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Integrated optimization of a waste water treatment plant using statistical analysis

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ABSTRACT

In this research, a waste water treatment plant is systematically optimized. The waste water treatment plant is used to remove aluminium from waste water using precipitation, flocculation and flotation. In total 40 variables influence the combined unit. After systematic selection, the number of variables was reduced to six: the waste water flow, pH, agitation velocity, amount of poly-electrolyte, amount of dissolved air and aluminium concentration. For these variables an experimental design was set up and executed and the results were analyzed by means of ANOVA. With the results of the ANOVA, an empirical model was constructed. The model was used for maximization of the aluminium removal. Subsequently, validation experiments were performed to confirm the findings. The study showed that the amount of poly-electrolyte is a key factor for combined unit operation.

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1. Introduction

FUJIFILM Manufacturing Europe B.V. produces and distributes photographic materials. At the Tilburg site in the Netherlands photo paper and offset plates are produced and innovative R&D projects are accomplished.

The offset plates are used for printing of e.g. newspapers, magazines and advertising. During the manufacturing process of the offset plate, waste water containing aluminium is produced. The characteristics of the waste water feed stream are mentioned in Table 1. The aluminium is separated from the waste water, using a waste water treatment facility. The waste water is treated with chemicals to precipitate aluminium as aluminium hydroxide [1,2]. The aluminium hydroxide is separated from the waste water using flocculants and flotation techniques [3–5]. The effluent is normally discharged to the drain if the amount of aluminium and aluminium hydroxide is $\leq$20 mg/l, based on Dutch government legislations obtained by the water board "Brabantse Delta" permit no; 08U007801.

Precipitation, flocculation and flotation are common technologies. However, by combining these technologies, the process becomes increasingly difficult to control; variables from the processes will interact and influence the amount of separated aluminium denoted as the % removal.

The objective of this research is to identify which variables and settings to select, for optimal operation of the waste water treatment facility. In this paper we introduce a systematic method describing the optimization approach of the combined unit operation. Several articles were published concerning optimization problems and approaches [6–9]. However, mainly all the articles focus on experimental design and statistical analysis or experimental design and empirical modeling to find the optimum variable settings for solving the optimization problem. This research uses the combination of experimental design, statistical analysis and empirical modeling, leading to an effective solution of the optimization problem. A new approach is used where the experimental design is constructed in such a way that with half the number of experiments, the optimization problem can be solved. This approach is very powerful, as the variables and interaction variables of the total unit operation can be analyzed independently. This will give a clear insight into how the processes are really working. In Fig. 1 the solution method is schematically depicted.

At the initial stage, all potential variables that influence the % removal are identified on the basis of a literature study and supporting experiments. These variables are categorized into controllable and uncontrollable factors. The controllable factors are the factors that can be manipulated.

At the second stage, an experimental design is set up. Five controllable factors are evaluated. The controllable factors are varied at a high and low level based on the minimal and maximal operational settings of the process. As the control factors are varied at two levels, 2^5 experimental runs are required. A new approach of experimental design is described in this article, which gives addi-

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Nomenclature

K	sum of squares
S	variance
F	value of F-test
C	contrast
X	experimental value
e	residual
y	% removal
n	number of experiment repetitions
x	average

Greek letters

ϕ	degree of freedom
σ	standard deviation

Table 1
Waste water characteristics.

Characteristics	Boundary	
	Low	High
Temperature (°C)	15.3	22.3
Al^{3+} (mg/l)	100	800
NO_3^- (mg/l)	1229	2446
SO_4^- (mg/l)	41	549
pH	4	4.6
Conductivity (25 °C S/m)	20.5	54.8

tional information of the uncontrollable factors without need for any extra experiments. The collected data are used in an analysis of variance (ANOVA) to compute which factors and which combinations of factors significantly influence the % removal.

At the third stage, an empirical model is constructed using the outcomes of the experimental design. The empirical model is then used for calculation of the optimal operational settings of the control factors. Combining empirical modeling and statistical analysis results in optimal settings for the control factors, i.e. maximizing the % removal within the lowest economic impact and within the limits of the government legislation. Ultimately, validation experiments are performed to test whether the optimized settings actually will give the predicted results.

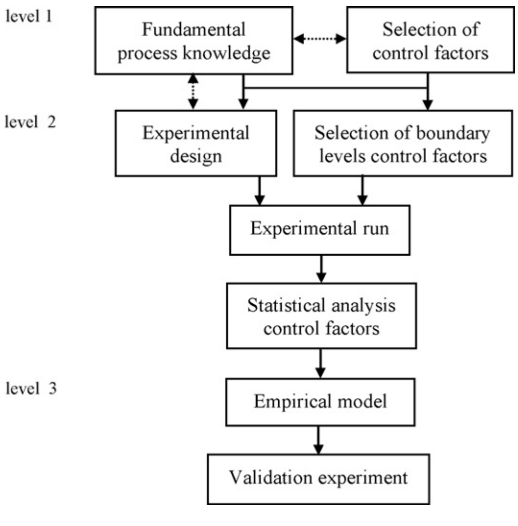

Fig. 1. Hierarchical optimization structure.

