of the regression equation (Eq. 8) is analyzed using analysis of variance (ANOVA) and results are summarized in Table III. ANOVA compares the variation of regression data (the residual) about the mean. Such kind of comparison of variations is very much useful to evaluate the significance of the regression analysis and to predict the exactness of the response function. The significance of regression can be evaluated using F-test (Fischer's test) value and P-value (null-hypothesis test). The F-value predicts the quality of the entire model considering all design variables at a time. The P-value is the probability of the independent design variable having very little or insignificant effect on the dependent variable (response). Larger F-value signifies better fit of the RSM model to the experimental data. The quality of the overall model can be improved by increasing F-value which can be achieved by eliminating some terms and/or independent variables in the RSM model with a low t-test value. The P-value is used to interpret the values obtained by t-test. P-value less than 0.05 means that the design variable (factor), when considered in the model equation, will contribute less than 5% change in the dependent variable. If the P-value is greater than 0.05, a strong argument can be put up to eliminate a particular independent variable from the RSM model. The values of degrees of freedom (DF), sum of squares (SS), mean square (MS) for the model and residuals, F-value, P-value, and coefficient of determination (R^2) are determined by ANOVA and presented in Table III. ANOVA analysis as given in Table III shows that a larger F-value (greater than unity) and P-value near about zero are obtained for Eq. 8. The value of R^2 (= 0.98) indicates that the RSM regression model is found to be very significant and explains about 98% of the experimental data with the model predicted data (Fig. 1).

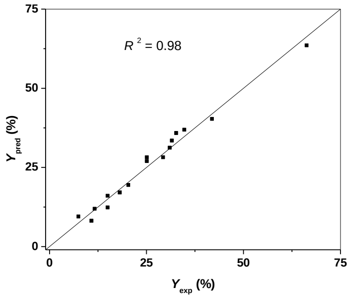

Fig. 1. RSM model predicted versus experimental degree of extraction.

TABLE III: ANALYSIS OF VARIANCE (ANOVA) FOR RSM MODEL

Source	DF	SS	MS	F-value	P-value	R^2
Regression	7	3118.98	445.57	55.97	3.61×10^{-6}	0.98
Residual	8	63.68	7.96			
Total	15	3182.66				

The effects of design variables (C_{in}, $C_{TOA,o}$, and M) on Y are determined by plotting response surface plots on 2-D planes and shown in Figs. 2-4. Effect of C_{in} on Y at various $C_{TOA,o}$ and at fixed modifier composition (M = 50 %v/v) is shown in Fig. 2. This figure also indicates the effect of interaction between both variables (C_{in} and $C_{TOA,o}$). As indicated by the Fig. 2, an increase in the acid concentration decreases Y for a fixed amount of TOA composition. At higher acid concentration the competency between acid molecules to attach with the extractant molecules becomes more and hence less amount of acid molecule can be extracted by the amine molecule decreasing Y. At higher amine composition there are sufficient amount of amine molecules available for a particular acid concentration and hence a greater value of *Y*.

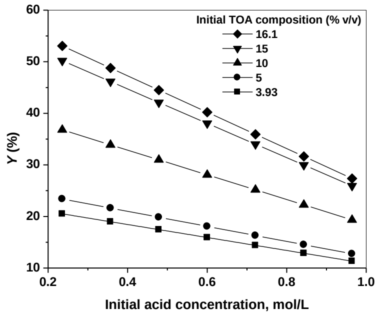

Fig. 2. Effect of C_{in} and $C_{TOA,o}$ on *Y* (%) at *M* = 50 %v/v.

Fig. 3 elaborates the variation of degree of extraction as a function of C_{in} at different *M* values and at $C_{TOA,o}$ = 10 %v/v. Since TOA has a relatively high viscosity and density, it is used along with diluents, which could facilitate good phase separation in the continuous extraction process. Diluents chosen in the study are cyclohexane from the inactive chemical class, and 1-decanol as modifier from active chemical class to examine the effect of diluent-complex interactions. These interactions generally have been found to affect the stoichiometry of reaction and magnitude of the corresponding equilibrium constants.

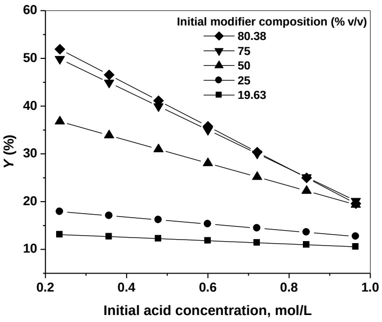

Fig. 3. Effect of C_{in} and *M* on *Y* (%) at $C_{TOA,o}$ = 10 %v/v.

From the Fig. 3 it can then be observed that the solubility of extracted species increases in the organic phase. So, *Y* of GA increases with an increase in the concentration of 1-decanol (modifier) in the mixture of TOA and cyclohexane.

