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Flocculant testing through the application of experimental design techniques and the application of a flocculant pipe reactor

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Abstract

This work presents the experience with the application of a flocculent pipe reactor for flocculant evaluation and selection at Langer Heinrich Mine. Other processes could give different results. Parameters considered in determining the conditions that generate the optimum solids settling rate and overflow clarity are: slurry density, reaction time, turbulent shear rate (slurry flow rate), flocculent make-up concentration, flocculant dosing rate and type of flocculant.

Two polyacrylamide flocculants used in the process were tested: Alclar 665 and Magna 333.

The data was generated and processed in accordance with the response surface methodology. This methodology renders a mathematical model from which the optimum for each parameter in combination with the other parameters can be determined. The distinctive feature of this method of conducting settling tests is the ability to verify the interaction between parameters and higher order effects of parameters.

1 Introduction

The testing of flocculant in the laboratory is an important topic when thickeners and flocculant for a process are specified. Flocculation of minerals is a function of the mineral particle and process solution properties (Atesok et al., 1988; Ntshabele et al., 2008). In most operations, these properties do not fluctuate significantly but could drift over time. The challenge is to identify and select the most suited flocculant for the specific process.

At Langer Heinrich Mine (LHM) it was found that the laboratory data could not always be used for flocculant selection and confirmation of the results in the plant is required before a change of flocculant could be implemented. The data obtained from the traditional batch and static cylinder settling testing need careful interpretation when flocculant types and doses are compared. The interpretation of batch cylinder static tests has been studied, discussed and published extensively (Concha and Burger, 2003; Weir and Moody, 2003).

Recent studies indicated that the rate of flocculation can be expressed as a function of the local turbulent shear rate, particle concentration and the flocculant concentration (Heath and Koh, 2003).

The thickener feedwell design and flocculant dosing point(s) are equipment parameters that can be assumed to be constant. The thickener feed volume is a process parameter that should normally stay within an operating window. These stable parameters result in a local turbulent shear rate window and contact time that is specific to the plant.

The flocculant concentration and flocculant dosing rate are the parameters that can be influenced by the plant operator. The plant operator is faced with the challenge of optimising these parameters. It is costly to establish the optimum settings on plant scale, therefore, LHM decided to develop a procedure of establishing the best plant settings in the laboratory more reliably. This goal was approached through a literature review of the most recent developments in testing equipment as well as data interpretation tools.

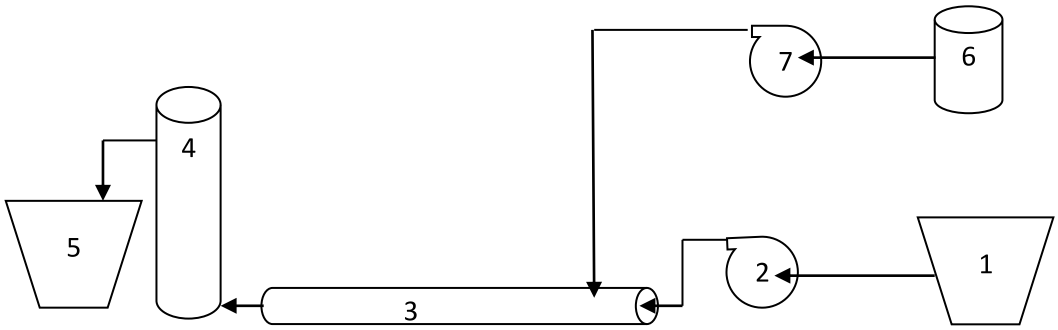
A flocs generator reactor (FGR) (Carissimi and Rubio, 2005) and linear pipe reactor (LPR) (Owen et al., 2008) was developed to study flocculation reactions. The development of these reactors enabled the control of flocculation parameters one at a time. The influence of several parameters was thus studied and reported (Owen et al., 2008).

For experimental design and data interpretation the application of response surface methodology (RSM) enabled the study of parameter effect and parameter interdependence (Rigas et al., 2000; Carley et al., 2004; Bradley, 2007). Experimental design based on RSM can optimise the number of experimental runs. Data from a well designed experiment can also be utilised for parameter screening. The focus can then be put on the most significant parameters, parameters that interact to enhance or reduce effects, nonlinear parameters and parameters with higher order effect. Commercially available software packages reduce the computational effort significantly.

In this study the influence and interaction of slurry solids concentration, flocculent dosing rate and contact time at constant shear rate were studied for two different flocculants.

2 Methodology

Two variable speed peristaltic pumps were used to control the flow rates. The slurry feed container was a baffled vessel fitted with a stirrer. A schematic diagram of the experimental setup is shown in Figure 1.



1. Stirred slurry feed container
2. Feed pump
3. LPR
4. Settling tube
5. Overflow container
6. Flocculant container
7. Flocculant dosing pump

Figure 1 Representation of the experimental flow diagram

A three level full factorial experimental design was used. The coded experimental design is given in Table 1. For ease of reading the runs are presented in systematic order.

The experimental parameter values were set by selecting a mid point for each parameter and then deviating from this set point by a step change up and one down. The size of the step change for each parameter was selected during the experimental design. These values were then codified in accordance with published procedures (Carley et al., 2004; Bradley, 2007).

