Optimization of Nano Calcium Carbonate Production Process Using Taguchi Method

Gh. Tahmasebi Pour and S. M. Mirzaee Moghadam

***Abstract*—This paper presents optimization of nano calcium carbonate production parameters to achieve the optimum production rate by using Taguchi method. The combination of optimum level of process parameters was obtained by using the analysis of signal-to-noise (S/N) ratio. The level of importance of the process parameters on production rate was determined by using analysis of variance (ANOVA). It was found that the optimum level of process parameters are solution flow rate of 9 lit/min, gas flow rate of 20 lit/min, and solution concentration of 70 gr/lit within the range of experiments and the process parameters in terms of impact significance were found to be gas flow rate, solution concentration, and solution flow rate, respectively. By using the optimum level of the process parameters, the production rate was enhanced by 168% in comparison to the mean value of the experimental results.**

***Index Terms*—Calcium carbonate, nano calcium carbonate, taguchi method, rotation packed bed.**

I. Introduction

With development of nanotechnology, nanopowders have found wide and diverse applications in many industries such as electronics, pharmaceutics, optics, ceramics, paper, and chemical and metallurgical industries. Unique properties of calcium carbonate nanopowder make it applicable in different industries such as pharmaceutics, cosmetics, paint, and composite industry.

One of the applied methods used for calcium carbonate nanopowder production is the high gravity reactive precipitation (HGRP) process. HGRP technology creates a high centrifugal gravity environment up to several hundred times greater than the earth's gravity using a rotating packed bed (RPB) system. This system was invented by Ramshaw and Mallinson in 1981 [1] as a gas–liquid contactor. In a high gravity environment, the liquid is uniformly distributed within the RPB and interacted with the gas stream well. The HGRP has a wide application in gas-liquid-solid, gas-liquid, and liquid-liquid multiphase reaction systems such as absorption [2]–[12], distillation [13], [14], deaeration [14]–[19], stripping [20]–[23], ozone oxidation [24]–[27], and reactive precipitation for nanoparticles synthesis [28].

To author's knowledge, effects of nano calcium carbonate production parameters in HGRP method such as the solution flow rate, gas flow rate, and the solution concentration on the nano calcium carbonate production rate and optimization of these parameters to achieve the optimum production rate have not been studied until now. In this research work, by studying the effects of the nano calcium carbonate production parameters on the production rate using the Taguchi method, the optimum level of production parameters and their priority for achieving optimum production rate have been investigated.

In the next section, experimental procedure will be discussed. Section III contains process optimization by employing Taguchi method. Section IV has been dedicated to analysis of experimental results. Confirmation experiment will be discussed in Section V and finally the concluding remarks will be given in Section VI.

II. Experimental Procedure

The high gravity reactive precipitation (HGRP) process was used to produce the nano calcium carbonate in this research. Fig. 1 shows schematic diagram of the experimental set-up used for the nano calcium carbonate production.

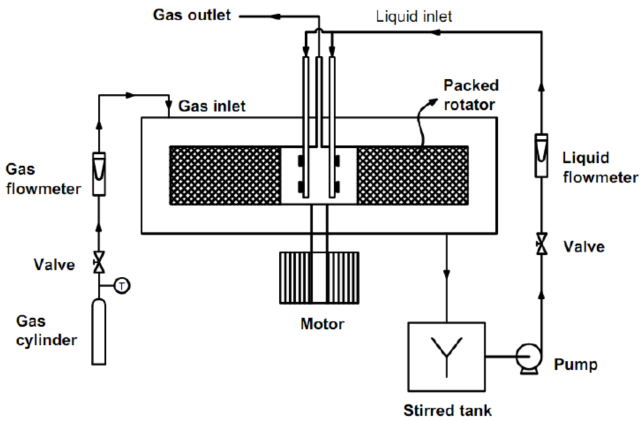

Fig. 1. Schematic diagram of the experimental set-up used for the nano calcium carbonate production.

As shown in Fig. 1, the main part of this system is a packed rotator that has been installed inside a stationary casing. It rotates at a speed of several thousand rpm. Liquid (calcium hydroxide in deionized water solution) is sprayed into the inside edge of the packed rotator via a slotted pipe connected to the liquid inlet. The liquid flows in the radial direction under centrifugal force, passing outward the packing and finally leaves the RPB through the liquid outlet. Gas (CO_2) is introduced from the outer edge of the packed rotator to flow inward counter currently against the liquid in the packing, and finally leaves the RPB via the gas outlet.

