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Operating Conditions Corresponding to Optimal Final Properties of Activated Sludge Using the DOE and RSM Techniques

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Abstract: This paper presents the influence of four relevant factors on the flocculation behavior in the activated sludge process: organic loading rate (COD), solid retention time (SRT), dissolved oxygen (DO), and calcium ion concentration, and links them to a selected set of process responses: sludge volume index (SVI), turbidity, organic removal rate (COD), and suspended solids (SS) removal. The "Design of Experiments" (DOE) and the "Response Surface Methods" (RSM) approaches are used to establish the operating conditions corresponding to optimal final properties of the activated sludge. Using these techniques, the results show that it is indeed feasible to locate the operating conditions which optimize the flocculation process and the sludge settling properties. The study represents a first attempt to evaluate the flocculation process in activated sludge using the DOE/RSM approach.

Keywords: Activated sludge, Design of Experiments (DOE), flocculation process, Response Surface Methods (RSM)

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INTRODUCTION AND REVIEW

The flocculation of activated sludge is an active physical, chemical, and biological process. Due to the complex nature of the flocs, they therefore display a wide variation in physical, chemical and biological properties (1). Further to this, the performance of the activated sludge flocculation process depends on many factors, such as shear rate, cationic concentration, temperature, dissolved oxygen concentration, organic loading, solid retention time, pH and so on. These factors thus facilitate an improvement or degradation in the ultimate process performance.

An understanding of the flocculation process and of the solid-liquid separation can be improved only by a careful analysis of the factors that govern and affect the performance of the process. During the flocculation process, the factors described above can interact and have a simultaneous influence. However, by performing experiments in a carefully controlled way, the effect of just one factor at a time may be investigated. This effect will depend on the fixed values of the other independent factors.

The optimization of operation conditions is necessary to improve the efficiency and reliability of the activated sludge process. Therefore, the effect of a set of factors on a set of responses is investigated. In this work, the influence of four relevant factors: organic loading rate (COD), solid retention time (SRT), dissolved oxygen (DO), and calcium ion concentration is evaluated on a set of selected process responses: sludge volume index (SVI), turbidity, organic removal rate (COD), and SS removal.

Solids Retention Time

The solids retention time (SRT), or sludge age in effect, represents the average period of time during which the sludge has remained in the system. SRT is the most critical parameter for activated sludge design, as SRT affects the treatment process performance, aeration tank volume, sludge production rate, and oxygen requirements.

However, since determination of the amount of biomass in the clarifier stage is difficult, it is more commonly measured as the ratio of biomass in the reactor to the rate of biomass wastage. Biomass disposal requirements are typically lower at higher sludge ages. To maintain a pilot plant at different sludge ages, a calculated volume of mixed liquor is wasted daily from the aeration unit, taking into account the mixed liquor suspended solids concentration.

Volumetric Organic Loading Rate

The volumetric organic loading rate is defined as the amount of BOD or COD applied to the aeration tank volume per day. Organic loading, expressed in kg BOD or COD/$m^3 \cdot$day, may vary from 0.3 to more than 3.0. Palm et al. (2) investigated the relationship between organic loading, dissolved oxygen concentration, and sludge settleability in the completely mixed activated sludge process. They found that a consistent relationship existed between the substrate (COD) removal rate and the aeration basin DO concentration of the completely mixed activated sludge process treating settled domestic wastewater. This relationship allows the establishment of COD removal rate/aeration basin DO concentration combinations that will prevent the development of large populations of filamentous organisms and the occurrence of bulking. These specific empirical relationships are consistent with previous general experiences.

Calcium Ions

It is generally known that divalent cations facilitate microbial flocculation by cationic bridging of extracellular polymers. Its use has been reported both for the improvement of activated sludge settling and for the formation of new biological films in biofilm reactors. Among the divalent cations, calcium has been identified as a significant factor affecting floc formation and process performance, as well as being among the most common divalent in the environment.

Bruss et al. (3) confirmed the importance of the Ca^{2+} ion for the structure of activated sludge flocs. It was investigated whether the removal of Ca^{2+} would result in disintegration or deflocculation of the sludge flocs, and, if so, whether this change in particle size distribution to smaller particles would be reflected in a decreased dewaterability. The results demonstrate a relationship between the extraction of calcium from the sludge matrix and the increase of the specific resistance to filtration, through an increase in the number of small particles. Such small particles could originate from either the sludge matrix or the liquid phase.

Sanin and Vesilind (4) demonstrated that the removal of calcium ions from the sludge floc matrix causes the sludge flocs to disintegrate, as indicated by a decrease in filterability and particle size, an increase in turbidity, and in the solution carbohydrate concentration. The results from this study also proposed that calcium ions are effective in a two-stage floc formation process. Colony formation is the initial stage of floc formation, and then, once the colonies are formed, calcium ions are further used to bridge the colonies with each other in forming final flocs.

Dissolved Oxygen

It is generally accepted that, in the full scale activated sludge process, the supply of dissolved oxygen provides a limiting factor to further increases in loading rates in the treatment facility. Oxygen deficiency is one of the main factors involved in biological dysfunction. Many studies available in the literature focus on the relationship between the DO concentration and microbial community evolution, and especially with the growth of filamentous bacteria.

It has been demonstrated sufficiently that low levels of dissolved oxygen in the aeration tank favor the growth of filamentous organisms that cause sludge bulking, which is one of the most serious operational problems encountered in activated sludge processes (5). On the contrary, a high DO activated sludge process can efficiently repress development of filamentous organisms in the aeration tank (6). Consequently, a high oxygen process shows great industrial potential for the minimization of excess sludge production as well as for improvement in the system operation.

The Design of Experiment (DOE) Method

In a planned experiment, one or more process variables are changed in order to observe the effect these changes have on one or more response variables. The DOE method is an efficient procedure for planning experiments so that the data obtained can be analyzed to yield valid and objective conclusions.

DOE begins with determining the objectives of an experiment and selecting the process factors for the study. An experimental design is the laying out of a detailed experimental plan prior to executing the experiment. Well-chosen experimental designs maximize the amount of "information" that can be obtained for a given amount of experimental effort.

