Applying process analytical technology framework
to optimize multiple responses in wastewater treatment process

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Abstract:　　In this research, the process analytical technology (PAT) framework is used to optimize the performance of the wastewater treatment process in poultry industry. Two responses, turbidity and sludge volume index (SVI), are of main manufacturer's interest. Initially, the moving average (MA) and moving range (MR) control charts are established for each response. The 3^3 full factorial design with two replicates is then used for conducting experimental work. The weighted additive model in fuzzy goal programming is formulated, and then employed to determine the combination of optimal factor settings. Finally, confirmation experiments follow at the combination of optimal factor settings. The results show that the actual process index for turbidity is improved from 1.34 to 5.5, while it is enhanced from 1.46 to 1.93 for SVI. Moreover, the multiple process capability index is improved significantly from 1.95 to 10.6, which also indicates that the treatment process becomes highly capable with both responses concurrently. Further, the process standard deviations at initial (optimal) factor settings are 2.16 (1.27) and 6.02 (3.39) for turbidity and SVI, respectively. These values show significant variability reductions in turbidity and SVI by 41.22% and 77.75%, respectively. Such improvements will lead to huge savings in quality and productivity costs. In conclusion, the PAT framework is found to be an effective approach for optimizing the performance of the wastewater treatment process with multiple responses.

Key words: Fuzzy goal optimization, Multiple responses, Wastewater, Process analytical technology (PAT)
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1 Introduction

In recent years, there has been an increased interest in wastewater treatment for environmental conservation. As a consequence, the industrial sector is obligated to treat its own waste and lower its organic load to a level that the ecosystem can cope with. The efficient employment of engineering concepts in parallel with process knowledge can lead to tangible improvements in process capability and potential.

The industrial wastewater treatment process can be defined as chemical, biological and/or mechanical procedures applied to industrial discharge in order to increase the quality of wastewater by reducing its organic and inorganic pollutants. The treated water quality can be described by several physical parameters, such as turbidity and sludge volume index (SVI), which are considered as vital quality responses that have large effects on effluent water quality.

Process analytical technology (PAT) is a system that analyses and controls manufacturing processes based on timely measurements of critical quality characteristics and performance attributes of raw materials and in-process products to ensure acceptable end-product quality (CDER, 2004). The PAT framework is a combination of tools that, when used within a system, can provide useful means for acquiring information resulting in continual process improvement, such as multivariate techniques, data acquisition and analysis, process analysis methods, process control tools, and continual improvement and a knowledge management approach. A summary of the tools that are commonly used in the PAT framework is displayed in Fig. 1.
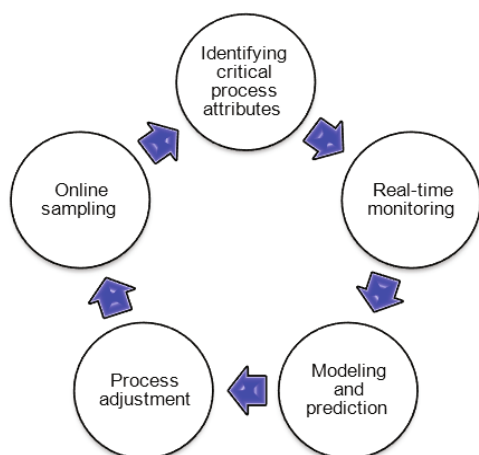
Fig. 1 The PAT framework

Recently, improving the performance of the wastewater treatment process has received significant research attention. Wang *et al.* (2007) used response surface methodology to optimize the coagulation-flocculation process for paper-recycling wastewater treatment. Wang *et al.* (2011) applied a uniform design in parallel with response surface methodology to optimize the coagulation-flocculation process for pulp mill wastewater treatment. Furthermore, several approaches have been proposed for optimizing process performance with multiple responses (Al-Refaie *et al.*, 2008; 2009; Al-Refaie, 2009; 2010; 2011; 2012; Al-Refaie and Al-Tahat, 2011; Muhammad *et al.*, 2012; Salmasnia *et al.*, 2012). Among the efficient optimization approaches is the weighted additive model in fuzzy goal programming, which considers the preferences for quality responses when determining the combination of optimal process factor's settings. The weighted additive model has been applied to optimize process performance in several industrial applications (Yaghoobi *et al.*, 2008; Yücel and Guneri, 2011; Al-Refaie and Li, 2011; Al-Refaie *et al.*, 2012). This research aims at applying the PAT framework, including the weighted additive model, to optimize the performance of the coagulation-flocculation process in wastewater treatment.