As an example, the flocculant concentration midpoint was selected as 0.07%. The step change was selected as 0.03, therefore, the flocculant concentrations used for the experimental runs was 0.1%, 0.07% and 0.04%.

Stock solutions (0.1%, 0.07% and 0.04%) of the flocculants were prepared by the slow addition of the required mass (1 g, 0.7 g and 0.4 g respectively) of flocculant powder to 1 L of rapidly stirred tap water. This solution was then left to hydrate for two hours. The working solution of flocculant was prepared from this stock solution by ten times dilution.

The feed slurry was prepared by diluting a sample from the pre-leach thickener underflow to the required concentration.

The slurry feed pump was kept at a constant flow for all the experimental runs. The flocculant dosing pump was set to the required set point. The slurry feed pump was then started and operated until the slurry reached the settling tube. The flocculant dosing pump was then started and operated until the settling tube was filled with flocculated slurry. The pumps were then stopped and the solids level in the settling tube recorded over time and a supernatant sample collected for total suspended solids (TSS) determination. The TSS was determined at the site laboratory as non-routine samples, according to plant methodology and procedures.

The settling rate and TSS in the overflow were determined for each run, 27 runs were completed for each flocculant type.

Table 1 The coded experimental design with the settling rate and TSS achieved

Run Number	Solids Concentration	Flocculant Dosing Rate	Reaction Time	Settling Rate	TSS	Settling Rate	TSS
				Magna 333		Alclar 665	
1	1	1	1	32.9	128	18.0	40
2	1	1	0	27.1	234	11.4	0
3	1	1	-1	36.6	300	6.3	90
4	1	0	1	35.1	132	39.7	16
5	1	0	0	26.3	334	30.8	78
6	1	0	-1	32.9	178	25.3	100
7	1	-1	1	1.0	86	6.1	118
8	1	-1	0	2.8	116	4.2	64
9	1	-1	-1	2.1	146	4.7	74
10	0	1	1	12.2	122	30.3	200
11	0	1	0	18.1	138	27.1	194
12	0	1	-1	18.9	214	1.1	240
13	0	0	1	40.1	110	3.3	162
14	0	0	0	49.1	188	3.7	148
15	0	0	-1	36.5	274	4.1	122
16	0	-1	1	55.1	158	1.4	136
17	0	-1	0	23.4	188	17.3	134
18	0	-1	-1	31.0	182	2.9	100
19	-1	1	1	31.3	128	49.8	210
20	-1	1	0	31.9	234	45.2	184
21	-1	1	-1	33.1	321	39.6	175
22	-1	0	1	51.6	132	40.2	114
23	-1	0	0	37.1	334	40.5	78
24	-1	0	-1	38.1	345	19.1	18
25	-1	-1	1	28.6	86	9.5	288
26	-1	-1	0	18.4	116	8.1	108
27	-1	-1	-1	8.3	214	7.6	140

3 Results

The experimental settings and results were processed using the statistical program JMP™. Some of the available results and statistics are given in tables and as figures below.

3.1 Magna 333 settling rate parameter screening

The parameter screening is done as a first step. The screening results are given as Figure 2. Generally only terms with an individual p value less than 0.05 would be used in the prediction model.

Contrasts

Term	Contrast		Lenth t-Ratio	Individual p-Value	Simultaneous p-Value
Floc Dosing (g/t)	3.26986		1.53	0.1307	0.9293
Slurry Conc	-3.08318		-1.44	0.1506	0.9612
Reaction Time	2.29693		1.07	0.2744	0.9996
Floc Dosing (g/t)*Floc Dosing (g/t)	-6.75004	*	-3.15	0.0101*	0.1561
Floc Dosing (g/t)*Slurry Conc	0.81657	*	0.38	0.7194	1.0000
Slurry Conc*Slurry Conc	-3.05945	*	-1.43	0.1534	0.9648
Floc Dosing (g/t)*Reaction Time	-3.08150	*	-1.44	0.1508	0.9616
Slurry Conc*Reaction Time	-1.83252	*	-0.86	0.3772	1.0000
Reaction Time*Reaction Time	1.49418	*	0.70	0.4766	1.0000
Floc Dosing (g/t)*Floc Dosing (g/t)*Floc Dosing (g/t)	3.44882	*	1.61	0.1131	0.8889
Floc Dosing (g/t)*Floc Dosing (g/t)*Slurry Conc	2.07318	*	0.97	0.3193	1.0000
Floc Dosing (g/t)*Slurry Conc*Slurry Conc	8.09908	*	3.79	0.0034*	0.0655
Floc Dosing (g/t)*Floc Dosing (g/t)*Reaction Time	-0.17277	*	-0.08	0.9405	1.0000
Floc Dosing (g/t)*Slurry Conc*Reaction Time	0.96768	*	0.45	0.6719	1.0000
Slurry Conc*Slurry Conc*Reaction Time	-0.69176	*	-0.32	0.7613	1.0000
Floc Dosing (g/t)*Reaction Time*Reaction Time	-0.81342	*	-0.38	0.7206	1.0000
Slurry Conc*Reaction Time*Reaction Time	0.30110	*	0.14	0.8952	1.0000
Null19	-1.51155		-0.71	0.4698	1.0000
Null20	1.01468		0.47	0.6562	1.0000
Null21	-1.25530		-0.59	0.5806	1.0000
Null22	1.68266		0.79	0.4168	1.0000
Null23	-1.25742		-0.59	0.5800	1.0000
Null24	-1.35856		-0.63	0.5484	1.0000
Null25	-0.27187		-0.13	0.9067	1.0000
Null26	-2.53161		-1.18	0.2284	0.9973
Null27	-0.39337		-0.18	0.8635	1.0000