The statistical theory underlying DOE generally begins with the concept of process models. It is common to begin with a process model of the "black box" type, with several discrete or continuous input factors that can be varied at will by the experimenter and one or more measured output responses. The output responses are assumed to be continuous. Experimental data are used to derive an empirical model linking the outputs and inputs. These empirical models generally contain first- and second-order terms.

Often the experiment has to account for a number of uncontrolled factors. These may be discrete factors, such as different machines or

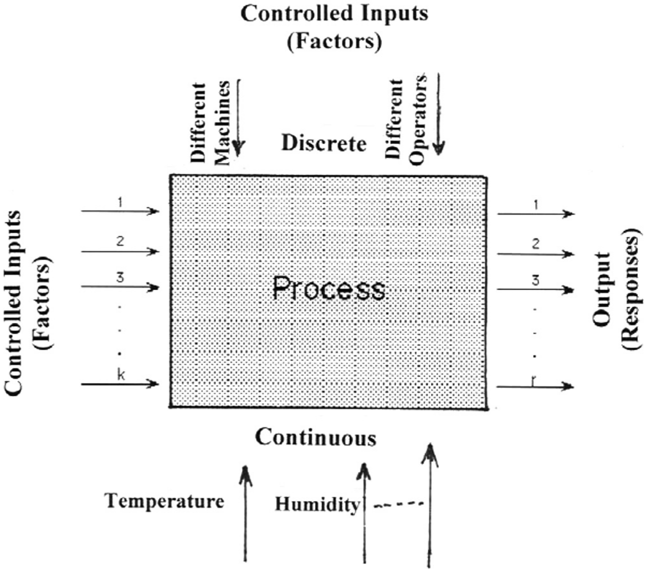

Figure 1. A 'Black Box' process model schematic.

operators, and/or they may be continuous factors such as ambient temperature or humidity. Figure 1 illustrates this situation. The most common empirical models fit to the experimental data assume either a linear or a quadratic form.

Linear model: A linear model with two factors, X_1 and X_2, can be written as

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_{12} X_1 X_2 + \text{experimental error} \tag{1}$$

Here, Y is the response for given levels of the main effects X_1 and X_2, and the X_1X_2 term is included to account for a possible interactive effect between X_1 and X_2. The constant β_0 is the response of Y when both main effects are 0.

For a more complicated example, a linear model with the three factors X_1, X_2, X_3 and one response, Y, would look like (if all possible terms were included in the model):

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X3 + \beta_{23} X_2 X_3 + \beta_{123} X_1 X_2 X_3 + \text{experimental error} \tag{2}$$

The three terms with single "X's" are the most significant terms. There are $k(k-1)/2 = 3*2/2 = 3$ two-way interaction terms and 1 three-way interaction term (which is often omitted, for simplicity). When the experimental data are analyzed, all unknown "β" parameters are estimated, and the coefficients of the "X" terms are tested to see which ones are significantly different from 0.

Quadratic model: A second-order (quadratic) model does not include the three-way interaction term, but adds three more terms to the linear model, namely:

$$\beta_{11}X_1^2 + \beta_{22}X_2^2 + \beta_{33}X_3^2 \quad (3)$$

Response Surface Methodology (RSM)

Response surface methods are used to examine the relationship between one or more response variables and a set of quantitative experimental variables or factors. These methods are often employed after you have identified a "vital few" controllable factors and you want to find the factor settings that optimize the response. Designs of this type are usually chosen when you suspect curvature in the response surface.

Response surface methods may be employed to:

- find factor settings (operating conditions) that produce the "best" response
- find factor settings that satisfy operating or process specifications
- identify new operating conditions that produce demonstrated improvement in product quality over the quality achieved by current conditions
- model a relationship between the quantitative factors and the response

Response Surface Design

Response surface models may involve just the main effects and interactions, or they may also have quadratic and possibly cubic terms to account for curvature. Under some circumstances, a model involving only the main effects and interactions may be appropriate to describe a response surface when:

1. Analysis of the results revealed no evidence of "pure quadratic" curvature in the response of interest.
2. The design matrix originally used included the limits of the factor settings available to run the process.

In other circumstances, a complete description of the process behavior might require a quadratic or cubic model:

Quadratic

$$\hat{y} = b_0 + b_1x_1 + b_2x_2 + b_3x_3 + b_{12}x_1x_2 + b_{13}x_1x_3 + b_{23}x_2x_3 + b_{11}x_1^2 + b_{22}x_2^2 + b_{33}x_3^2 \quad (4)$$

Cubic

$$\hat{y} = \text{quadratic model} + b_{123}x_1x_2x_3 + b_{112}x_1^2x_2 + b_{113}x_1^2x_3 + b_{122}x_1x_2^2 + b_{133}x_1x_3^2 + b_{223}x_2^2x_3 - b_{233}x_2x_3^2 + b_{111}x_1^3 + b_{222}x_2^3 + b_{333}x_3^3 \quad (5)$$

Objectives of Current Work

The objective of this work is to evaluate the considered responses using a full quadratic model, which is constructed from the experimental data. The interaction effects between all four factors considered in the proposed Design of Experiment (DOE) on the responses are quantified. To this end, the DOE and RSM approaches are used to optimize the operating conditions corresponding with the final properties of the activated sludge. Furthermore, identification of the set of experimental conditions that optimize the responses according to some optimization criteria is possible.

EXPERIMENTAL METHODS

The influence of physical and chemical parameters on the flocculation behavior and final property characteristics of activated sludge was investigated in an activated sludge reactor. This set up permits long-term flocculation behavior experiments, and allows control of conditions such as pH, temperature, and dissolved oxygen (DO) concentration.

Experimental Set-up

The operating procedure basically consisted of filling the reaction vessel with activated sludge from the activated sludge reactor. A five-liter, continuous-flow bench-scale reactor was used to simulate a full-scale activated sludge process. The reactor configuration is shown in Fig. 2.