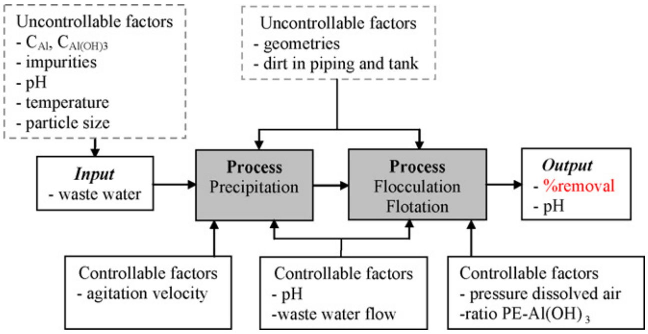

Fig. 2. Model waste water treatment plant.

2. Theory

2.1. Precipitation, flotation and flocculation

At the waste water treatment plant, aluminium and caustic soda are added in a continuous stirred tank reactor (CSTR), where under the influence of mixing, the aluminium precipitates towards aluminium hydroxide. Uniform mixing of aluminium and caustic soda is very important for an even particle size distribution. The final particle size of aluminium hydroxide is very small <10 μm [1,2,4,10–14].

Flotation is used for aggregation of the particles. Aluminium hydroxide particles in water have a negative surface charge at a pH value below the iso-electric-point (i.e.p.) and their surface charges are the same. This will result in repulsive forces between the particles and because of their surface charge, the particles are resistant to aggregation. There is a balance between the repulsion of the particles to each other, resulting in a stable suspension [4,15–17]. Flocculation is the process in which the particle suspension is destabilized as a result of charge neutralization of the electric forces. At the waste water treatment plant, flocculation of aluminium hydroxide particles takes place in a coiled pipe flocculator (CPF). A counter ionic poly-electrolyte is used for charge neutralization of the aluminium hydroxide particles, which is added in the CPF [4,5,15–20].

Finally, flotation is used for separation of the particles from the waste water. Dissolved air is added in the CPF and as a result of pressure difference small bubbles are formed. The air bubbles will be entrapped within the flake structure which are distributed by the flow to a flotation tank. In the flotation tank the flake will rise to the liquid surface were a froth layer is formed. This froth layer is removed mechanically by a skimmer and the clean effluent is disposed to the drain [20–22].

2.2. Variables total unit operation

From literature [1–5,10–15,18–22] over 40 variables that influence the efficiency of the separation process can be identified. Plant and laboratory experiments can be used to test the factors independently. These key variables that influence the efficiency of the separation process are categorized in controllable and uncontrollable factors. The uncontrollable factors are grouped into fixed and disturbance factors. The fixed factors are related to the geometries of the precipitation, flocculation and flotation unit. These factors can be adjusted, but it can result in expensive modifications. The disturbance factors cannot be adjusted and in general their value is also not known. The controllable factors are the process variables and are used for optimization of the % removal.

Fig. 2 shows all factors important in the waste water treatment plant. This figure is also according to the process flow diagram. After reduction, five controllable factors and eight uncontrollable

Table 2
Factors and levels.

Factor	Symbol	Levels	
		Low (−1)	High (+1)
Flow waste water (m^3/h)	A	30	55
pH effluent	B	7	8.5
Agitation velocity (rpm)	C	1000	1450
Ratio $Al(OH)_3$-PE	D	18[a] 25[b]	28[a] 38[b]
Pressure dissolved air (bar)	E	4.5	6

[a] Old unit.
[b] New unit.

factors remain. From the 8 uncontrollable factors, the geometries of the precipitation unit, flocculation unit and flotation unit are fixed. These factors will influence the output, but are time invariant. The temperature, the aluminium and aluminium hydroxide concentration, the amount of impurities, the particle size of aluminium hydroxide, the pH of the influent and dirt, are real disturbance factors.

2.3. Design of experiments and ANOVA

To test which of the factors are significantly influencing the % removal, an experimental design is set up. The outcomes of the experimental are analyzed by the Analysis of Variance method (ANOVA). This method is used to compare the magnitude of the effects of factors with the magnitude of experimental error. If the magnitude of a factor effect is large in comparison to the experimental error, the changes in the response are considered to be the effects of the influencing factors. The factors that are responsible for producing a variation in the response are called significant [23]; which indicates that they have a significant influence in the % removal.

2.3.1. Experimental design

Five factors are varied at two levels denoted as high (+1) and low (−1). In Table 2, the factors A, B, C, D, E are denoted, respectively as the waste water flow, the pH after the precipitation process, the agitation velocity, the amount of poly-electrolyte e.g. PE and the pressure of dissolved air. The waste water treatment plant contains two units, which use the combined technologies of precipitation, flocculation and flotation for separation of aluminium from waste water. These units are operated in parallel. The units are indicated as “old unit” and “new unit”. The old unit was installed in 1991 and the new unit was installed in 2006. There are several differences in geometry between both units. Table 2 also shows the high and low experimental settings of the control factors of the old and the new unit. These settings are based on process or equipment limitations and represent the boundaries for combined unit operation.