The significant effect of *M* on *Y* (response) with $C_{TOA,o}$ is shown in Fig. 4 at C_{in} = 0.6 mol/L. In this study 1-decanol has been used as a modifier which is an active polar solvent (dipole moment, μ = 2.62 *D*). Use of non-polar solvents (e.g. cyclohexane, μ = 0 *D*) at higher initial concentration of glycolic acid in aqueous solutions, could lead to the formation of a stable emulsion and dimer in the organic phase. Therefore, a modifier is generally added to the organic phase to avoid such kind of problems and assures a higher solubility of the formed acid-amine complex in the organic phase. Active diluent, 1-decanol is having an active group (–OH, proton donor), which enhance the extractability of low polar TOA. On the other hand, non-polar diluents do not affect the extraction process significantly. Fig. 4 dictates that with increase in both values of *M* and $C_{TOA,o}$, *Y* increases.

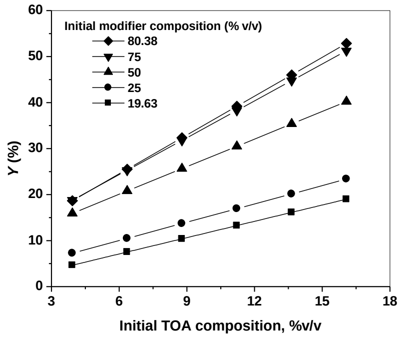

Fig. 4. Effect of $C_{TOA,o}$ and *M* on *Y* (%) at C_{in} = 0.6 mol/L.

C. Optimization using Differential Evolution

In science and engineering, optimization is defined as the method of minimizing or maximizing an objective function comprised of different independent variables and finding the values of those variables for which the objective function takes on minimum or maximum value within the defined domains of variables.

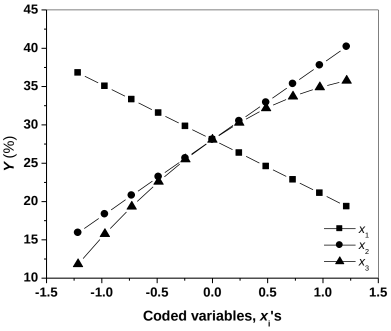

Fig. 5. Effect of various factors on the degree of extraction.

Fig. 5 describes the effect of one of the parameters as coded variable on *Y*. It can be seen that with the increase in the values of x_1, there is a decrease in the *Y*, but with the increase in the values of x_2 and x_3, the *Y* increases. It means there is a trade-off or balance between the values of x_i's which will optimize (maximize in this case) the response function. During the last two decades there has been a growing interest in optimization algorithms, which are based on the principle of evolution (survival of the fittest). The best-known algorithms in this class include Genetic Algorithm (GA), Differential Evolution (DE), Evolutionary Programming, Evolution Strategies and Genetic Programming. A brief review of the evolutionary computation techniques is presented by Babu, 2004 [18]. DE is a generic name for a group of algorithms that are based on the principles of GA, but have some inherent advantages over GA. DE algorithms are very robust and efficient and are able to find the global optimum of a function with ease and accuracy. DE algorithms are faster than GA. GA evaluates the fitness of a point in the search for optimum. In other words, GA evaluates a vector's suitability. In DE, this vector's suitability is called its cost or profit depending on whether the problem is a minimization or maximization. In GA only integers are used and coding is done in binary format while in DE, there is no coding involved and floating point numbers are directly used. In GA, when mutation is performed, bits are flipped at random with some mutation probability. This is essentially an XOR operation. In DE, direct addition is used. As already stated, DE in principle is similar to GA. So, as in GA, a population of points is used in the search for optimum. The population size in DE is denoted by *NP*. The dimension of vector (parameter) is denoted by *D*. *NP* number of competitions is to be carried out to decide the next generation. To start the DE, there is a population of *NP* vectors within the range of the vectors and one of these *NP* vectors is selected as the target vector. After that, two more vectors from the population are randomly selected and the difference between them (vector subtraction) is found out. This difference is multiplied by a factor *F* (specified at the start) and added to a third randomly selected vector from the population. The resultant vector is called the noisy random vector. Subsequently, crossover is performed between the target vector and the noisy random vector considering a crossover ratio (*CR*) between 0 and 1, to produce the trial vector. Then, a competition between the trial vector and the target vector is performed and the winner is replaced into the population. The same procedure is carried out *NP* times to decide the next generation of vectors. This sequence is continued till some convergence criterion is met. However, certain guidelines and heuristics are available for the choice of these parameters. *NP* should be 5-10 times the value of *D* (dimension of the problem). Initially, $F = 0.5$ is recommended. If this leads to premature convergence, then *F* needs to be increased. The range of *F* is 0-1.2 but the optimal range is 0.4-1.0. Values of $F < 0.4$ and $F > 1.0$ are seldom effective. $CR = 0.9$ is a good first guess. $CR = 0.9$ is to be tried first and then $CR = 0.1$ to be tried. Judging the speed, a value of *CR* between 0-1 is to be chosen. Based on these heuristics, the values of DE key parameters for the present problem are set as population size (NP) = 30, cross-over frequency (CR) = 0.7; multiplication factor (F) = 0.8. The fitness function, which is to be minimized, is considered as:

$$MSE = \sum_{j=1}^{j=N} (Y_j^{\exp} - Y_j^{pred})^2 \qquad (10)$$

where *MSE* is mean-squared error and *N* is the number of experiments.

For the optimization of RSM fitness function a computer code has been developed in MATLAB (v 7.0.1). DE has converged to the optimal value only after 3 generations. Therefore, it can be said that DE is comparatively faster than other optimization techniques [24]. The optimal solution obtained by means of RSM-DE involves the following conditions: C_{in} = 0.24 mol/L, $C_{\text{TOA,o}}$ = 16.1 %v/v, and M = 80.38 %v/v with predicted *Y* is about 72.18% by RSM model and about 69.25% from experiment. This value is greater than the value of 66.38% that is obtained for the run number 2 from initial experimental design (Table II). Therefore, the scope of optimization has been achieved by RSM-DE for this process.

IV. CONCLUSION

In this work, RSM and DE methods are applied for modeling and optimization of equilibrium reactive extraction process of glycolic acid considering three design variables (C_{in}, $C_{\text{TOA,o}}$ and M). The reactive extraction of glycolic acid from aqueous solutions with TOA (amine) dissolved in organic solvents (cyclohexane and 1-decanol) has been considered as the case study. The regression equation in coded variables has been constructed by RSM to describe the empirical functional relationships between input variables (factors) and response (degree of extraction) with R^2 for empirical model equal to 0.98. The optimum conditions are found to be: C_{in} = 0.24 mol/L, $C_{\text{TOA,o}}$ = 16.1 %v/v, and M = 80.38 %v/v with *Y* of 72.18 % and 69.25 % by the RSM model and by experiment, respectively. These results and findings can be used to design and optimize the recovery process of glycolic acid from aqueous solution using reactive extraction.

REFERENCES

[1] L. N. Xu, W. Fox, N. Zaher, and C. F. Dicosimo, "Method for the production of glycolic acid from ammonium glycolate by direct deammoniation," U. S. Patent WO/2006/069129, June, 23, 2006.

[2] A. S. Kertes and C. J. King, "Extraction chemistry of fermentation product carboxylic acids," *Biotechnol. Bioeng*. vol. 28, pp. 269-282, 1986.

[3] E. Ince, S. I. Kirbaslar, and S. Sahin, "Liquid-liquid equilibria for ternary systems of water + formic acid + dibasic esters," *J. Chem. Eng. Data*, vol. 52, pp. 1889-1893, 2007.

[4] A. D. John, *Lange's Handbook of Chemistry*, XXth ed. New York, U.S.A.: *McGraw-Hill Book Co. Inc*, 1972.

[5] S. Kumar, D. Datta, and B. V. Babu, "Experimental data and theoretical (chemodel using the differential evolution approach and linear solvation energy relationship model) predictions on reactive extraction of monocarboxylic acids using tri-n-octylamine," *J. Chem. Eng. Data*, vol. 55, pp. 4290-4300, 2010.

[6] S. Kumar, D. Datta, and B. V. Babu, "Estimation of equilibrium parameters using differential evolution in reactive extraction of propionic acid by tri-*n*-butyl phosphate, *Chem. Eng. Proc.* vol. 50, pp. 614-622, 2011.

[7] D. Datta and S. Kumar, "Reactive extraction of glycolic acid using tri-n-butyl phosphate and tri-n-octylamine in six different diluents: experimental data and theoretical predictions," *Ind. Eng. Chem. Res*, vol. 50, pp. 3041-3048, 2011a.

[8] D. Datta and S. Kumar, "Reactive extraction of 2-methylidenebutanedioic acid with *N*, *N*-dioctyloctan-1-amine dissolved in six different diluents: experimental and theoretical equilibrium studies at (298 ± 1) K, " *J. Chem. Eng. Data,* vol. 56, pp. 2574–2582, 2011b.

[9] H. Vogel, J. N. Starr, and C. J. King, "Extraction of carboxylic acids from aqueous solutions with the extractant system alcohol/tri-*n*-alkylamines," *Chem. Eng. Technol*, vol. 23, pp. 70-74, 2000.