The reactor consisted of a complete mixing zone and a settling zone with volumes of 5 and 1 L, respectively, separated by a slanted baffle. An aeration stone provided air and mixing to the system. The hydraulic retention time (HRT) in the aeration tank was 10 hours, and the settling time in the clarifier was 2 hours. The sludge age was 10 days. The sludge age was controlled by withdrawl of a certain volume of waste sludge. The concentration of mixed liquor suspended solids (MLSS) in the aeration tank was measured. The dissolved oxygen (DO) was measured and controlled daily by a DO meter.

Wastewater and activated sludge for mixed-liquor feed were obtained from Stoke Bardolph sewage wastewater treatment plant in Nottingham, UK. After collection, the samples were returned to the laboratory (within 1 hour) and stored at 4°C. All the samples were kept for a maximum of five days. The activated sludge in the reactor was first fed with mixed

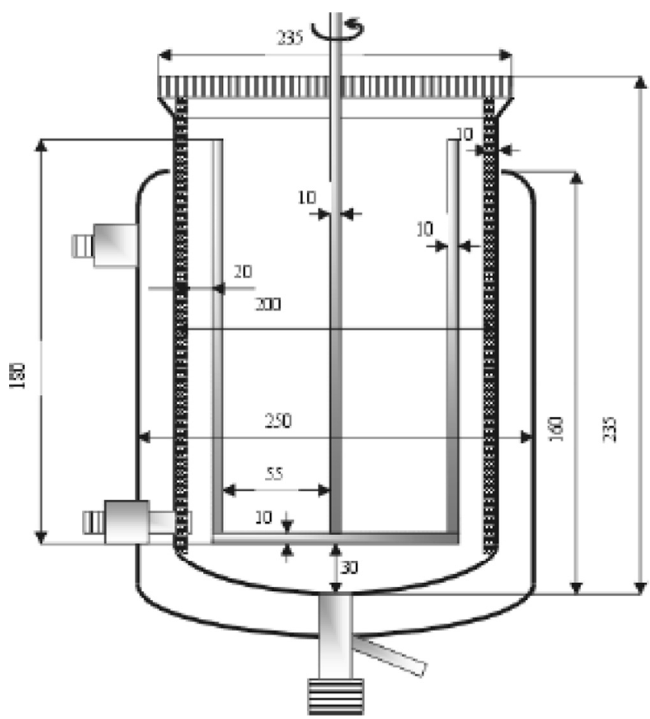

Figure 2. Flocculation vessel.

liquor from Stoke Bardolph. The activated sludge reactor was operated until steady state was achieved, typically after 20 days of operation.

The imposed experimental conditions are maintained by specific sensors. During an experiment, a certain volume of sludge is withdrawn from the reaction vessel to maintain the specific solid retention time (sludge age), dependent on the current experiment. The pH, DO, and temperature were recorded by specific sensors. Off-line analysis of the activated sludge, such as mixed liquor suspended solids (MLSS), suspended solids (SS), turbidity of the supernatant, sludge volume index (SVI), calcium ion concentration, and chemical oxygen demand (COD) were performed prior and after the designed experiment for process performance evaluation.

The activated sludge flocculation behavior was investigated in a 5 L reaction vessel (Isotherm TRGN 7392, see Fig. 2). Near the vessel wall and connected to the vessel cover, four baffles were placed to obtain homogenous mixing. The shape of the mixing blade and its dimensions are given in Fig. 2. It was specially constructed to allow a uniform mixing in the vessel and also not to interfere with the other sensors which were placed in the vessel in between the rotating mixer blades. A double wall allows control of the temperature in the vessel.

Experimental Design: Background

Traditional DOE techniques suggest a preliminary selection of a large number of independent variables (factors), to monitor and explain the behavior of a set of dependent variables (responses). The relative importance of each of these factors on the response(s) under investigation should be assessed by performing a first-order DOE, with the purpose of screening for the least important factors based on statistical indicators. This is subsequently followed by their removal from the initial set of factors, which is thus reduced to a more restricted set of more important factors. The reduction of the number of factors to be simultaneously considered in a designed experiment is a prerequisite in order to limit the required work to a reasonable number of experiments. In this study, the set of factors was restricted based on the following observations:

- An abundant literature exists in which the effect of a number of particular and well-known factors is discussed and acknowledged in their influence on the dynamics of the flocculation process. Still, sometimes, their role in the flocculation process is subject to controversy.
- The selected factors are among those that are easily controllable in a laboratory environment.

- The number of experiments to be performed, including those of the screening phase, must be kept to a minimum; this constraint is dictated by the necessity of having a similar sludge for the entire duration of the experiment.

With these remarks in mind, the four most important factors to be considered in the experimental design are:

- DO concentration (DO)
- Calcium addition (Ca)
- Solid Retention Time (SRT or sludge age)
- Chemical Oxygen Demand (COD) loading rate

The following variables were considered as responses:

1. Suspended Solids of supernatant (SS)
2. Sludge Volume Index (SVI)
3. Turbidity of the supernatant
4. Chemical Oxygen Demand (COD) removal rate

DOE Considerations: Working Methodology and Experimental Matrix

Design Properties, Response Models, and Experimental Effort

The aim is to build empirical models of the set of considered responses and eventually use it for predicting optimal operating conditions. This requires at least a second-order model, which means an experiment in which each factor takes at least three levels. An important attribute of a DOE that allows unambiguous determination of the response model coefficients is the orthogonality of the design. This guarantees a non-singular design matrix and, furthermore, the model coefficients have minimal variance.

Therefore, the number of experiments that is needed to perform the design is $2^n + 2n + k$, where n is the number of factors, k is the number of centre point experiments, 2^n is the factorial number of experiments and 2n is the axial number of experiments. An important (statistical) aspect concerns randomization of the experiments, which minimizes the influence of nuisance variables, ensures a uniform noise level, etc. The experiments have accounted for all these facts, but also had to consider the setup limitations. An important condition for the overall quality of the study is the stability of sludge samples properties. A statistical software (MINITAB) has been used in this research to create the matrix

Table 1. Central Composite Design for experiments created by MINITAB

Factors	4
Base runs	31
Base blocks	1
Level factorial	Full factorial
Cube points	32
Centre points in cube	14
Axial points	16
Centre points in axial	0
Alpha	2
Replicates	2
Total runs	62
Total blocks	1

design. A total of 62 experiments with 2 replicates has been run. The detailed result of the Central Composite Design created by MINITAB statistic software is presented in Table 1.