Generally speaking, the values of the disturbance factors are unknown. However the value of the disturbance factor “aluminium concentration” can be estimated by turbidity. The aluminium concentration depends on the output of the production plant and will vary between $100 \leq C_{Al} \leq 800$ mg/l. Probably the aluminium concentration influences the % removal considerably. However it is an uncontrollable disturbance factor and may not be taken into account in the factorial design matrix. When the aluminium concentration is not included in the factorial design, the effect on the % removal cannot be measured, which is not preferable.

The experimental runs should be executed in a randomized fashion [23], but because of the disturbance factor “aluminium concentration”, serious errors may occur. The variance in aluminium concentration during the experiments can lead to additional influence on the % removal. For this reason, the disturbance factor “aluminium hydroxide” must be structurally built into the design. This is a new approach in setting up the experimental design resulting in additional information on the disturbance factor aluminium concentration without extra experiments. The structure is based on coupling a control factor towards the disturbance factor “aluminium concentration”. With this structure only 2^5 experimental runs are required instead of 2^6.

First a selection is made as to which controllable factor should have the lowest influence on the % removal. The waste water flow, agitation velocity, ratio $Al(OH)_3$-PE and pressure of dissolved air are all important factors influencing the mixing behavior in the CSTR and the CPF. The mixing behavior in the CSTR can be controlled by the waste water flow and the agitation velocity. When the mixing behavior is uniform, also the pH is uniform and stable from which can be concluded that the pH has the smallest influence on the % removal. It is noticed that the pH value must be lower than the iso-electric point and higher than the minimum solubility of the aluminium hydroxide [4,16]. This indicates that the controllable factor pH can be coupled with the disturbance factor aluminium concentration. The influence of the pH and aluminium concentration can be investigated by studying the mixing behavior in the stirred tank. A full factorial experimental design scheme can be found in Table 3 [23], were the disturbance factor F is represented as the aluminium concentration.

Certain experiments can be coupled, for example the experiment where the pH varies between high (+1) and low (−1) and where all other control factors have the same sign. These coupled experiments are shown in Table 3.

If the coupled experiments are executed only at low aluminium concentrations, the % removal should probably be very good. However if the coupled experiments are executed at high aluminium concentrations, the % removal should be probably very low. Both situations give no reliable results. For this reason, each coupled experiment must be performed once at a high aluminium concentration and once at a low aluminium concentration. In this way the disturbance factor aluminium concentration is an intrinsic part of the experimental design.

In Table 3, the final sign of the disturbance factor aluminium concentration, represented as F is also shown. The opposite sign of pH is chosen, to prevent additional influences of disturbances, related to amount added reactant. The opposite sign of pH against aluminium concentration results in a more uniform amount of added NaOH comparable to normal unit operation, yielding more reliable experiments.

Now, a reliable full factorial design scheme can be constructed, incorporating all controllable factors, including the influence of the most important disturbance factor and with a limited number of experiments!

2.3.2. ANOVA

In this study, the so called F-test was used in the analysis of variance. The F-value is given as:

$$F_A = \frac{S_A^2}{S_{\text{Error}}^2} \quad (1)$$

where S_{Error}^2 is the variance of the overall error and where S_A^2 is the variance with respect to factor A, estimated according to:

$$S_A^2 = \frac{K_A}{\phi_A} \quad (2)$$

where ϕ_A is the degree of freedom with respect to factor A and K_A is the sum of squares with respect to factor A. The sum of squares is calculated from its contrast according to:

$$K_A = \frac{C_A^2}{i^x \cdot n} \quad (3)$$

Table 3
Experimental design matrix and experimental result.

Combination	Run	A	B	C	D	E	F	% Removal old unit		% Removal new unit	
(1)	1	−1	−1	−1	−1	−1	1	99,4	99,9	89,4	86,0
b	3	−1	1	−1	−1	−1	−1	99,2	98,3	99,0	99,0
a	2	1	−1	−1	−1	−1	1	99,8	99,9	56,3	59,1
ab	4	1	1	−1	−1	−1	−1	79,2	77,2	0,0	0,0
c	5	−1	−1	1	−1	−1	1	99,9	100,0	83,9	81,8
bc	7	−1	1	1	−1	−1	−1	98,8	98,7	98,3	96,7
ac	6	1	−1	1	−1	−1	1	97,9	97,8	48,8	51,0
abc	8	1	1	1	−1	−1	−1	98,7	98,7	25,5	24,1
d	9	−1	−1	−1	1	−1	1	98,9	98,9	98,4	97,4
bd	11	−1	1	−1	1	−1	−1	99,0	99,1	98,2	98,2
ad	10	1	−1	−1	1	−1	1	100,0	100,0	100,0	100,0
abd	12	1	1	−1	1	−1	−1	96,8	97,2	90,2	89,6
cd	13	−1	−1	1	1	−1	1	100,0	100,0	69,2	70,0
bcd	15	−1	1	1	1	−1	−1	99,3	99,4	99,6	99,6
acd	14	1	−1	1	1	−1	1	99,8	99,7	100,0	100,0
abcd	16	1	1	1	1	−1	−1	94,2	93,8	90,9	94,4
e	17	−1	−1	−1	−1	1	1	99,9	99,2	86,4	86,5
be	19	−1	1	−1	−1	1	−1	96,2	96,2	98,9	98,8
ae	18	1	−1	−1	−1	1	1	100,0	99,9	98,4	97,8
abe	20	1	1	−1	−1	1	−1	96,4	96,2	84,3	84,6
ce	21	−1	−1	1	−1	1	1	98,8	99,9	48,7	48,1
bce	23	−1	1	1	−1	1	−1	99,3	99,3	99,7	99,6
ace	22	1	−1	1	−1	1	1	100,0	99,9	98,7	98,3
abce	24	1	1	1	−1	1	−1	98,8	98,9	47,3	46,9
de	25	−1	−1	−1	1	1	1	99,8	99,8	56,2	54,8
bde	27	−1	1	−1	1	1	−1	94,7	95,1	98,6	98,6
ade	26	1	−1	−1	1	1	1	100,0	100,0	100,0	100,0
abde	28	1	1	−1	1	1	−1	96,7	96,7	99,4	99,4
cde	29	−1	−1	1	1	1	1	99,9	100,0	99,9	99,9
bcde	31	−1	1	1	1	1	−1	97,5	97,5	98,8	99,1
acde	30	1	−1	1	1	1	1	100,0	100,0	100,0	100,0
abcde	32	1	1	1	1	1	−1	99,4	99,3	97,1	93,7