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was used as raw materials to synthesis the nano calcium carbonate in a gas-liquid-solid reactant system. The reaction equation can be written as:

$$Ca(OH)_2(s)+CO_2(g)+H_2O(l)\rightarrow CaCO_3(s)+2H_2O(l) \quad (1)$$

III. Process Optimization by Using Taguchi Method

TABLE I: Orthogonal Array of Experiments and Their Results (Lowest Value of Input Parameters Coincide with Lowest Level of Those Parameters)

Expt. No.	R_S (lit/min)	R_G (lit/min)	C_S (gr/lit)	R_P (gr/h)	S/N ratio (dB)
1	2	3	10	660.5	56.3976
2	2	10	40	1367	62.7152
3	2	20	70	1582.6	63.9872
4	5	3	40	787.5	57.9250
5	5	10	70	2180.9	66.7728
6	5	20	10	1339.6	62.5392
7	9	3	70	1710.5	64.6624
8	9	10	10	1416.5	63.0242
9	9	20	40	2354	67.4361

Taguchi method [29]-[33] is a quite effective way to deal with responses dependent upon multi-variables. This method is a powerful tool for design of experiments and provides a simple, efficient and systematic approach to determine optimum process parameters. Compared with the conventional approach, this method reduces drastically the number of experiments that are required to model the response functions. Traditional experimentation involves variation of one-factor-at-a-time, wherein the rest are held constant. For studying the individual effects of all factors, a lot of time and money must be spent. Taguchi technique overcomes all these drawbacks. In this method, the main effect is defined as the average value of the response function at a particular level of a parameter. The effect of a factor level is the deviation it causes from the overall mean response. The Taguchi method was devised for process optimization and identification of optimum combinations of factors for given responses. The steps involved in the Taguchi method are depicted as follow:

1) Identifying the main functions and the process parameters to be evaluated.
2) Determination of the number of levels for the process parameters.
3) Selecting the appropriate orthogonal array and assigning the process parameters to the orthogonal array and conducting the experiments accordingly.
4) Studying the experimental results by analysis of signal-to-noise (S/N) ratios to determine the optimum level of process parameters.
5) Investigating the results by analysis of variance (ANOVA) to identify the significance level of the process parameters on the main function.
6) Verifying the optimum process parameters through a confirmation experiment.

In this research, production rate taken as the main function of the nano calcium carbonate production process. The most important influencing parameters on the production rate are the solution flow rate (R_S), gas flow rate (R_G), and the solution concentration (C_S).

Regarding Taguchi quality design concept, process parameters, R_S, R_G, and C_S were defined in 3 levels and an L_9 orthogonal array table was chosen for the experiments as shown in Table I. The experiments were conducted according to the selected orthogonal array. The calculated values of the production rates (R_P) are given in Table I.

IV. Experimental Results Analysis and Discussion

A. Analysis of Signal-to-Noise Ratio

Based on Taguchi method [29]-[33], optimum level of process parameters is determined by analysis of S/N ratio. There are several categories of performance characteristics in the analysis of the S/N ratio as lower is better (LB), nominal is best (NB) and higher is better (HB). Since production rate of the nano calcium carbonate should be as high as possible, the higher is better (HB) was selected for obtaining optimum level of process parameters. The S/N ratio can be calculated as a logarithmic transformation of the loss function. For HB, the S/N ratio can be expressed as:

$$\eta = -10\log_{10}\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_i^2}\right) \quad (2)$$

where y_i is the response of the *ith* experiment and n is the repetition of each experiment. Regardless of the category of the performance characteristic, a larger S/N ratio corresponds to a better performance characteristic. Therefore, the optimum level of the process parameters is the level with the highest S/N ratio. The S/N ratios of L_9 experiments are shown in Table I. As an example, the calculation of S/N ratio for experiment No.1 is as follows:

$$\eta = -10\log_{10}\left(\frac{1}{660.5^2}\right) = 56.3976 \quad (3)$$

The mean of S/N ratio values for a process parameter in a defined level is determined by using the S/N ratio values given in Table I. For example the mean value of S/N ratios for R_S in level 1 is:

$$\frac{1}{3}(56.3976+62.7152+63.9872)=61.03\,dB \quad (4)$$

Fig. 2 (A-C) shows the mean value of S/N ratio against different levels of R_S, R_G, and C_S parameters. According to the results shown in Fig. 2, it can be concluded that:

1) Increase of solution flow rate (R_S) to 9 lit/min improves the production rate (R_P). Consequently, the best level of R_S for optimization of the production rate is 9 lit/min (level 3) within the range of experiments.
2) Increase of the gas flow rate (R_G) to 20 lit/min causes increase of the production rate again. Therefore the best gas flow rate is 20 lit/min (level 3) within the range of

experiments.

3) Increase of the solution concentration (C_S) to 70 gr/lit gives rise to a better production rate. Therefore, the best level of the solution concentration for optimization of the production rate is about 70 gr/lit (level 4) within the range of experiments.

Based on the mentioned conclusions, it is found that the best level of R_S, R_G, and C_S to achieve optimum production rate is level 3.