In order to minimize the time-confounding effect, the experiments were performed in the optimal order, as resulting after minimization of the correlation between the time effect and the main effects of the factors. The experimental design matrix in terms of coded variables was then defined together with the chronological order of the experiments.

Range Selection for the Different Factors

Dissolved Oxygen. A dissolved oxygen (DO) concentration of 1 to 2 mg/l is sufficient for aerobic activated sludge treatment. In this study, the dissolved oxygen concentration limits have been set between anaerobic conditions (DO = 0 mg/l) and an aerobic level at DO = 4 mg/l.

Calcium concentration:

A consensus exists between all mechanisms available, and the beneficial effect of calcium in flocculation is well recognized. The calcium concentrations considered in the design were calculated by taking into account the available studies (7), which demonstrate that a sodium to divalent cation ratio larger than approximately 2 would produce a deterioration of settling properties. By considering these studies, the central point of the design was decided to be between a lowest Ca^{2+}(0 mM) and a highest Ca^{2+}(20 mM). Calcium was added as $CaCl_2 \cdot 2H_20$ dissolved into 50 ml filtered effluent.

Solid retention time (SRT):

It is clear that SRT is a primary factor determining the performance of activated sludge systems. Selection of biological retention time (θ_c), to provide a particular soluble COD removal efficiency, can now be accompanied by a semi-quantitative prediction of the settling characteristics, which will exist at that θ_c. Perhaps even more importantly, the relationship between settling properties and θ_c can predict the regions in which little or no solids separation will occur. Based on total biomass in the effluent, the best overall solids removal occurred at the values of θ_c in the range from 4 to 9 days (8). Temperature and other factors also affect SRT in various treatment applications of activated sludge processes. The SRT applied in these experiments was varied from 2 to 10 days.

Chemical oxygen demand loading rate:

The volumetric organic loading rate is defined as the amount of BOD or COD applied to the aeration tank volume per day. Organic loading, expressed in kg BOD or COD/$m^3 \cdot$day, may vary from 0.3 to more than 3.0. Higher volumetric loadings generally result in higher required oxygen transfer rates per unit volume for the aeration system. Wastewater and activated sludge for mixed-liquor feed were obtained from Stoke Bardolph sewage wastewater treatment plant in Nottingham, UK. The COD concentration was varied from 150 to 300 mg/L, depending on the season. The COD loading rate applied in these experiments was varied from 0.1 to 0.2 kg COD/$m^3 \cdot$day.

Regression and Analysis of Variance (ANOVA)

The 62 design matrix experiments were conducted to test the effect of operational conditions on the flocculation behavior and final properties of activated sludge. The design of experiments was based on the DOE and RSM method. The results were analyzed by MINITAB (statistical software) and interpreted by regression and analysis of variance (ANOVA). The factors and responses were coded from X1 to X8. Table 2 shows the full experimental results from the design matrix, with 2 replications in terms of un-coded variables created by the MINITAB statistical software.

The coded values are presented as follows:

X1: COD loading rate [mg/l $\cdot$ day]
X2: SRT [day]
X3: DO [mg/l]
X4: Ca [mM]

Table 2. Experimental results

Standard order	Run order	COD loading rate (X_1) mg/l·day	SRT Sludge age (X_2) day	DO (X_3) mg/l	Ca (X_4) mM	COD Outlet mg/l	COD Removal rate (X_5) mg/l·day	SS Outlet (X_6) mg/l	SVI (X_7) ml/g	Turbidity (X_8) NTU
30	1	150	6	2	10	37	113	19	107	13
56	2	150	6	2	10	38	112	18	112	12
57	3	150	6	2	10	36	114	18	108	14
41	4	175	4	1	15	44	131	21	93	12
27	5	150	6	2	10	35	115	17	108	13
14	6	175	4	3	15	41	134	19	95	13
54	7	150	6	2	0	36	114	27	121	19
16	8	175	8	3	15	36	139	22	90	16
29	9	150	6	2	10	34	116	17	102	14
55	10	150	6	2	20	34	116	16	89	22
25	11	150	6	2	10	36	114	17	108	14
1	12	125	4	1	5	31	94	21	104	16
18	13	200	6	2	10	52	148	22	115	15
26	14	150	6	2	10	37	113	19	112	16
7	15	125	8	3	5	28	97	27	107	13
58	16	150	6	2	10	35	115	19	106	15
19	17	150	2	2	10	38	112	22	142	20
31	18	150	6	2	10	38	112	21	111	16
36	19	125	4	3	5	34	91	23	103	18

(*Continued*)

Table 2. Continued

Standard order	Run order	COD loading rate (X_1) mg/l·day	SRT Sludge age (X_2) day	DO (X_3) mg/l	Ca (X_4) mM	COD Outlet mg/l	COD Removal rate (X_5) mg/l·day	SS Outlet (X_6) mg/l	SVI (X_7) ml/g	Turbidity (X_8) NTU
24	20	150	6	2	20	36	114	15	86	21
52	21	150	6	0	10	52	98	24	118	17
59	22	150	6	2	10	34	116	20	106	16
11	23	125	8	1	15	31	94	19	93	14
22	24	150	6	4	10	36	114	17	106	11
13	25	125	4	3	15	31	94	19	94	14
50	26	150	2	2	10	42	108	20	137	19
9	27	125	4	1	15	34	91	22	96	16
32	28	125	4	1	5	30	95	20	104	17
28	29	150	6	2	10	39	111	18	112	15
40	30	125	4	1	15	35	90	24	97	17
15	31	125	8	3	15	30	95	21	91	12
8	32	175	8	3	5	38	137	25	106	14
60	33	150	6	2	10	40	110	19	112	15
51	34	150	10	2	10	30	120	25	120	10
5	35	125	4	3	5	33	92	24	98	17
38	36	125	8	3	5	30	95	25	107	11
43	37	175	8	1	15	40	135	20	93	18
62	38	150	6	2	10	36	114	17	107	14
12	39	175	8	1	15	41	134	22	92	17
3	40	125	8	1	5	29	96	20	103	15