where i is the number of levels of the factors, x is the number of factors, n is the number of experiment repetitions and C_A is the contrast with respect to A. The contrast with respect to A, can be calculated as a summation of the outcomes of the experiments:

$$C_A = -X_1 + X_{\mathrm{a}} - X_{\mathrm{b}} + \ldots..X_{\mathrm{abcde}} \tag{4}$$

where X_1 is the experimental outcome of experiment (1), X_{a} is the outcome of experiment (a), X_{b} is the outcome of experiment (b), etc. The plus and minus signs in Eq. (4) can be found from a matrix table proposed in [23].

2.4. Empirical model

The full factorial experimental design can be used for the construction of an empirical model. The contrast represents the effect of a factor. The high (+1) and low (−1) levels of the factor influence the effect of a factor on the % removal.

The empirical model has the following structure;

$$y_{\mathrm{emp}} = \hat{y} + \left(\frac{C_A}{2}\right) \cdot x_A + \left(\frac{C_B}{2}\right) \cdot x_B + \left(\frac{C_{AB}}{2}\right) \cdot x_A x_{B\backslash} + \ldots + \left(\frac{C_{ABCDE}}{2}\right) \cdot x_A x_{B\backslash} x_{C\backslash} x_{D\backslash} x_{E\backslash} \tag{5}$$

The empirical model can be used for prediction of the % removal, y_{emp}. $\hat{y}$ is the average % removal of the experiments. The last terms of the model include all the contrasts of the main factors represented as C_A, C_B ... C_E. Each contrast is multiplied with a coded variable x_A, x_B ...x_E. This coded variable has values between high (+1) and low (−1), similar to the factor levels as mentioned in Table 2. The contrast of the second and higher order factor interaction is multiplied by the coded variables of each main factor included in the interaction.

With this model the % removal can be predicted as function of the main factors, taking into account all possible main and interaction influences of the controllable variables [23].

3. Experimental

In total 32 experiments were executed in a randomized order with the factor signs as represented in Table 3 and factor values as mentioned in Table 2. Experiments were performed over a period of several weeks, covering the total production cycle of Fujifilm. This production cycle contains all the PS-plates products and is representative for all the uncontrollable factors. Experiments were performed at low (−1) and high (+1) levels of factor F of respectively ≤400 mg/l and >400 mg/l.

Parallel experiments were performed for the old and new unit and the experimental runs were performed in two-fold. Samples of the waste water were taken before and after treatment, and the aluminium concentration was analyzed with an ICP spectrometer Optima 5300 DV. The % removal was calculated for each experiment and the result is mentioned in Table 3. With a sign matrix as shown in Table 3, the F-value for each factor and interaction between factors was calculated.

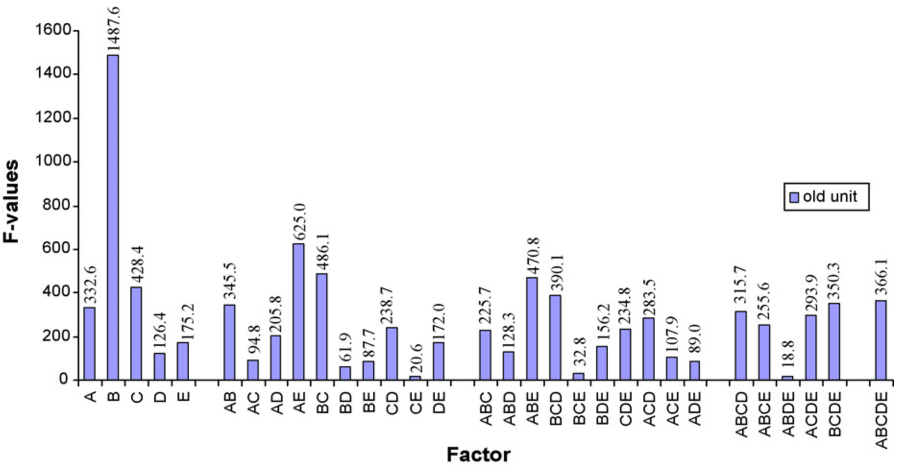

Fig. 3. *F*-values % removal experiment old unit.