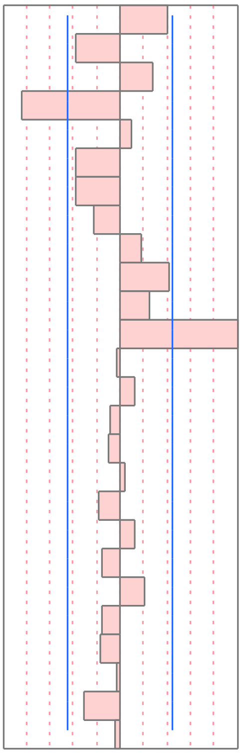


Figure 2 The screening as done in JMP

3.2 The expression describing the settling rate response with Magna 333

Some terms with higher p values during screening were included in this model. A t-test probability for each term is given in Table 2. The inclusion of the additional terms resulted in four terms with t-test estimates less than 0.05 in the model, as given in Table 2. These terms include the two terms predicted during parameter screening.

The estimate of the regression coefficient for each term in the polynomial model is presented in Table 2. From these estimates the prediction estimate model can be constructed.

This model with the chosen terms resulted in a good fit as the R^2 of 0.84 and the adjusted R^2 of 0.76 given in Table 3 indicate. The adjusted R^2 is lower than the R^2 because of the four terms in the model that have little effect on the settling rate as indicated by the t-test probability that is larger than 0.05. The aim of more iteration would be the reduction in difference between the R^2 and adjusted R^2.

The analysis of variance is given in Table 4. The degrees of freedom (DF) for regression model and unexplained error or residual error after the fitting of the model, as well as the total degrees of freedom, are given in the first column. The sum of squares (SS) column accounts for the variability measured in the response. It is the sum of squares of the differences between the fitted response and the actual response.

As the JMP software explanation states: "Note that large values of Model SS as compared with small values of Error SS lead to large F-ratios and low p values, which is what you want if the goal is to declare that terms in the model are significantly different from zero. Most practitioners check this F-test first and make sure that it is significant before delving further into the details of the fit."

Table 2 Regression coefficient estimates for expression of settling rate with Magna 333

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	52.563697	81.63657	0.64	0.5278
Floc dosing (g/t)	-0.242622	1.319931	-0.18	0.8562
Slurry conc (11.4, 19.5)	-4.561329	1.667799	-2.73	0.0136*
Reaction time	1.1139475	0.651957	1.71	0.1047
(Floc dosing (g/t)-60.5989)*(floc dosing (g/t)-60.5989)	-0.023253	0.004224	-5.50	<.0001*
Slurry conc*slurry conc	-6.496295	3.944699	-1.65	0.1169
(Floc dosing (g/t)-60.5989)*(reaction time-5.04222)	-0.07085	0.030725	-2.31	0.0332*
(Floc dosing (g/t)-60.5989)*(floc dosing (g/t)-60.5989)*(floc dosing (g/t)-60.5989)	-0.000499	0.002016	-0.25	0.8073
(Floc dosing (g/t)-60.5989)*slurry conc*slurry conc	1.0263011	0.170653	6.01	<.0001*

Table 3 Summary of fit for expression of settling rate with Magna 333

Term	Value
R^2	0.836126
R^2 adjusted	0.763293
Root mean square error	6.975034
Mean of response	28.13333
Observations	27

Table 4 Analysis of variance for expression of settling rate with Magna 333

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	8	4,468.1402	558.518	11.4801
Error	18	875.7198	48.651	Prob > F
C. Total	26	5,343.8600		<.0001*

3.2.1 Prediction profiling of settling rate with Magna 333

The effect of changing a specific parameter can be evaluated through the prediction profiling. The curve that describes the parameter effect is shown in Figure 3. The flocculant dosing and slurry concentration curves are nonlinear and the reaction time curve remains linear over the operating envelope studied.

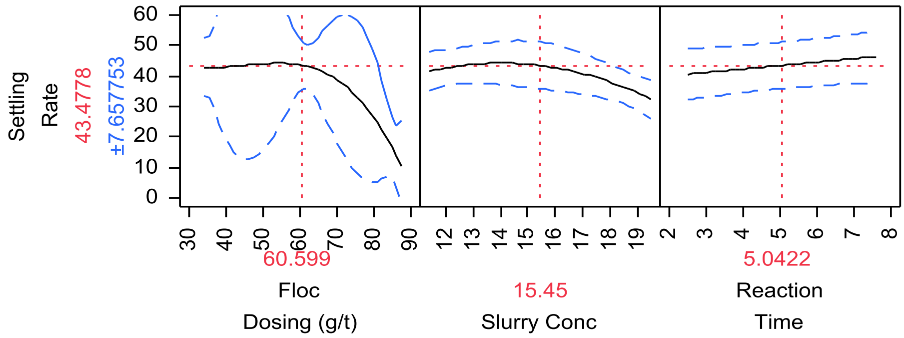


Figure 3 Prediction profiler for settling rate response to parameter changes when Magna 333 is used