37	41	175	4	3	5	46	129	24	101	19
53	42	150	6	4	10	33	117	15	100	12
35	43	175	8	1	5	40	135	26	101	15
42	44	125	8	1	15	32	93	20	96	13
23	45	150	6	2	0	38	112	28	127	20
44	46	125	4	3	15	29	96	21	93	15
17	47	100	6	2	10	34	66	18	96	10
61	48	150	6	2	10	36	114	18	112	15
48	49	100	6	2	10	32	68	16	102	11
39	50	175	8	3	5	36	139	26	100	13
4	51	175	8	1	5	38	137	27	101	16
49	52	200	6	2	10	50	150	24	109	16
34	53	125	8	1	5	28	97	20	108	16
33	54	175	4	1	5	44	131	22	106	17
6	55	175	4	3	5	48	127	25	101	18
21	56	200	6	0	10	56	144	25	124	15
46	57	125	8	3	15	28	97	20	88	11
20	58	150	10	2	10	28	122	21	114	9
10	59	175	4	1	15	45	130	23	92	11
45	60	175	4	3	15	42	133	20	97	12
2	61	175	4	1	5	43	132	23	111	18
47	62	175	8	3	15	38	137	21	92	15

X5: COD removal rate [mg/l · day]
X6: SS of supernatant [mg/l]
X7: SVI [ml/g]
X8: Turbidity of supernatant [NTU]

Interpreting the Experimental Results

The results were analysed by MINITAB (statistical software) and interpreted by regression and analysis of variance (ANOVA). Several of Minitab's ANOVA procedures, however, allow models with both qualitative and quantitative variables.

The experimental results presented in this paper are given in terms of statistical analysis (residual) and surface/contour plots. The two input factors with the most significant effect on each response were selected by random combination of these factors through surface/contour plots. The model in this research work has more than two input factors, but the factor(s) not selected in the plot are held constant. The values held by the remaining factors can be specified.

Residual plots are used to examine the quality of model fit in regression and ANOVA analysis. Examining residual plots helps to determine if the ordinary least squares assumptions are being met. If the assumptions are satisfied, then ordinary least squares regression will produce unbiased coefficient estimates with the minimum variance. MINATAB provides the following residual plots, which are presented in this work:

Normal probability plot of residuals. The points in this plot should generally form a straight line if the residuals are normally distributed. If the points on the plot depart from a straight line, the normality assumption may be invalid.

Histogram of the residuals. An exploratory tool to show the general characteristics of the residuals, including typical values, spread, and shape. A long tail on one side may indicate a skewed distribution. If one or two bars are far from the others, those points may be outliers.

Residuals versus fitted values. This plot should show a random pattern of residuals on both sides of zero. If a point lies far from the majority of points, it may be an outlier. There should not be any recognizable patterns in the residual plot. For instance, if the spread of residual values tends to increase as the fitted values increase, then this may violate the constant variance assumption.

Residuals versus order of data. This is a plot of all residuals in the order that the data was collected and can be used to find non-random error, especially of time-related effects. This plot help to check the assumption that the residuals are uncorrelated with each other.

Surface/Contour plots. A surface plot provides a three-dimensional view, which may provide a clearer picture of the response surface. A contour plot provides a two-dimensional view, where all points that have the same response are connected to produce contour lines of constant responses.

RESULTS AND DISCUSSION

Operational Conditions Corresponding to Organic Removal Rate

Statistical Analysis

The ANOVA statistical analysis and the model regression quality are not presented. However, the relatively large F-ratio = 239.66, associated with a small P-value = 0.000, indicates that there is a good relation between the set of variables and the response. In addition, the P-value <0.05, the model is assumed to explain a significant amount of the variation present in the data and the model is statistically significant.

However, for the interaction, the small F-ratio of 2.22 associated with a relatively large P-value of 0.058 indicates that the model does not entirely explain a significant interaction fraction of the variation in the response. Moreover, the lack-of-fit error had an F-ratio of 8.25, associated with the P-value of 0.000, indicating that lack-of-fit is significant with a probability of 95%.

The corresponding regression coefficient R^2 was 0.986, which is very close to the value of a perfect match. The statistical significance of the model is indirectly suggested by the adjusted regression coefficient, which penalizes the model complexity. This has a value, $R^2_{adj} = 0.982$ which is again a very good result, close to the ideal value of 1. Both values indicate that the model fits the data well.

Residual and Contour/Surface Plots

The residual plots for COD removal rate are shown in Fig. 3. As seen in Fig. 3, the histogram indicates that an outlier may exist in the data, shown by the one bar on the far left side of the plot. The normal probability plot shows an approximately linear pattern, consistent with a normal distribution. There is one point in the lower left corner of the plot which may be an outlier. The plot of residuals versus the fitted values shows a random pattern of residuals on both side of zero. The residuals are very close to the reference line, except one-point which lies far from