2 PAT framework

The PAT framework was implemented to improve the performance of the wastewater treatment process and is described as follows.

2.1 Identifying critical process attributes

The wastewater treatment process is mapped as shown in Fig. 2. The inflow stream is screened at first using two rotary screens to remove coarse materials that could damage the subsequent process equipment. The screened flow is then physically treated in a dissolved air flotation unit (DAF) where it is exposed to a pressurized air stream in order to float its oil and fat content. The floating fat layer is then scraped off by a skimming device. The treated stream is passed through an aeration process using a selector tank to enhance the growth of floc-forming bacteria instead of filamentous bacteria to provide an activated sludge with better settling and thickening properties. The aerated stream is then biologically treated in a treatment tank where the dissolved and particulate constituents are biologically degraded. The last treatment process is coagulation-flocculation in which sludge is separated from water by adding certain coagulant and flocculant agents. The flocculated sludge will float by means of a pressurized air stream where it is scraped by a skimming device. On the other hand, the treated water is then chlorinated and stored, to be used for irrigation purposes.
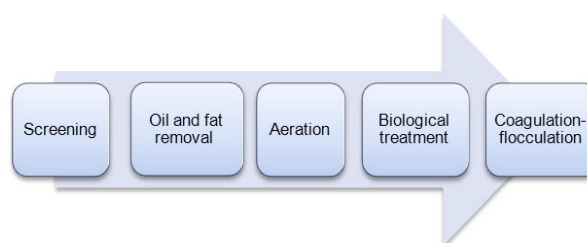

Fig. 2 Process mapping of treatment process

2.2 Real-time monitoring

A control chart plots the average of measured values of quality responses in samples taken from the process versus the sample number. The chart has a center line (CL) as well as upper and lower control limits (UCL and LCL, respectively). The CL represents where this process characteristic should fall if no assignable causes for variability are present. The moving average (MA) and moving range (MR) control charts are found to be effective ones in detecting small process shifts and process variability, respectively. The turbidity is measured using a portable DR/890 colorimeter instrument, while the SVI is

measured manually using the standard method. The MA and MR control charts for turbidity and SVI are constructed and depicted in Figs. 3 and 4, respectively. In Fig. 3 for turbidity, the UCL, CL, and LCL of the MA control chart are calculated and found respectively equal to 24.81, 20.22, and 15.64 NTU (nephelometric turbidity units), while the corresponding parameter values in the MR control chart are equal to 7.964, 2.438, and 0 NTU, respectively. In MA and MR control charts, neither point falls outside the control limits nor does a significant pattern exist in the charts. Hence, it is concluded that the process is in control for turbidity and SVI. Furthermore, the corresponding lower limit (LSL) and upper specification limit (USL) for the turbidity are 0 and 30 NTU, respectively, while the smaller SVI value is preferred within the specification range. Observing the CL (=20.22 NTU) of the MA control chart for turbidity, it is noted that the turbidity is significantly larger than the above maximum allowable target value of 15 NTU. From Fig. 4 for SVI, the UCL, CL, and LCL of the MA (MR) control chart are estimated

respectively as 92.69 (22.19), 79.92 (6.79), and 67.15 (0) ml/g. The LSL and USL of SVI are 50 and 100 ml/g, respectively, where it is preferred at the nominal value of 70 ml/g. Hence, the SVI is considered the nominal best type response. It is found that the CL (=79.92 ml/g) for SVI is shifted from the nominal value. Consequently, improvement actions are required to improve process performance.