4. Results and discussion

The ANOVA results for the old and the new unit are shown in the Figs. 3 and 4, respectively. The magnitude of the *F*-value represents the influence of the factor on the % removal. A high *F*-value indicates that the factor has a large influence on the % removal. When the *F*-value exceeds the critical *F*-value, the effect of the factor is called significant. For this experimental design, the critical *F*-values are 4.15 and 7.5. If $F > 4.15$, the effect of the influence factor is 95% significant and if $F > 7.5$, the effect of the influence factor is 99% significant [23].

4.1. ANOVA of the old unit

ANOVA results of the % removal experiments from the old unit shows that all *F*-values exceed the critical *F*-value of 7.5, as shown in Fig. 3. From the computed *F*-values, the conclusion can be drawn that all factors are 99% significant.

Factor *B*, has compared to the other factors, the largest influence on the % removal. Factor *B* represents the pH or the aluminium concentration, because they are coupled. By analysis of the mixing effect in the neutralization unit, the influence of pH and aluminium concentration can be investigated. The mixing effect is represented as interaction factor *AB*–*AC*–*ABC*. Low levels of *A* (waste water flow) and *C* (stirrer blade velocity) interacting with *B* (pH) result in (i) poor reactant distribution, (ii) low yield and (iii) higher *F*-values compared to factor *B*. However, the *F*-values of interaction factors *AB*–AC–*ABC* are all lower than factor *B*. This indicates that the pH is stable, which was also observed during the experiments. These outcomes show that factor *B* represents the aluminium concentration.

To optimize the overall process, the factors that influence the process need to be identified. The total unit operation is represented by the higher order interaction factors. The *F*-values of $ABCDE \approx BCDE \approx ACDE \approx ABCE \approx ABCD \neq ABDE$. This shows that factor *C* (agitation speed) is the most important factor in the total unit operation. Factor *B* has a large *F*-value but with higher order interaction factors, the influence decreases.

4.2. ANOVA of the new unit

The ANOVA results of the % removal experiments from the new unit show that factor *B* and the interaction factor *AC* are lower than the critical *F*-value of 7.5, as shown in Fig. 4. This indicates that the estimated effect of factor *B* (pH/aluminium concentration) and interaction factor *AC* (waste water flow – stirrer blade velocity) is for 99% related to the experimental error. The outcomes show that all other factors significantly influence the % removal.

Factor *B* represents the pH or aluminium concentration because they are coupled. When factor *B* represents the pH, the interaction factor *AB* should not have a large *F*-value, because during the experiments the pH was stable; resulting in (i) a uniform reactant distribution and (ii) a uniform precipitation. This observation proofs that factor *B* represents the aluminium concentration.

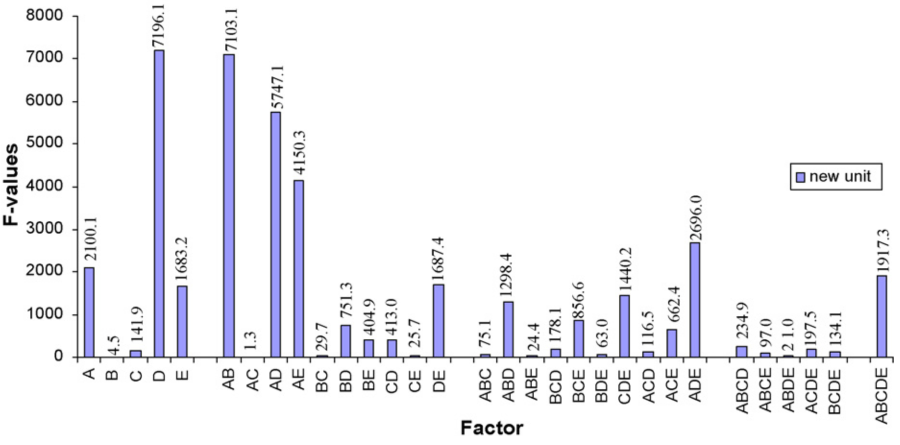

Fig. 4. *F*-values % removal experiment new unit.

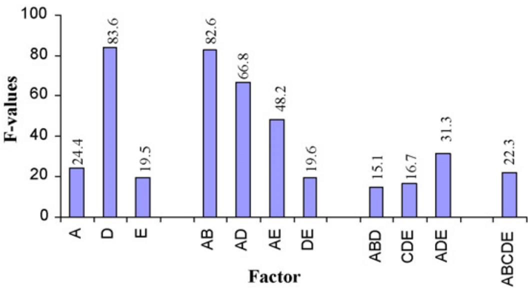

Fig. 5. *F*-values with lack of fit % removal experiment new unit.

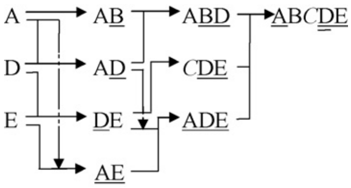

Fig. 6. Hierarchical level of factors.