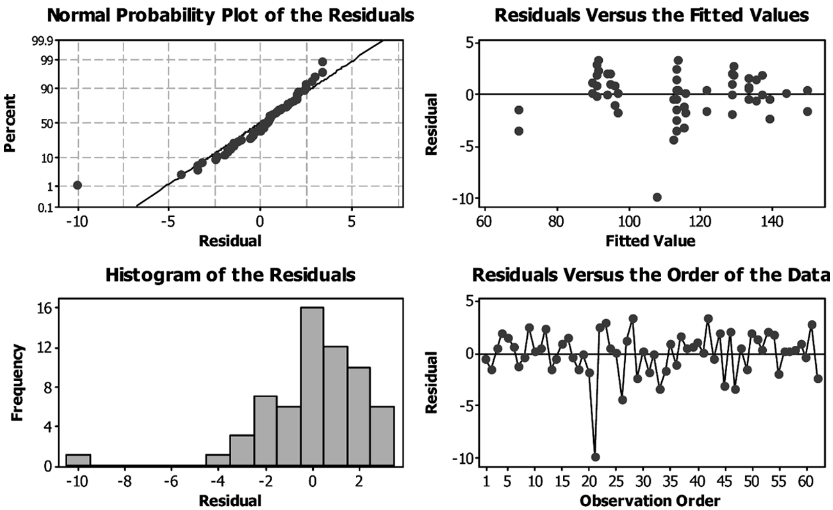

Figure 3. Residual plots for COD removal rate.

the majority of the points; it may be an outlier. This plot indicates that the residuals have constant variance. The plot of residuals versus the order of the data shows a positive correlation, indicated by a clustering of residuals with the same sign except points 21 and 26.

The results of residual plots for COD removal rate do not indicate any problems with the model. The full quadratic model with the p-value for lack-of-fit is 0.000, suggesting that this model fits the data extremely well. A plot of the response as a function of COD loading rate and DO concentration is shown in Fig. 4. It shows the 3D plot and the contour

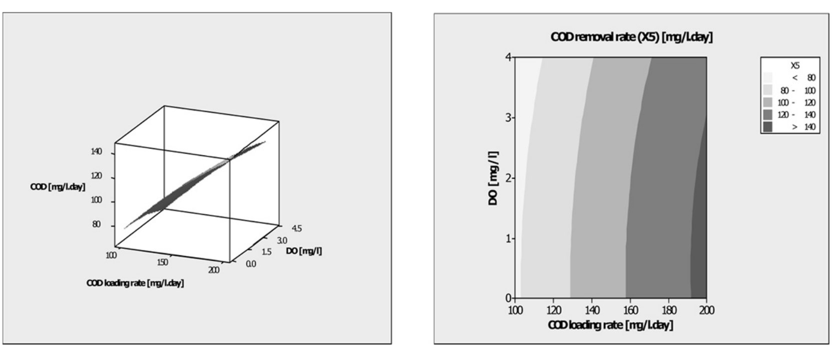

Figure 4. Contour/surface plots for COD removal rate.

plot, while the remaining factors have been fixed at the values corresponding to the constrained optimum.

In fact, the projection of the response in the (COD, DO) space behaves as a near-stationary rising ridge surface, for which the sensitivity of the response with COD loading variation is much more pronounced when compared with that of the DO. Therefore, by changing the COD loading rate a little, the effect of the DO may be fully compensated. Accordingly, the model showed that the activated sludge sample treated during the flocculation process experiments has a more effective COD removal rate for a high COD loading rate and a high DO concentration.

Operational Conditions Corresponding to Total Suspended Solid of Supernatant

Statistical Analysis

The large F-ratio of 8.01 associated with the small P-value of 0.000 indicates that there is a relationship between the set of variables and the response. In addition, with a P-value <0.05, the model is statistically significant.

However, for the interaction, the small F-ratio of 2.53 associated with a relatively large P-value of 0.033 indicates that the model does not entirely explain a significant interaction fraction of the variation in the response. Moreover, the lack-of-fit error had an F-ratio of 8.69, associated with the P-value of 0.000, indicating that lack-of-fit is significant with a probability of 95%. The regression coefficient was calculated to be $R^2 = 0.705$ which can be considered a satisfactory fit when accounting for the possible off-line sampling or experimental errors.

Residual and Contour/Surface Plots

The residual plots for suspended solids of supernatant are shown in Fig. 5. As seen in Fig. 5, the histogram indicates no outlier exists in the data. The normal probability plot shows an approximately linear pattern, consistent with a normal distribution. The plot of residuals versus the fitted values shows a random pattern of residuals on both side of zero. The residuals get smaller (closer to the reference line) as fitted values increase, which may indicate the residuals have non-constant variance. The plot of residuals versus the order of the data shows a positive correlation, which is indicated by a clustering of residuals with the same sign except points 20, 21, and 42. The full quadratic model with a p-value for lack-of-fit is 0.000, suggesting that this model fits the data extremely well.

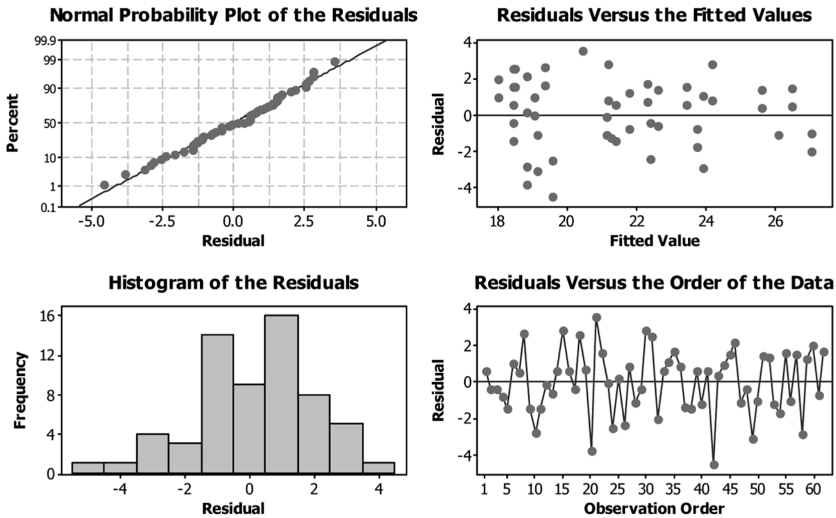

Figure 5. Residual plots for total suspended solid of supernatant.