2.3 Modeling and prediction

In the third step of the PAT framework, a mathematical relationship is developed for both turbidity, y_1, and SVI, y_2, with the critical process factors. Based on process knowledge, the main controllable process factors thought to affect the wastewater quality characteristics are flocculant dose (x_1, mg/L), coagulant dose (x_2, mg/L), and pH (x_3). The current process factor settings for the wastewater treatment process are: flocculant dose=8 mg/L, coagulant dose=50 mg/L and pH=6. Each factor is assigned at three levels: 1: low, 2: middle, and 3: high. The physical values for factor levels are listed in Table 1.
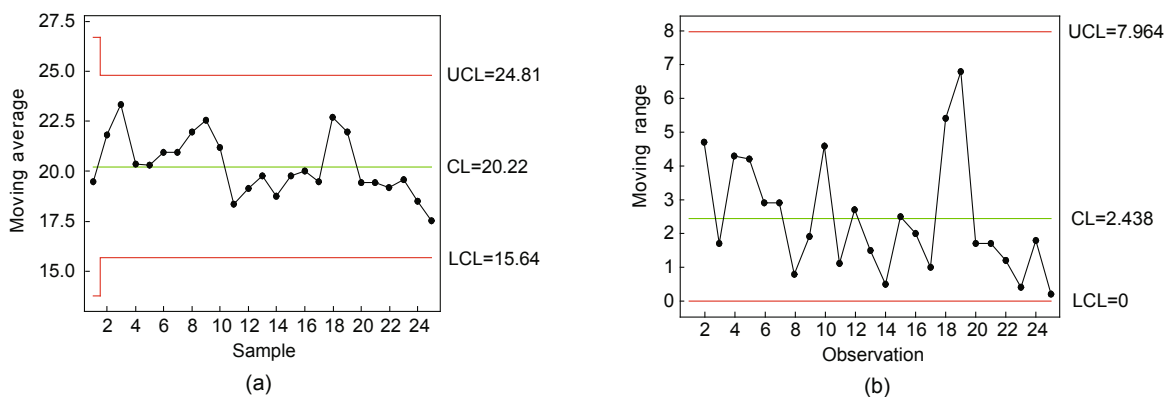

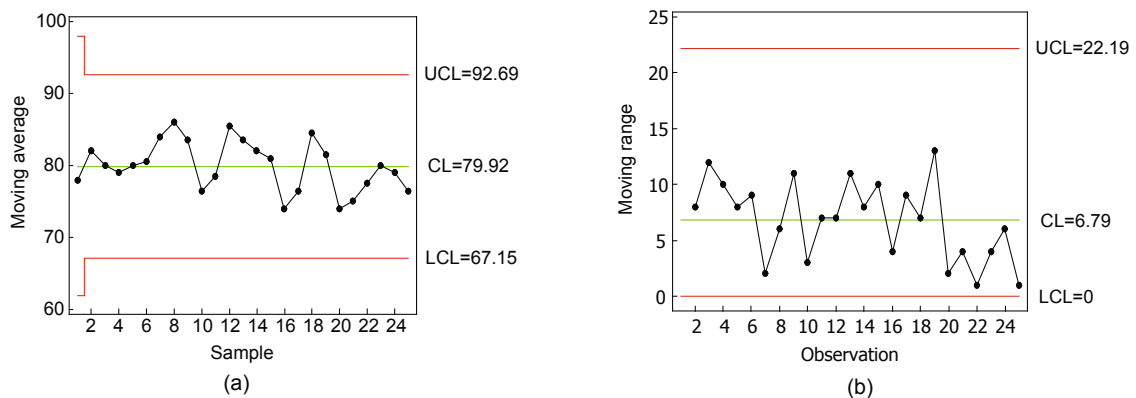**Fig. 3 MA chart (a) and MR chart (b) for turbidity at initial factor settings**

Fig. 4 MA chart (a) and MR chart (b) for SVI at initial factor settings

For the present process, 3^3 full factorial design with two replicates I and II, enables investigation of three three-level factors with their interactions without the need to ignore the three-way interaction. The designed experiment is conducted and tested using jar test apparatus which is specially designed to be used with conventional wastewater treatment processes such as coagulation, flocculation, and sedimentation. Turbidity and SVI are measured once in each replicate. Table 2 displays the experimental results for both quality responses.