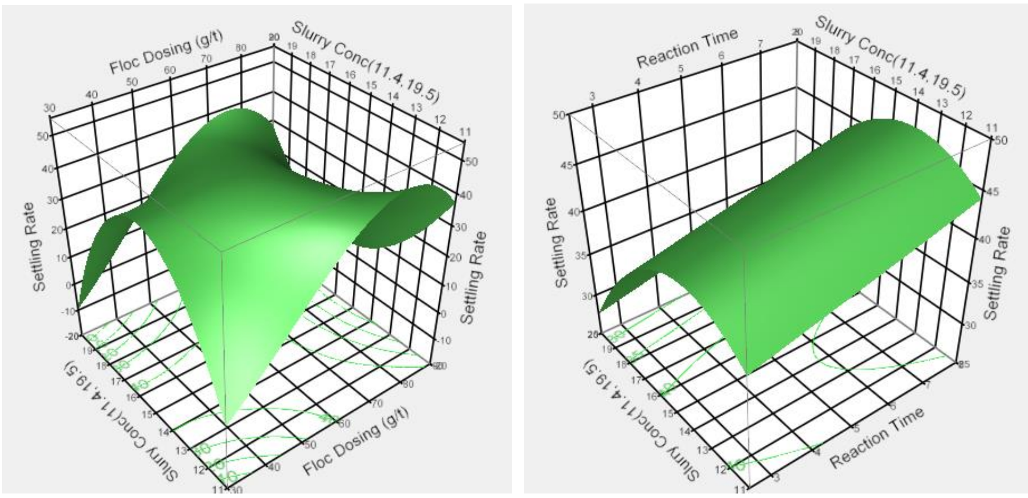


Figure 4 The surface area describing the settling rate response to parameter combinations when Magna 333 is used

The response surface areas describing the settling rate response to the parameter combinations are presented in Figure 4.

The interaction, or not, between any two parameters investigated can be predicted through the interaction profiles. Two orthogonal views of the profile curves are given. Interaction between two parameters can be expected when the profile curves are not parallel or crossover. When two parameters do not interact the profile curves will be parallel to each other from both views; as can be seen in Figure 5 in the blocks presenting the combination of slurry concentration and reaction time.

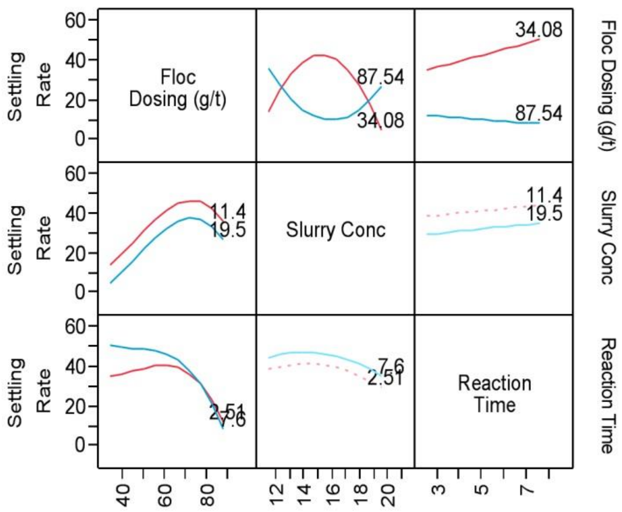


Figure 5 Parameter interaction profiles for settling rate prediction when Magna 333 is used

3.3 Prediction of the TSS value when using Magna 333

The regression coefficients of the prediction expression for the TSS value are given in Table 7. Inspection of the t-test probabilities reveals that two terms have results larger than 0.05. The R^2 value of 0.68 and adjusted R^2 value of 0.61 presented in Table 6 indicates that the model predicts the TSS value only moderately accurately. The large SS and relative small F ratio given in Table 6 confirm this observation.

Table 5 Summary of fit for expression of TSS with Magna 333

Term	Value
R^2	0.683015
R^2 adjusted	0.607542
Root mean square error	50.76647
Mean of response	190.2963
Observations	27

Table 6 Analysis of variance for expression of TSS with Magna 333

Source	DF	Sum of Squares	Mean Square	F Ratio
Model	5	116,617.70	23,323.5	9.0498
Error	21	54,121.93	2,577.2	Prob > F
C. Total	26	170,739.63		0.0001*

Table 7 Regression coefficient estimates for expression of TSS with Magna 333

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	398.19682	64.55661	6.17	<.0001*
Reaction time	-24.0903	4.745141	-5.08	<.0001*
Floc dosing (g/t)	-0.828468	0.935993	-0.89	0.3861
Slurry conc (11.4, 19.5)	-16.82567	11.72082	-1.44	0.1659
(Floc dosing (g/t)-60.5989)*(floc dosing (g/t)-60.5989)	-0.077733	0.030696	-2.53	0.0194*
(Floc dosing (g/t)-60.5989)*slurry conc*slurry conc	2.7584185	1.134543	2.43	0.0241*

3.3.1 Prediction profiling of TSS with Magna 333

The prediction profiler as shown in Figure 6 indicates a non-linear response from the flocculant dosing. An increase in reaction time and slurry concentration would result in a decrease in TSS.