An example of the response as a function of SRT and Ca addition is given in Fig. 6. It shows a 3D plot and a contour plot, while fixing the other factors at the values corresponding to the constrained optimum (COD loading rate = 150 mg/l · day; DO = 2 mg/l). It is observed in Fig. 6 (right) that a decrease in the response value, corresponding to increases in the SRT and to increases in Ca addition, leads to a minimum or stationary point in the fitted surface. Accordingly, the model shows that the activated sludge sample treated during the experiments has

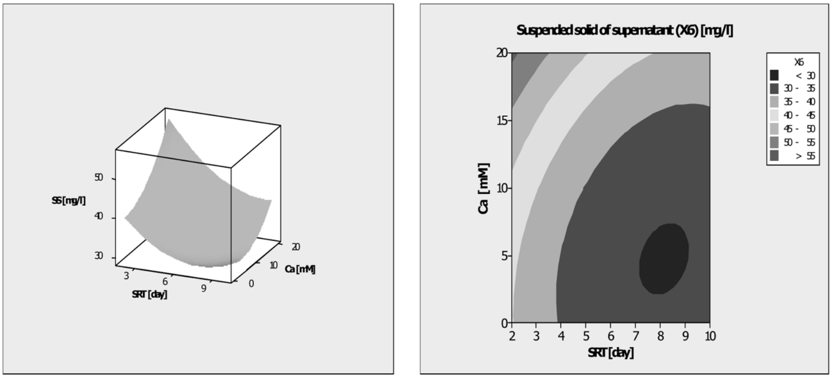

Figure 6. Contour/surface plots for total suspended solid of supernatant.

optimum total suspended solids content in the supernatant for a moderate calcium addition and a high SRT.

Operational Conditions Corresponding to Sludge Volume Index

Statistical Analysis

The large F-ratio of 3.91 associated with a small P-value of 0.000 indicates that there is a relation between the set of variables and the response. In addition, with a P-value <0.05, the model is assumed to explain a significant amount of the variation present in the data.

However, for the interaction, the relatively small F-ratio of 0.38 associated with a large P-value of 0.89 indicates that the model does not entirely explain a significant interaction fraction of the variation in the response. Moreover, the lack-of-fit error had an F-ratio of 34.73 associated with a P-value of 0.000, indicating that lack-of-fit is significant with a probability of 95%.

The regression coefficient was calculated to be $R^2 = 0.538$ which indicates a rather modest fit. The adjusted regression coefficient, which penalizes the model for unnecessary terms, showed a low value of 0.401. This indicates that there might be model terms that do not really contribute to the response values.

Residual and Contour/Surface Plots

The residual plots for sludge volume index (SVI) are shown in Fig. 7. As seen in Fig. 7, the histogram indicates that no outlier exists in the data. The normal probability plot shows an approximately linear pattern consistent with a normal distribution. The plot of residuals versus the fitted values shows a random pattern of residuals on both sides of zero. The residuals get larger (further to the reference line) as the fitted values increase, which may indicate the residuals have non-constant variance. The plot of residuals versus the order of the data shows a positive correlation, as indicated by a clustering of residuals with the same sign except points 17, 45, and 56.

The results of residual plots for sludge volume index (SVI) do not indicate any problems with the model. The full quadratic model has a p-value for lack-of-fit of 0.000, suggesting that this model fits the data extremely well.

Figure 8 shows a 3D plot and a contour plot, while fixing the other factors at the values corresponding to the constrained optimum

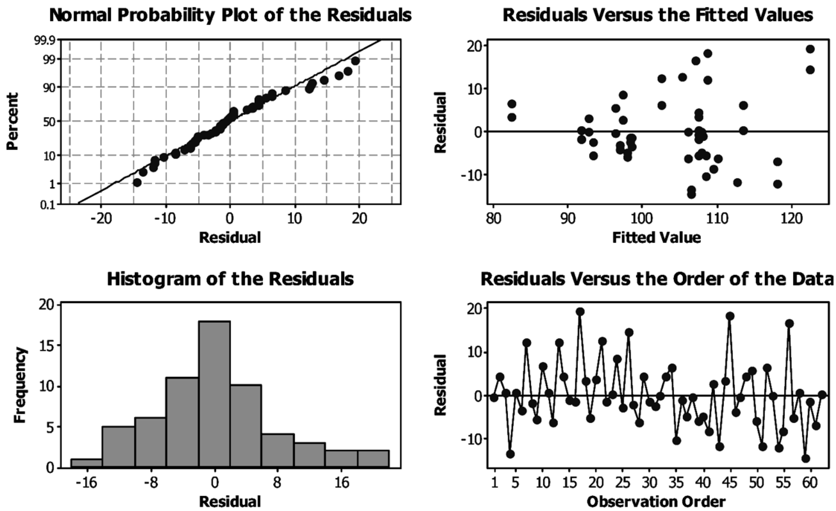

Figure 7. Residual plots for sludge volume index (SVI).

(SRT = 6 day; DO = 2 mg/l). It is observed in Fig. 8 (right) that a maximum and a stationary point in the fitted surface follow an increase of the response value corresponding to an increase in the COD loading rate and an increase in Ca addition. Accordingly, the model shows that the activated sludge sample treated during the experiments has an optimum settling for a moderate calcium addition and a high COD loading rate.

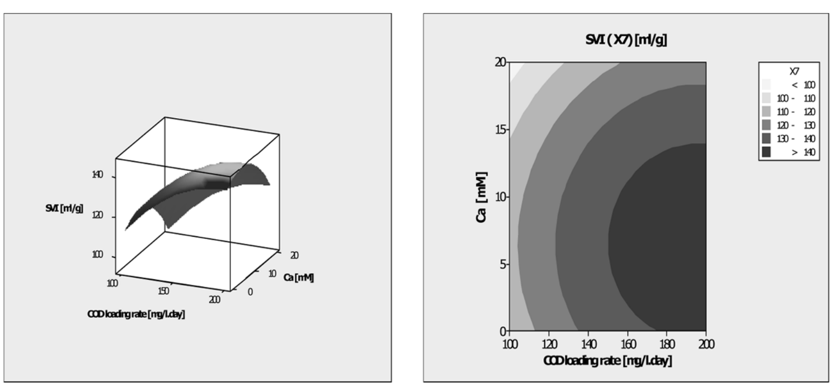

Figure 8. Contour/surface plots for sludge volume index (SVI).