Table 1 Physical values for factor levels

Level	Factor		
	x_1	x_2	x_3
1	6	40	4
2	12	60	6
3	18	80	8

Table 2 Experimental results for both quality responses

Exp. No.	Process factor			Turbidity (y_1) replicate		SVI (y_2) replicate	
	x_1	x_2	x_3	I	II	I	II
1	6	40	4	9.3	9.2	89.2	90.1
2	6	40	6	25.7	24.0	101.6	102.2
3	6	40	8	23.2	24.6	103.2	105.3
4	6	60	4	20.8	20.1	107.9	108.2
5	6	60	6	29.1	28.7	106.7	107.5
6	6	60	8	26.5	28.2	121.3	123.4
7	6	80	4	45.3	46.4	122.4	121.8
8	6	80	6	36.6	35.2	133.6	132.8
9	6	80	8	40.2	39.7	136.9	137.1
10	12	40	4	4.5	4.2	78.8	77.2
11	12	40	6	10.1	9.8	98.6	96.4
12	12	40	8	13.3	12.9	104.3	103.9
13	12	60	4	14.5	14.3	106.0	104.2
14	12	60	6	19.1	18.3	103.1	101.3
15	12	60	8	16.6	17.2	101.9	105.2
16	12	80	4	20.2	21.8	115.0	118.1
17	12	80	6	30.2	29.4	124.1	119.2
18	12	80	8	25.4	24.3	126.3	129.8
19	18	40	4	8.2	8.9	76.1	79.8
20	18	40	6	9.5	10.2	73.3	75.2
21	18	40	8	10.3	9.6	83.8	87.9
22	18	60	4	8.1	7.7	79.2	79.4
23	18	60	6	10.7	10.2	92.4	92.6
24	18	60	8	12.2	12.6	101.1	100.6
25	18	80	4	15.8	15.2	95.8	103.5
26	18	80	6	16.3	15.5	101.6	105.1
27	18	80	8	17.8	17.3	115.7	116.3

2.4 Process adjustment

The weighted additive model will be employed to obtain the optimal values of process factor settings while considering the preferences of process/product engineers as follows.

Step 1: Estimate the relationship between each of y_1 and y_2 responses and the process factors x_1, x_2, and x_3 using statistical multiple linear regression as follows:

$$y_k = f(x_1, x_2, \ldots, x_J), \quad k=1, 2, \tag{1}$$

where f is assumed to be a linear function, y_k denotes the kth quality response, and x_j represents the jth process factor, $j=1, 2, \ldots, J$.

Analyses of variance (ANOVA) for the experimental values of y_1 and y_2 are conducted to determine significant main and interaction effects. ANOVA results for both responses are displayed in Table 3. Obviously, all main two-way interactions and three-way interactions are found to be significant for both responses. Moreover, the corresponding significance tests of linear regression coefficients are listed in Table 4.

Finally, the normality tests for turbidity and SVI are constructed in Figs. 5 and 6, respectively. Obviously, in the plots of fitted values versus the normalized residual and the normal probability plots for residuals, there is no obviously significant pattern or outliers or a relationship between the fitted values and their corresponding residual values. Consequently, mathematical relationships for y_1 and y_2 are expressed in Eqs. (2) and (3), respectively.

$$\begin{aligned} y_1 = &-84.6 + 4.71x_1 + 1.83x_2 + 12.8x_3 - 0.0962x_1x_2 \\ &- 0.716x_1x_3 - 0.184x_2x_3 + 0.011x_1x_2x_3, \end{aligned} \tag{2}$$

$$\begin{aligned} y_2 = &31.5 + 0.28x_1 + 1.24x_2 + 7.11x_3 - 0.0363x_1x_2 \\ &- 0.241x_1x_3 - 0.0631x_2x_3 + 0.00424x_1x_2x_3. \end{aligned} \tag{3}$$

Step 2: Select proper membership function (MF) for y_1, y_2, and x_j. Let the MFs for turbidity and SVI be denoted as μ_{y_1} and μ_{y_2}, respectively, which are de-

cided as follows.

Table 3 Analysis of variance for turbidity

| Term	Degrees of freedom	Turbidity (y_1)				SVI (y_2)			
		Sum of squares	Mean sum of squares	F	P	Sum of squares	Mean sum of squares	F	P
x_1	2	2571.40	1285.70	3321.91	0.000	4389.11	2194.56	627.35	0.000
x_2	2	2027.67	1013.83	2619.48	0.000	7837.79	3919.40	1120.42	0.000
x_3	2	212.80	106.41	274.94	0.000	1758.74	879.37	251.38	0.000
x_1x_2	4	348.00	87.00	224.78	0.000	51.54	12.89	3.68	0.016
x_1x_3	4	38.68	9.67	24.98	0.000	61.09	15.27	4.37	0.007
x_2x_3	4	133.17	33.29	86.02	0.000	64.19	16.05	4.59	0.006
$x_1x_2x_3$	8	304.93	38.12	98.48	0.000	678.18	84.77	24.23	0.000
Error	27	10.45	0.39			94.45	3.50		
Total	53	5647.12				14936.09			
		*R^2=99.81%, R^2 (adjusted)=99.64%				R^2=99.37%, R^2 (adjusted)=98.76%			