The *F*-values shown in Fig. 4 vary between 0 and 6000. The factors with ≤2% influence are categorized as lack of fit error. The results of ANOVA, after removal of these factors are shown in Fig. 5. The remaining factors exceed the critical *F*-value of 7.5. Fig. 5 shows that all remaining factors significantly influence the % removal.

To optimize the overall process, the factors that influence the process need to be identified. Fig. 6 shows the importance of each factor for the total unit operation. As can be seen for the total unit operation, the interaction factor *ABCDE* is most important.

The interaction factor *ABCDE* is predominantly influenced by the interaction factor *ADE*. The factors *A*–*D*–*E* all influence the % removal, however factor *D* (ratio Al(OH)$_3$-PE) has the largest *F*-value. In order of increasing influence the following factors are listed: factor *D* (ratio Al(OH)$_3$-PE), factor *A* (waste water flow) and factor *E* (pressure dissolved air).

4.3. Empirical model

The empirical model of Eq. (5) is used to predict the % removal for each experimental run. The experimental design matrix shown in Table 3 is used for the levels of the coded variables. The predicted % removal is compared to the experimental % removal y_{exp} and their residual e is calculated with Eq. (6).

$$e = y_{\text{exp}} - y_{\text{emp}} \tag{6}$$

Because each experimental run is performed in two-fold, totally 64 residuals were calculated. The residuals against experimental % removal for the old and new unit are shown in Fig. 7. This figure shows that the empirical model of the old and the new unit can predict the % removal within an accuracy of 99.2% and 97.9%, respectively. The empirical model of the new unit is compared to the empirical model of the old unit applicable over a much wider % removal range.

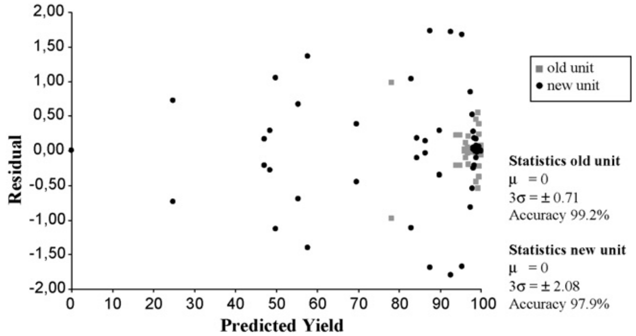

Fig. 7. Residuals versus predicted values % removal old and new unit.

The empirical models for the old and new unit are used to compute the optimal settings of the operational settings. The aluminium concentration (*B*) is an uncontrollable factor. This indicates that the levels of the coded variable for factor *B* are between (+1) and (−1). Also the waste water flow (*A*) will be taken into account as uncontrollable factor. This approach has the advantage that optimal settings will be found for factor *C* (stirrer blade velocity), factor *D* (ratio Al(OH)$_3$-PE) and factor *E* (Pressure dissolved air) within a wide operation window of flow and aluminium concentration. The optimal operational settings for the old and the new unit are listed in Table 4 in bold, indicated with their coded variables.

With the computed operational settings, a % removal of 99.4 ± 0.6 and 97.7 ± 2.3 can be obtained for the old and new unit, respectively. At a higher flow and aluminium concentration the % removal of the new unit decreases. A % removal of more than 97.5% should be obtained as result of environmental legislation. This means that the computed yield for the new unit is too low.

The limits of the empirical model are the coded variables which estimate an accurate % removal at levels of (+1) and (−1). However, this model also can be used for extrapolation and estimation of the % removal beyond the limits. To verify these predictions, validation experiments are performed.

The empirical model of the new unit is used for optimization of the % removal beyond the model limits. The ANOVA results are used for selection of the key variables for further increase of the % removal with lowest economic impact. The ANOVA shows that for the total operation of the new unit, the interaction factor *ADE* is most important. At a higher level, factor *D* (ratio Al(OH)$_3$-PE) is most important followed by factors *A* (waste water flow) and *E* (pressure dissolved air). The coded variables of these factors are used to compute the required settings for increased % removal.

The results of the empirical model for the new unit with the coded variables outside the limits are listed in Table 5. The coded

Table 4
Calculated values % removal old and new unit.

Coded variable			Old unit					New unit				
C	*D*	*E*	A–B (−1)(−1)	A–B (−1)(1)	A–B (1)(−1)	A–B (1)(1)	Average % removal	A–B (−1)(−1)	A–B (−1)(1)	A–B (1)(−1)	A–B (1)(1)	Average % removal
−1	−1	−1	99.6	98.7	99.9	78.2	89.0 ± 10.8	87.7	99.0	57.4	0.0	49.5 ± 49.5
1	−1	−1	99.9	98.8	97.9	98.7	98.8 ± 1.1	82.9	97.5	49.9	24.8	61.2 ± 36.3
−1	1	−1	98.9	99.1	100	97.0	98.5 ± 1.5	97.9	98.2	100	89.9	94.9 ± 5.1
1	1	−1	100	99.3	99.8	94.0	96.9 ± 3.0	69.9	99.6	100	92.7	84.8 ± 15.2
−1	−1	1	99.6	96.2	99.9	96.3	98.0 ± 1.9	86.4	98.8	98.1	84.4	91.6 ± 7.2
1	**−1**	**1**	**99.4**	**99.3**	**100**	**98.8**	**99.4 ± 0.6**	48.4	99.6	98.5	47.1	73.4 ± 26.3
−1	1	1	99.8	94.9	100	76.7	97.4 ± 2.6	55.5	98.6	100	99.4	77.8 ± 22.2
1	**1**	**1**	99.9	97.5	100	99.3	98.7 ± 1.3	**99.9**	**98.9**	**100**	**95.4**	**97.7 ± 2.3**

Table 5
Calculated values % removal new unit beyond boundaries.