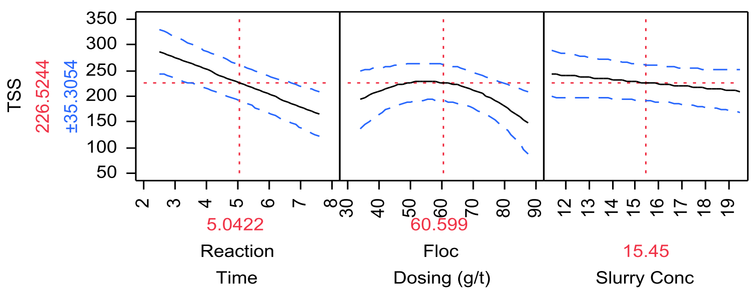


Figure 6 The prediction profiler for TSS response to parameter changes when Magna 33 is used

The parameter interaction prediction, as presented in Figure 7, indicates that the flocculant dosing and slurry concentration interact. No interaction between the reaction time and flocculant dosing or between reaction time and slurry concentration is expected.

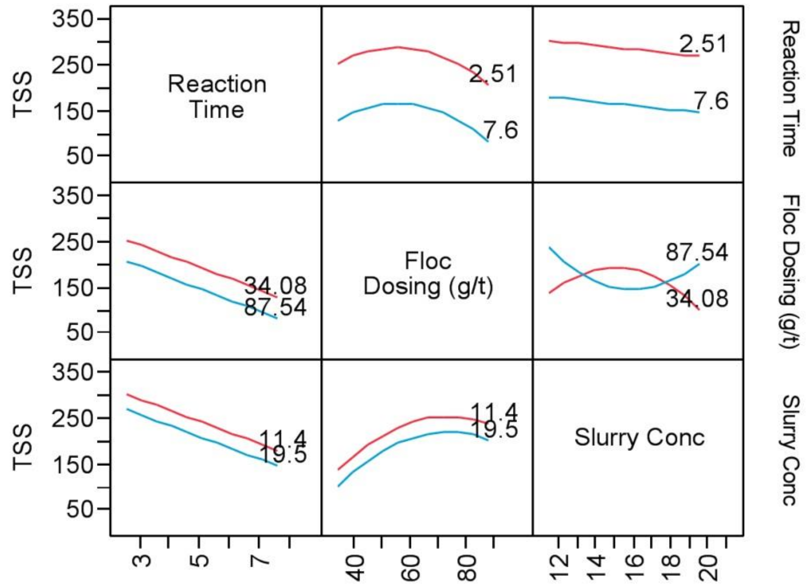


Figure 7 Interaction profiles of parameters for TSS prediction when using Magna 333

The surfaces describing the TSS response to parameter changes when Magna 333 is used are presented in Figure 8. As expected, the surface describing the interaction between flocculant dosing and slurry concentration complex with a saddle, clearly present.

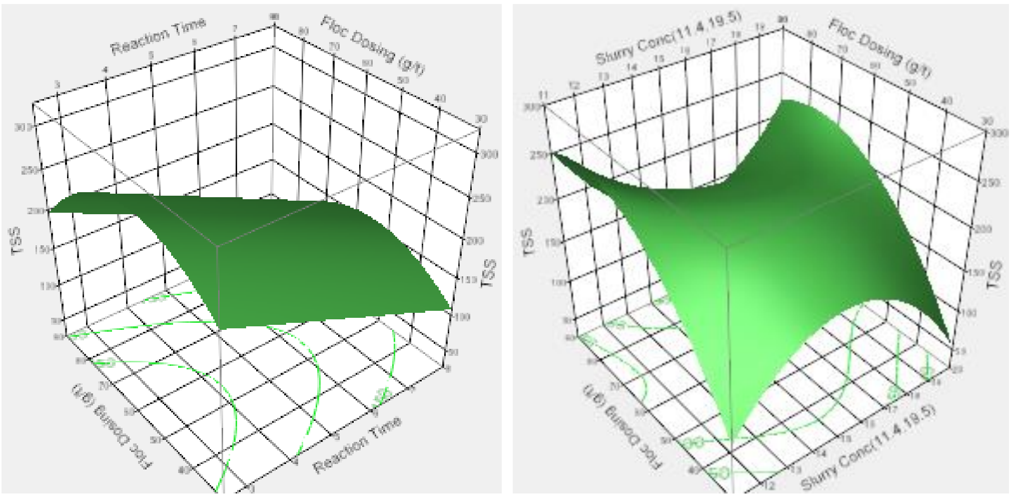


Figure 8 The surface area describing the TSS response to parameters combinations when Magna 333 is used

3.4 The expression describing the settling rate response with Alclar 665

The testing of Alclar 665 flocculant resulted in an expression which describes the settling rate response well. Inspection of Table 8 reveal R^2 and adjusted R^2 values of 0.81 and 0.74 respectively. The regression coefficients given in Table 9 and the associated t-test probabilities indicate that at least seven terms are required for the prediction, with one term not required. These terms include two second order terms and two third order terms. The square of the slurry concentration term has the biggest impact, as indicated by the regression coefficient of 19.5, which is more than three times the second biggest coefficient of 5.8, which is associated with the flocculant dosing.