*R^2: coefficient of determination

Table 4 Results of test of significance for factor coefficients

Predictor	Turbidity (y_1)				SVI (y_2)			
	Coefficient	Standard Error	T	P	Coefficient	Standard Error	T	P
Constant	−84.59	18.03	−4.69	0.000	31.46	25.58	1.23	0.225
x_1	4.718	1.391	3.39	0.001	0.279	1.974	0.14	0.888
x_2	1.8324	0.2900	6.32	0.000	1.2419	0.4114	3.02	0.004
x_3	12.767	2.900	4.40	0.000	7.111	4.114	1.73	0.091
x_1x_2	−0.09618	0.02237	−4.30	0.000	−0.03627	0.03174	−1.14	0.259
x_2x_3	−0.18417	0.04664	−3.95	0.000	−0.06312	0.06616	−0.95	0.345
x_1x_3	−0.7160	0.2237	−3.20	0.002	−0.2408	0.3174	−0.76	0.452
$x_1x_2x_3$	0.011042	0.003598	3.07	0.004	0.004245	0.005105	0.83	0.410
	*R^2=90.3%, R^2 (adjusted)=88.80%				R^2=92.6%, R^2 (adjusted)=91.50%			

*R^2: coefficient of determination
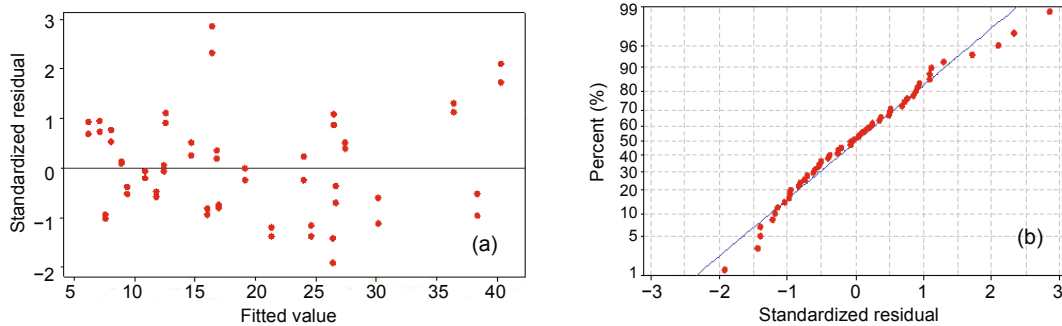
Fig. 5 Normality test results for turbidity (y_1)
(a) Residual versus the fitted value; (b) Normal probability plot of the residuals
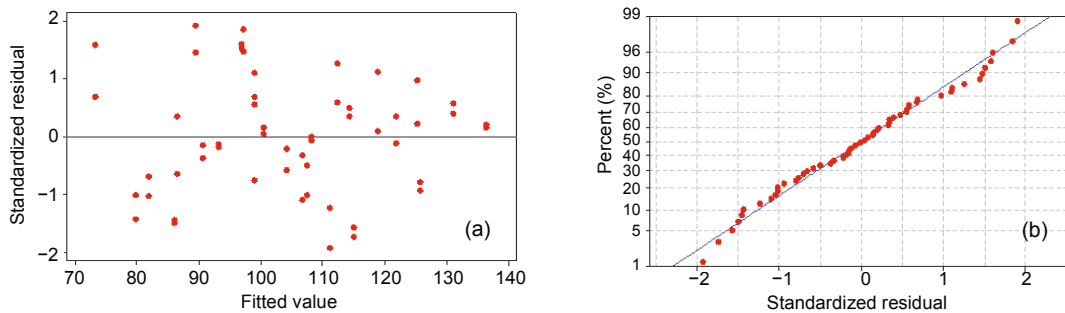
Fig. 6 Normality test results for SVI (y_2)
(a) Residual versus the fitted value; (b) Normal probability plot of the residuals