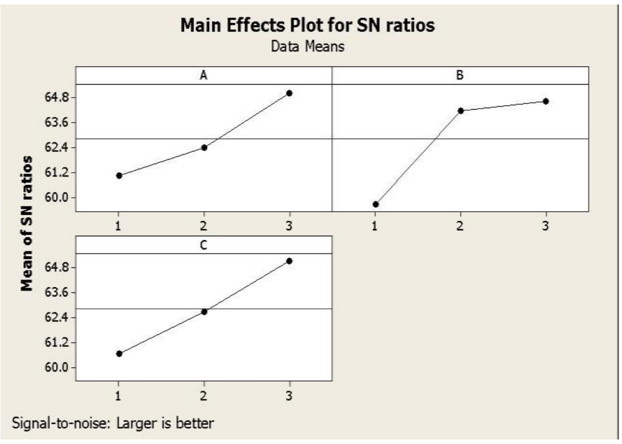

Fig. 2. Effect of different levels of process parameters on S/N ratio: A) R_S, B) R_G, and C) C_S.

B. Analysis of Variance (ANOVA)

The analysis of variance (ANOVA) is used to discuss the relative importance of process parameters on the production rate. The ANOVA is based on the following factors [29]:

- Degree of freedom (f): f denotes the number of independent variables and is calculated as:

$$f_T = N - 1, \; f_p = L_p - 1, \; f_e = f_T - \sum f_p \tag{5}$$

where f_T is the total degree of freedom, N is the total number of experiments, f_p is the degree of freedom for each parameter, L_p is the number of levels for the parameter, and f_e is the degree of freedom for error.

- Sum of squares (SS): SS_T, SS_p, and SS_e denote the total sum of squares, the sum of squares for each parameter, and the sum of squares of the error correlated to all parameters, respectively.

$$SS_T = \sum_{i=1}^{m} \eta_i^2 - \frac{1}{m}\left(\sum_{i=1}^{m} \eta_i\right)^2 \tag{6}$$

$$SS_p = \frac{1}{n}\sum_{j=1}^{n} (S_{\eta j})^2 - \frac{1}{m}\left(\sum_{i=1}^{m} \eta_i\right)^2 \tag{7}$$

$$SS_e = SS_T - \sum SS_p \tag{8}$$

where η_i is the *S/N* ratio of *ith* experiment, m is the total number of experiments, n is the repetition of each level of the parameter, $S_{\eta j}$ is the sum of the *S/N* ratio involving the parameter and level j (Table I).

- Variance (V): The variance related to each parameter (V_p) and the variance of error (V_e) is defined as follows:

$$V_p = \frac{SS_p}{f_p}, \; V_e = \frac{SS_e}{f_e} \tag{9}$$

- F-ratio (F): The F-ratio of each parameter (F_p) is given by

$$F_p = \frac{V_p}{V_e} \tag{10}$$

- Percentage of the contribution (P_p): P_p denotes the percentage of the total variance of each individual parameter.

$$p_p(\%) = \frac{SS_p}{SS_T} \times 100 \tag{11}$$

The results obtained from ANOVA of S/N ratios are shown in Table II. Greater F value and percentage contribution (P_p) for a parameter determine higher impact of the parameter on the production rate. Considering the ANOVA results given in Table II, the process parameters can be ranked in terms of their impact on the production rate as gas flow rate (R_G), solution concentration (C_S), and the solution flow rate (R_S), respectively.

TABLE II: ANALYSIS OF VARIANCE FOR S/N RATIOS OF PRODUCTION RATE

Source	f	SS	V	F	P_p (%)
R_S	2	24.872	12.436	3.90	23.2385
R_G	2	45.491	22.746	7.13	42.5034
C_S	2	30.286	15.143	4.75	28.2970
Error	2	6.381	3.191		5.9619
Total	8	107.029			100

V. Confirmation Experiment

In the Taguchi method, after the optimum level of the process parameters is determined, the final step is to verify the improvement of the performance characteristic using the optimum level of the process parameters. The confirmation experiment was conducted by using the optimum level of the process parameters. The production rate of 2502.72 gr/h was achieved in this experiment. The improvement of the S/N ratio was 5.1394 dB and the production rate was enhanced by 1.68 times in comparison to the mean value of the experimental results shown in Table II. These results confirmed the capability of the Taguchi method used for optimizing the production rate.

VI. Conclusion

The most important parameters that govern production process of nano calcium carbonate are the solution flow rate, gas flow rate, and the solution concentration. For optimization of the nano calcium carbonate production rate, the effects of process parameters were studied by using

Taguchi method. It was shown that the optimum levels of process parameters are: solution flow rate of 9 lit/min, gas flow rate of 20 lit/min, and solution concentration of 70 gr/lit. Based on the ANOVA results, it was shown that the process parameters in terms of impact significance are gas flow rate, solution concentration, and solution flow rate. By using the optimum level of the process parameters, the production rate was enhanced by 168% in comparison to the mean value of the experimental results.

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