Table 8 Summary of fit for expression of settling rate with Alclar 665

Description	Value
R^2	0.811218
R^2 adjusted	0.741667
Root mean square error	8.040445
Mean of response	18.41852
Observations	27

Table 9 Regression coefficient estimates for expression of settling rate with Alclar 665

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-357.2947	97.51785	-3.66	0.0017*
Floc dosing	5.8251337	1.580306	3.69	0.0016*
Slurry conc (11.4, 19.5)	0.7310986	3.30569	0.22	0.8273
Reaction time	1.9308978	0.751541	2.57	0.0188*
(Floc dosing-60.5989)*slurry conc	-0.338953	0.088589	-3.83	0.0011*
Slurry conc*slurry conc	19.532118	4.539464	4.30	0.0004*
(Floc dosing-60.5989)*(floc dosing-60.5989)*(floc dosing-60.5989)	-0.008039	0.002333	-3.45	0.0027*
(Floc dosing-60.5989)*(floc dosing-60.5989)*slurry conc	-0.017197	0.006161	-2.79	0.0116*

3.4.1 Prediction profiler of settling rate with Alclar 665

The prediction profiler as presented in Figure 9, indicates significant nonlinear settling rate response to flocculant dosing and slurry concentration. The reaction time response is linear.

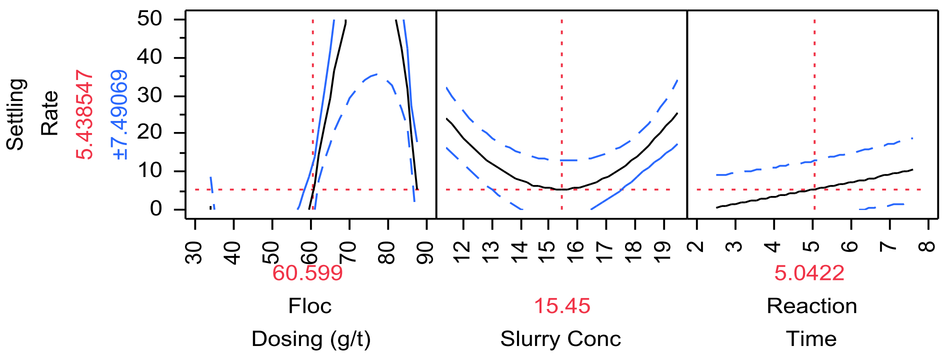


Figure 9 The prediction profiler for settling rate response to parameter changes when Alclar 665 is used

In Figure 10 the prediction of interaction between flocculant dosing and slurry concentration is clear. Other interactions are not clear because of the extent of reaction to changes.

The settling rate response surfaces when Alclar 665 is used are presented in Figure 11.

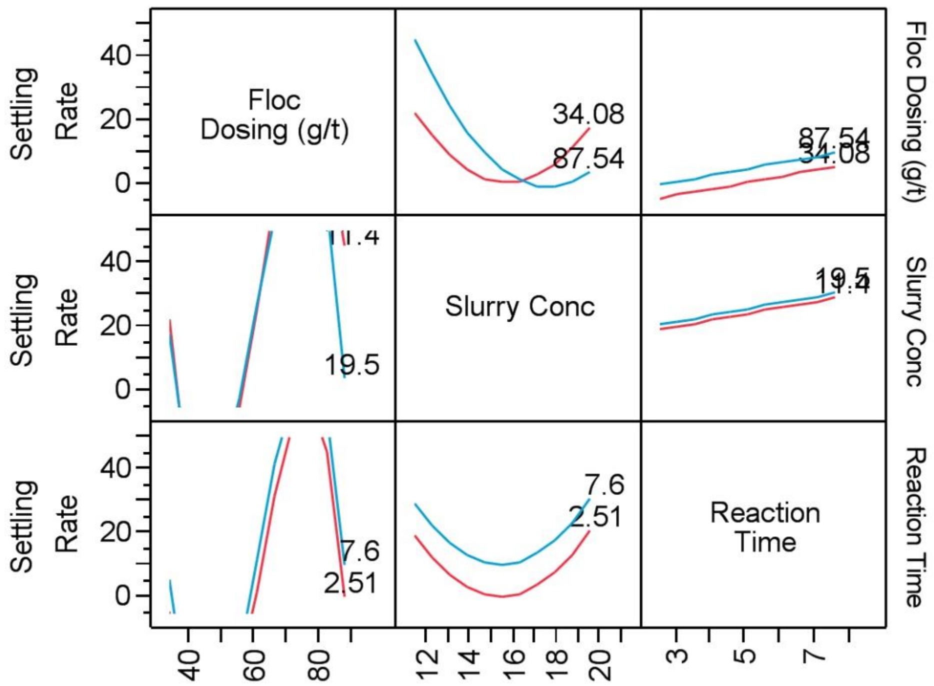


Figure 10 The interaction profiles of parameters for settling rate prediction when Alclar 665 is used