1. For the turbidity, y_1, the suitable μ_{y_1} is depicted in Fig. 7a and is defined as

$$\mu_{y_1} = \begin{cases} 1, & 0 \leq y_1 < g_{y_1}, \\ 1 - \dfrac{y_1 - g_{y_1}}{\Delta_{y_1}^+}, & g_{y_1} \leq y_1 < g_{y_1} + \Delta_{y_1}^+, \\ 0, & y_1 \geq g_{y_1} + \Delta_{y_1}^+, \end{cases} \tag{4}$$

where g_{y_1} is the imprecise fuzzy value of y_1 specified by process engineers, and $\Delta_{y_1}^+$ is the maximal positive admissible violation from g_{y_1}. The corresponding goal constraints are given by

$$y_1 - \delta_{y_1}^+ \leq g_{y_1}, \quad \mu_{y_1} + \dfrac{\delta_{y_1}^+}{\Delta_{y_1}^+} = 1, \quad 0 \leq \delta_{y_1}^+ \leq \Delta_{y_1}^+, \tag{5}$$

where $\delta_{y_1}^+$ denotes the positive deviation of y_1 from g_{y_1}. Based on the engineers' preferences, the values of g_{y_1} and $\Delta_{y_1}^+$ are decided to be 15 NTU, while the maximal acceptable value of y_1 is 30 NTU. Consequently,

$$\mu_{y_1} = \begin{cases} 1, & 0 \leq y_1 < 15, \\ 1 - \dfrac{y_1 - 15}{15}, & 15 \leq y_1 < 30, \\ 0, & y_1 \geq 30, \end{cases}$$

and

$$y_1 - \delta_{y_1}^+ \leq 15, \quad \mu_{y_1} + \dfrac{\delta_{y_1}^+}{15} = 1, \quad 0 \leq \delta_{y_1}^+ \leq 15.$$

2. Similarly, the suitable MF, μ_{y_2}, for SVI is shown in Fig. 7b, or mathematically

$$\mu_{y_2} = \begin{cases} 0, & y_2 < g_{y_2} - \Delta_{y_2}^-, \\ 1 - \dfrac{g_{y_2} - y_2}{\Delta_{y_2}^-}, & g_{y_2} - \Delta_{y_2}^- \leq y_2 < g_{y_2}, \\ 1 - \dfrac{y_2 - g_{y_2}}{\Delta_{y_2}^+}, & g_{y_2} \leq y_2 < g_{y_2} + \Delta_{y_2}^+, \\ 0, & y_2 \geq g_{y_2} + \Delta_{y_2}^+, \end{cases} \tag{6}$$

where g_{y_2} is the target of y_2. $\Delta_{y_2}^-$ and $\Delta_{y_2}^+$ denote the maximal negative and positive admissible violations from g_{y_2}, respectively. The corresponding goal constraints are expressed as

$$y_2 + \delta_{y_2}^- - \delta_{y_2}^+ = g_{y_2}, \quad \mu_{y_2} + \dfrac{\delta_{y_2}^-}{\Delta_{y_2}^-} + \dfrac{\delta_{y_2}^+}{\Delta_{y_2}^+} = 1, \tag{7}$$
$$0 \leq \delta_{y_2}^- \leq \Delta_{y_2}^-, \quad 0 \leq \delta_{y_2}^+ \leq \Delta_{y_2}^+,$$

where $\delta_{y_2}^-$ and $\delta_{y_2}^+$ represent the negative and positive deviations from g_{y_2}, respectively. The process engineer decided that the values of g_{y_2}, $\Delta_{y_2}^-$, and $\Delta_{y_2}^+$ are 60, 20, and 40 ml/g, respectively. The minimal and maximal acceptable values of y_2 are 50 and 100 ml/g. Then,

$$\mu_{y_2} = \begin{cases} 0, & y_2 < 50, \\ 1 - \dfrac{70 - y_2}{20}, & 50 \leq y_2 < 70, \\ 1 - \dfrac{y_2 - 70}{30}, & 70 \leq y_2 < 100, \\ 0, & y_2 \geq 100, \end{cases}$$

and

$$y_2 + \delta_{y_2}^- - \delta_{y_2}^+ = 70, \quad \mu_{y_2} + \dfrac{\delta_{y_2}^-}{20} + \dfrac{\delta_{y_2}^+}{30} = 1,$$
$$0 \leq \delta_{y_2}^- \leq 20, \quad 0 \leq \delta_{y_2}^+ \leq 30.$$