Coded variable			*A–B*						Average % removal
C	*D*	*E*	(−1)(−1)	(0)(−1)	(1)(−1)	(−1)(1)	(0)(1)	(1)(1)	
1	1	1	99.9	99.9	100	98.9	97.2	95.4	97.7 ± 2.3
1	1	1.25	103.7	101.8	100	98.8	97.3	95.7	99.7 ± 4.0
1	1	1.5	107.5	103.7	100	98.8	97.4	96.1	101.8 ± 5.7
1	1.25	1	106.3	103.3	100.2	98.8	100.1	101.4	102.6 ± 3.8
1	1.25	1.25	111.1	105.3	99.4	98.7	100.1	101.5	104.9 ± 6.2
1	1.25	1.5	115.9	107.3	98.7	98.6	100	101.5	107.3 ± 8.7
1	**1.5**	**1**	**112.8**	**106.6**	**100.4**	**98.8**	**103.1**	**107.5**	**105.8 ± 7.0**
1	1.5	1.25	118.6	108.7	98.9	98.6	102.9	107.2	108.6 ± 10.0
1	1.5	1.5	124.4	110.9	97.3	98.4	102.7	106.9	110.9 ± 13.6

variable *C* (stirrer blade velocity) is kept at a high level of (+1) for stable mixing behavior in the CSTR. The data shows that an increase in level of the coded variable *E* (dissolved air pressure), results in a small increase in the % removal. However, the increase of dissolved air pressure is not preferable, as expensive modifications of the installation would be required. The increase in level of the coded variable *D* (ratio $Al(OH)_3$-PE) also increased the % removal strongly. Using a high level (+1.5) of coded variable *D*, together with an intermediate (+0) and high (+1) level of coded variable *A* (waste water flow) will increase the % removal to theoretical values over 100%.

4.4. Validation experiments

The optimal operational settings of the new unit are based on the ANOVA results and the empirical model. The optimized value of the coded variable *D* (ratio $Al(OH)_3$-PE)) gives levels beyond (+1). The optimal value of the coded variable *D* should be (+1.5). However, increasing the value of the coded variable *D* beyond level (+1), can result in increased poly-electrolyte repulsive forces and a poor % removal. The optimal operational settings for the old and new unit are based on fixed flow settings. However, it is preferable that both units operates at variable flow.

Validation experiments were performed for both units with variable flow. The purpose of these experiments was to verify the optimal operational settings, as summarized in Table 6. Before performing the experimental run, additional experiments were executed to verify the influence of the variable flow settings. These experiments were performed with settings as mentioned in Table 6, with the levels of coded variable *D* (ratio $Al(OH)_3$-PE)) of (−1) and (+1.5) for the old and new unit, respectively. The effluent of both units was inspected visually. The experiment showed that the effluent of the old and the new unit still contained flakes. However, the effluent of the new unit contained more flakes. The increase of the ratio $Al(OH)_3$-PE up to a level of (+1) and (+3.8) for respectively the old and new unit, resulted in a clean effluent.

The differences between the empirical and experimental coded values of the old and new unit are related to unsteady flow. However, the differences between the empirical and experimental coded values of the new unit are higher compared to the old unit. This difference is also related to empirical % removal prediction with a linear model and with coded values outside the boundaries of the model. This experiment showed that the ratio $Al(OH)_3$-PE in relation to the % removal is not a linear but an exponential function. It must be noted that processes can behave extremely non-linear but in this case the linear approach is useful because the amount of PE is varied within a small range.

In total 20 validation experiments were performed with settings of the variables as mentioned in Table 6, divided into two levels of factor *D*. Ten experiments were performed with levels of factor *D* at (−1) and (+3.8) and ten experiments were performed at levels of (+1) and (+6.8), for the old and new unit, respectively. The level of (+6.8) for the new unit was chosen to create extra safety, because the optimal ratio of $Al(OH)_3$-PE at level (+3.8) was verified by a limited number of experiments.

Experiments were performed over a six week period and all experiments were performed in two-fold. Samples of waste water were taken before and after treatment and the aluminium concentration was measured with an ICP spectrometer Optima 5300 DV. The result of the amount of aluminium in the effluent for the old and new unit is shown in Fig. 8. The amount of aluminium must be ≤20 mg/l based on the Dutch government legislations. In Fig. 8 can be seen that with level (+1) and (+6.8) of factor *D* for respectively the old and new unit, the amount of aluminium in the effluent is $0.04 \leq C_{Al^{3+}} \leq 7.4$ and $0.01 \leq C_{Al^{3+}} \leq 9.34$ mg/l. Both values are within the environmental limits.