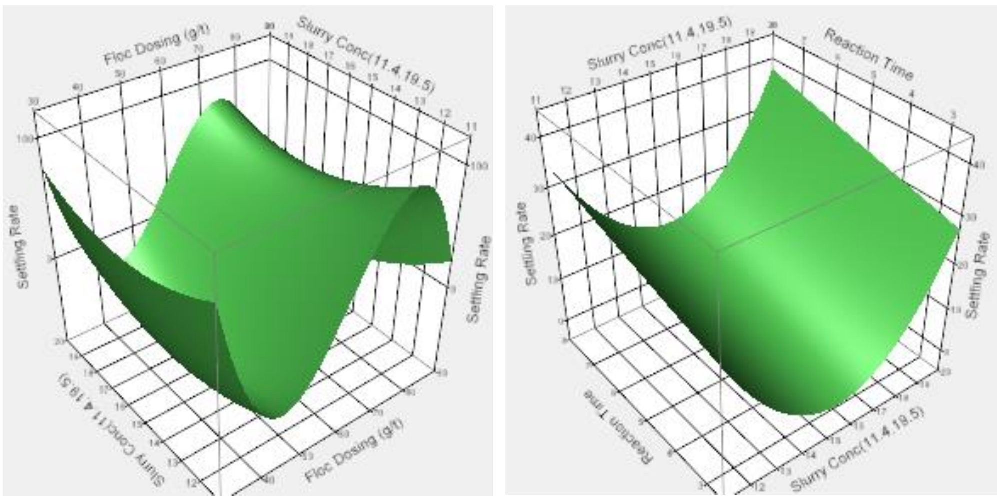


Figure 11 The surface area describing the settling rate response to parameter combinations when Alclar 665 is used

3.5 Prediction of the TSS value when using Alclar 665

The testing of Alclar 665 flocculant resulted in an expression which describes the TSS response well. Inspection of Table 10 reveals R^2 and adjusted R^2 values of 0.81 and 0.73 respectively. The regression coefficients given in Table 11 and the associated t-test probabilities indicate that at least six terms are required for the prediction, with one term not required. These terms include three second order terms and two third order terms. The square of the slurry concentration term has the biggest impact as indicated by the regression coefficient of -82.4, which is almost more than seven times the slurry concentration and reaction time interaction coefficient, which is second biggest coefficient at -12.1. It should be noted that the slurry concentration occurs in both of the two largest expression terms.

Table 10 Summary of fit for expression of TSS with Alclar 665

Term	Value
R^2	0.815696
R^2 adjusted	0.733783
Root mean square error	35.84733
Mean of response	123.3704
Observations	27

Table 11 Regression coefficient estimates for expression of TSS with Alclar 665

Term	Estimate	Std Error	t Ratio	Prob>|t|
Intercept	-9.943593	46.8545	-0.21	0.8343
Slurry conc (11.4, 19.5)	-0.934646	14.42356	-0.06	0.9490
Slurry conc*slurry conc	-82.44287	17.16253	-4.80	0.0001*
Slurry conc*(reaction time-5.04222)	-12.0903	4.013084	-3.01	0.0075*
(Floc dosing-60.5989)*(floc dosing-60.5989)	0.0780309	0.021841	3.57	0.0022*
Slurry conc*slurry conc*(floc dosing-60.5989)	-2.543273	0.801416	-3.17	0.0053*
Slurry conc*(floc dosing-60.5989)*(floc dosing-60.5989)	-0.086581	0.025559	-3.39	0.0033*
Floc dosing	2.1384276	0.661525	3.23	0.0046*
Reaction time	6.3561237	3.380968	1.88	0.0764

3.5.1 Prediction profiler of TSS with Alclar 665

The prediction profiler as presented in Figure 12 indicates nonlinear settling rate response to flocculant dosing and slurry concentration. The reaction time response is linear.

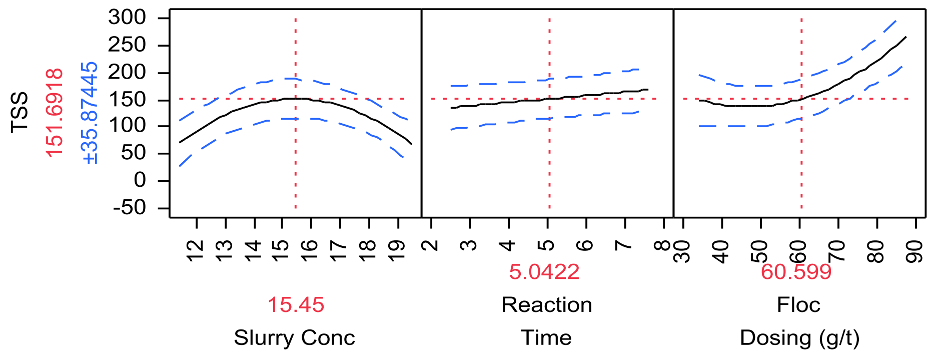


Figure 12 The prediction profiler for TSS response to parameter changes when Alclar 665 is used

The parameter interaction prediction as presented in Figure 13 indicates that the flocculant dosing and slurry concentration, as well as slurry concentration and reaction time, interact. No interaction between the reaction time and flocculant dosing is expected.