Operational Conditions Corresponding to Turbidity of Supernatant

Statistical Analysis

The large F-ratio of 8.22 associated with a small P-value of 0.000 indicates that there is a relationship between the set of variables and the response. In addition, with a P-value <0.05, the model is statistically significant.

In addition, for the interaction, the relatively large F-ratio of 4.18 associated with a small P-value of 0.002 also indicates that there is an interactive relationship within the set of variables and the response. Moreover, the lack-of-fit error had an F-ratio of 11.18, associated with the P-value of 0.000, indicating that lack-of-fit is significant with a probability of 95%. The regression coefficient was calculated to be $R^2 = 0.710$ which can be considered a satisfactory fit when accounting for the possible off-line sampling or experimental errors.

Residual and Contour/Surface Plots

The residual plots for turbidity of supernatant are shown in Fig. 9. The histogram indicates that an outlier may exist in the data, shown by the one point on the far left side of the plot. The normal probability plot shows an approximately linear pattern, consistent with a normal distribution.

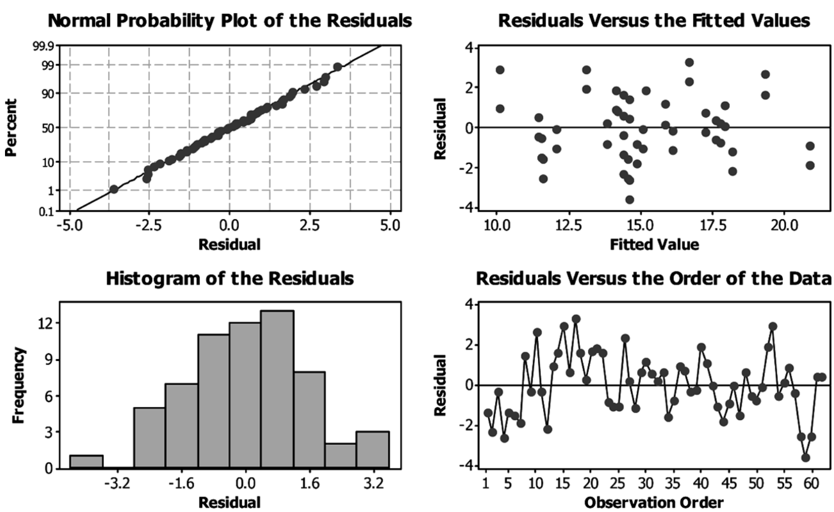

Figure 9. Residual plots for turbidity of supernatant.

The plot of residuals versus the fitted values shows a random pattern of residuals on both side of zero. The residuals are very close to the reference line, which may indicate that the residuals have constant variance. The plot of residuals versus the order of the data shows a positive correlation, indicated by a clustering of residuals with the same sign except points 15, 17, 53, and 59. The results of residual plots for turbidity of supernatant do not indicate any problems with the model. The full quadratic model has a p-value for lack-of-fit of 0.000, suggesting that this model fits the data extremely well.

The turbidity values are often linked to a series of other factors, such as: dissolved oxygen concentration (9,10), average velocity gradient (11), and cation addition (4).

An example of a fitted surface by using the optimal values is given in Fig. 10. It shows a 3D plot and the contour plot, while fixing the other factors at the values corresponding to the constrained optimum (COD loading rate = 150 mg/l · day; DO = 2 mg/l). It is observed in Fig. 10 (right) that, at low Ca addition, the response value shows a decrease corresponding to an increase in the Ca addition and to an increase in SRT, followed at a higher Ca addition by a minimum and a stationary point in the fitted surface. Increasing values of Ca addition (>10 mM) then have a negative (increasing) effect on the turbidity of the supernatant. Accordingly, the model shows that the activated sludge sample treated during the flocculation process experiments has an optimum turbidity of the supernatant for a moderate calcium addition and a high SRT.

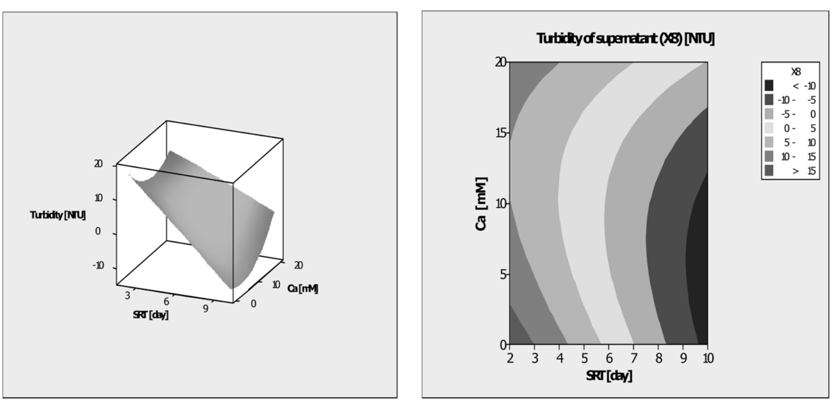

Figure 10. Contour/surface plots for turbidity of supernatant.

CONCLUSIONS

The activated sludge process flocculation performance was evaluated by using DOE and RSM. The effects of four factors, i.e. COD loading rate, solid retention time, dissolved oxygen concentration, and calcium addition on different process responses, i.e. COD removal rate, SVI, turbidity, and TSS, were investigated by fitting the full quadratic model to the obtained experimental data. Good models were obtained for the process responses, except moderately good quality statistical model predictions were found in the process response of SVI. A very good accuracy was found for the RSM which described the COD removal rate. The model predictions for the COD removal rate were also found to agree with the observations performed on the influence of each factor on the COD removal rate. The COD loading rate and the DO concentration proved to be the strongest factors affecting COD removal rate. This papers showed that it was possible to find the operating conditions which optimise the flocculation process and sludge settling properties. In particular, optimum TSS, SVI, and Turbidity are promoted by moderate calcium concentrations. However, these results still need to be validated experimentally for other activated sludge samples before the observed trends can be generalized.

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