From each coded value level of factor *D*, the average % removal with a confidence interval of 99% was calculated. The results are shown in Table 7. A % removal of 99.32 ± 0.3% is reached for the old unit with level (+1) of factor *D* and a % removal of 99.64 ± 0.35% is reached for the new unit with level (+6.8) of factor *D*.

The optimal settings of the variables are shown in Table 6 in bold. The settings of the variables of the old unit are the same as the currently employed operational settings. However, the final settings of the variables of the new unit are different from the current operational settings. The flow range is limited to a lower value of 42.5 m^3/h, instead of 30 m^3/h and the amount of poly-electrolyte that must be added is 2.6 times higher. The additional amount of

Table 6
Values optimal process variable settings old and new unit.

Factor	Symbol	Old unit		New unit	
		Coded variable	Process value	Coded variable	Process value
Flow waste water (m^3/h)	*A*	**$(-1) \leq x_A \leq (+1)$**	**30 – 55**	**$(0) \leq x_A \leq (+1)$**	**42.5 – 55**
pH		–	**7.75**	–	**7.75**
Stirrer blade velocity (rpm)	*C*	**(+1)**	**1450**	**(+1)**	**1450**
Ratio	*D*	(−1)	18	(+1.5)	44
$Al(OH)_3$-		**(+1)**	**30**	(+3.8)	60
PE				**(+6.8)**	**80**
Pressure dissolved air (bar)	*E*	**(+1)**	**6.0**	**(+1)**	**6.0**

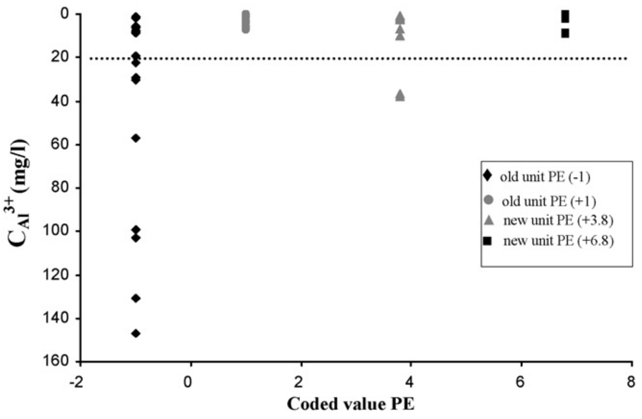

Fig. 8. Results validation experiment validation experiment.

Table 7
Experimental statistics.

	PE coded	$\bar{x}$	σ	$\mu = \bar{x} \pm t_{n-1} \cdot \frac{\sigma}{\sqrt{n}}$
Old unit	(−1)	95.4	3.8	95.4 ± 2.46
	(+1)	99.3	0.4	**99.32 ± 0.3**
New unit	(+3.8)	98.6	2.9	98.62 ± 1.9
	(+6.8)	99.6	0.5	**99.64 ± 0.35**

t_{n-1} 99%:2.86, $n = 20$.

poly-electrolyte results in an annual increase in operational cost of 65 Keuro, although the amount of aluminium separated is higher than the environmental minimum.

4.5. *The influence of unit geometries*

The ANOVA results can also be used to study the effect of the geometries of the unit as both units, the old and new unit have different geometries. The diameter of the coiled pipe flocculator (CPF) from the new unit is much smaller, than to the old unit. The smaller diameter results in a higher velocity of the flake and higher shear forces. The shear forces result in breakage of the flakes and a lower % removal at higher flow and aluminium concentration, which can only be prevented by increase of the amount of poly-electrolyte. This will result in a stronger flakes and a higher % removal. Reduction of the operational costs of poly-electrolyte is possible when the CPF of the new unit will be redesigned. When using the same design of the CPF from the old unit, the poly-electrolyte usage can be decreased with 65 Keuro yearly. The costs of a new CPF are 30 Keuro which results in a return of investment period of only half a year.

5. Conclusion

In this paper, a systematic approach for optimization of a waste water treatment plant is presented. It includes the identification of all the variables and the selection and optimization of the settings of the control variables. An experimental design was developed and executed and the results were analyzed by means of ANOVA. The results of the ANOVA were used for the construction of an empirical model. The outcome of the ANOVA and the empirical model were used for calculation of the optimal operational settings. Validation experiments were performed to confirm the optimal settings. In total 40 variables were identified as factors influencing the separation of aluminium from waste water and in total 5 variables were selected as control factors. The study showed that the amount of poly-electrolyte is a key factor for combined unit operation. For the new unit the amount of poly-electrolyte had to be increased 2.6 times compared to the old unit to comply with governmental legislation. The optimal settings of the control variables that were found resulted in a % removal of 99.32 ± 0.3% and 99.6 ± 0.35% for the old and new unit, respectively which is higher than the governmental limit of 97.5%. The increase of the poly-electrolyte for the new unit is a result of high shear forces on the flake, as the diameter of the CPF is smaller than the old unit. Instead of increased use of poly-electrolyte, the CPF from the new unit could be redesigned. When using the same design of the CPF of the old unit, the poly-electrolyte can be decreased significantly.

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