The TSS response surfaces when Alclar 665 is used are presented in Figure 14.

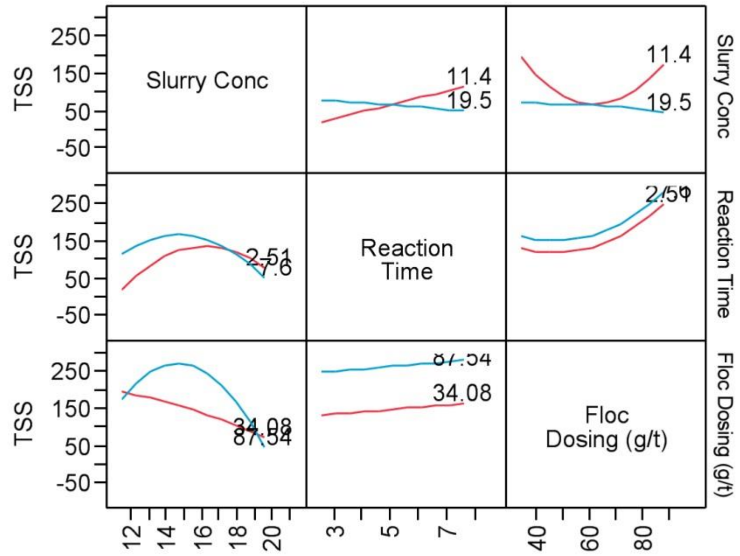


Figure 13 The prediction profiler for TSS response to parameter changes when Alclar 665 is used

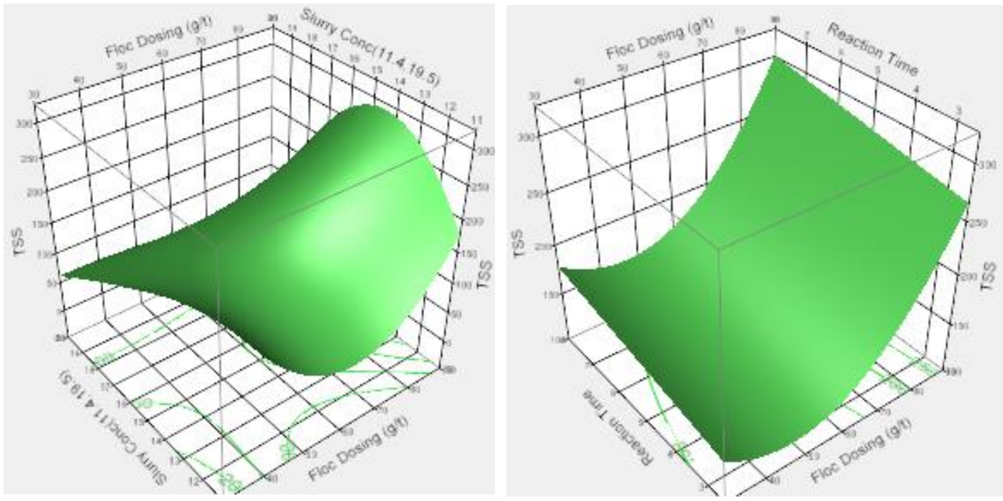


Figure 14 The surface area describing the TSS response to parameter combinations when Alclar 665 is used

4 Conclusions

The prediction expressions compiled for settling rates when using either one of the two flocculants are able to predict the actual values reasonably accurately. These expressions could be refined through iteration, but for the purpose of flocculant selection, the information is sufficient.

4.1 Selection for settling rate

The expression required to predict the Magna 333 flocculant settling rate has four significant terms of which two are second order terms and one is third order. The slurry concentration is the most significant parameter influencing the settling rate. The more dilute the slurry, the faster the settling. Care must be taken not to overdose the Magna 333 flocculant as this would reduce the settling rate.

The expression required to predict the Alclar 665 flocculant settling rate has seven significant terms of which two are second order, and two are third order. The Alclar 665 flocculant responded more vigorously to parameter changes when compared to Magna 333. It would be more challenging to keep the process at conditions where the Alclar 665 flocculant would perform optimally.

This result would explain the complaints from operations personnel that the 'flocculant may perform better in the laboratory, but it does not work in the plant'.

4.2 Selection for overflow clarity

The prediction of overflow clarity or the inverse, the TSS could only be done to a fair extent when using Magna 333 flocculant. This could be an indication that parameters that were not tested have a significant influence on the flocculant performance.

The prediction of the TSS when Alclar 665 is used could be accurately done. The expression has six terms of which three are second order terms, and two are third order.

A comparison of the flocculant performance based on overflow clarity testing would be more subjective. The higher order terms in the Alclar 665 prediction could be an indication of a flocculant that could cause fluctuations in TSS on relatively small process changes.

4.3 Flocculant selection

Based on the test work done, the Magna 333 flocculant would give a more robust settling performance in the Langer Heinrich process.

The experimental results for flocculant selection based on overflow clarity are not conclusive.

Experience on production scale is in line with the experimental findings; Magna 333 flocculant is easier to use because the thickeners would perform in a more predictable manner, but the overflow TSS would be lower when Alclar 665 is used.

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