[10] I. Inci, "Distribution of glycolic acid between water and different organic solutions," *Chem. Biochem. Eng. Q*, vol. 16, pp. 81-85, 2002.

[11] I. Inci, and H. Uslu, "Extraction of glycolic acid from aqueous solutions by trioctyl methylammonium chloride and organic solvents," *Chem. Eng. Data,* vol. 50, pp. 536-540, 2005.

[12] Y. Li, Y. Wang, Y. Li, and Y. Dai, "Extraction of glyoxylic acid, glycolic acid, acrylic acid, and benzoic acid with trialkylphosphine oxide," *Chem. Eng. Data,* vol. 48, pp. 621-624, 2003.

[13] S. Yunhai, S. Houyonga, L. Demingb, L. Qinghua, C. Dexing, and Z. Yongchuan, "Separation of glycolic acid from glycolonitrile hydrolysate by reactive extraction with tri-*n*-octylamine," *Sep. Purif. Technol.* vol. 49, pp. 20-26, 2006.

[14] X. Guan and H. Yao, Optimization of viscozyme lassisted extraction of oat bran protein using response surface methodology," *Food Chemistry*, vol. 106, pp. 345-351, 2008.

[15] M. Rajasimman, R. Sangeetha, and P. Karthik, "Statistical optimization of process parameters for the extraction of chromium (VI) from pharmaceutical wastewater by emulsion liquid membrane," *Chem. Eng. J,* vol. 150, pp. 275-279, 2009.

[16] O. S. Kuscua and D. T. Sponza, "Application of Box–Wilson experimental design method for 2,4-dinitrotoluene treatment in a sequential anaerobic migrating blanket reactor (AMBR)/aerobic

completely stirred tank reactor (CSTR) system," *J. Hazard. Mater*. vol. 187, pp. 222-234, 2011.

[17] R. J. Fox, D. Elgart, and S. C. Davis, "Bayesian credible intervals for response surface optima," *J. Statistical Planning and Inference*, vol. 139, pp. 2498-2501, 2009.

[18] B. V. Babu, *Process plant simulation*, New Delhi, India: Oxford University Press, 2004.

[19] R. H. Myers and D. C. Montgomery, *Response surface methodology: process and product optimization using designed experiments*, New York, U.S.A.: Wiley, 2002.

[20] M. A. Bezerra, R. E. Santelli, E. P. Oliveira, L. S. Villar, and L. A. Escaleira, "Response surface methodology (RSM) as a tool for optimization in analytical chemistry," *Talanta*, vol. 76, pp. 965-977, 2008.

[21] G. E. P. Box and J. S. Hunter, *Statistics for experiments*, New York, U.S.A.: Wiley, 1978.

[22] J. Garcia, F. Rodriguez, and J. A. Revenga, "Modeling solubility of solids in supercritical fluids using response surface methodology," *J. Chem. Technol. Biotechnol*, vol. 75, pp. 245-251, 2000.

[23] A. I. Khuri and J. A. Cornell, *Responses surfaces: design and analyses*, New York, U.S.A.: Marcel Dekker, 1987.

[24] N. Marchitana, C. Cojocarub, A. Mereuta, Gh. Duca, I. Cretescu, and M. Gonta, "Modeling and optimization of tartaric acid reactive extraction from aqueous, solutions: a comparison between response surface methodology and artificial neural network," *Sep. Purif. Technol*. vol. 75, pp. 273-285, 2010.

Dipaloy Datta is working as a Lecturer in Chemical Engineering Department at BITS PILANI, INDIA. He graduated in 2003 and obtained his Master's Degree in 2009 both in chemical engineering from BITS PILANI, INDIA. His research areas include Process Intensification, Reactive Extraction, Modeling & Simulation and Optimization. He has 28 research publications (10 journals, 16 conferences, 1 book and 1 book chapter) to his credit. Mr. Datta is a life associate member of IIChE.

Sushil Kumar is an Assistant Professor in Chemical Engineering Department at BITS-Pilani, INDIA. He earlier worked with Central Institute of Plastic Engineering and Technology (CIPET), Lucknow, INDIA, for two years as Technical Officer. He completed B Tech (chemical engineering) in May 2000 from Harcourt Butler Technological Institute (HBTI) – Kanpur, M Tech (chemical engineering) in January, 2003 from Indian Institute of Technology (IIT) – Kanpur and PhD (chemical engineering) in February, 2010 from Birla Institute of Technology and Science (BITS) – Pilani, INDIA. His research areas include Process Intensification, Separation Processes, Polymer Technology & Biopolymers, and Modeling & Simulation. He has 45 research publications (15 journals, 28 conferences and 2 book chapters) to his credit. Dr. Kumar is a life associate member of IIChE and a member of AIChE – 2010. He is the expert reviewer of 6 International Journals. He reviewed three books of Tata McGraw Hill publishers.