3. Similarly, there is no information on the exact target of x_1, x_2, and x_3. Then, the suitable MF, μ_{x_j}, is described in Fig. 7c and is defined as

$$\mu_{x_j} = \begin{cases} 0, & x_j < g^l_{x_j} - \Delta^-_{x_j}, \\ 1 - \dfrac{g^l_{x_j} - x_j}{\Delta^-_{x_j}}, & g^l_{x_j} - \Delta^-_{x_j} \le x_j < g^l_{x_j}, \\ 1, & g^l_{x_j} \le x_j < g^u_{x_j}, \\ 1 - \dfrac{x_j - g^u_{x_j}}{\Delta^+_{x_j}}, & g^u_{x_j} \le x_j < g^u_{x_j} + \Delta^+_{x_j}, \\ 0, & x_j \ge g^u_{x_j} + \Delta^+_{x_j}, \end{cases} \qquad (8)$$
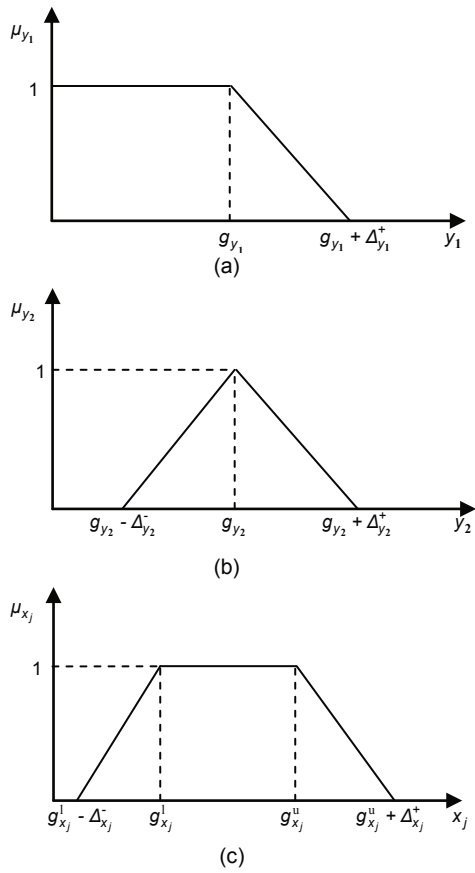

(a)

(b)

(c)

Fig. 7 MFs for responses and process factors
(a) MF for y_1; (b) MF for y_2; (c) MF for x_j

where $g^l_{x_j}$ and $g^u_{x_j}$ are the lower and the upper limits of x_j, respectively. $\Delta^-_{x_j}$ and $\Delta^+_{x_j}$ are the maximal negative and positive admissible violations from $g^l_{x_j}$ and $g^u_{x_j}$, respectively. The corresponding constraints are then formulated as

$$x_j + \delta^-_{x_j} \ge g^l_{x_j}, \quad x_j - \delta^+_{x_j} \le g^u_{x_j}, \quad \mu_{x_j} + \frac{\delta^-_{x_j}}{\Delta^-_{x_j}} + \frac{\delta^+_{x_j}}{\Delta^+_{x_j}} = 1, \qquad (9)$$

$$0 \le \delta^-_{x_j} \le \Delta^-_{x_j}, \quad 0 \le \delta^+_{x_j} \le \Delta^+_{x_j},$$

where $\delta^-_{x_j}$ and $\delta^+_{x_j}$ represent the negative and positive deviations from $g^l_{x_j}$ and $g^u_{x_j}$, respectively. It is decided that the values of $\Delta^-_{x_j}$ and $\Delta^+_{x_j}$ equal 0.1. For example, μ_{x_1} is defined as

$$\mu_{x_1} = \begin{cases} 0, & x_1 < 5.9, \\ 1 - \dfrac{6 - x_j}{0.1}, & 5.9 \le x_1 < 6, \\ 1, & 6 \le x_1 < 18, \\ 1 - \dfrac{x_j - 18}{0.1}, & 18 \le x_1 < 18.1, \\ 0, & x_1 \ge 18.1. \end{cases}$$

The corresponding constraints are defined as

$$x_1 + \delta^-_{x_1} \ge 6, \quad x_1 - \delta^+_{x_1} \le 18,$$

$$\mu_{x_1} + \frac{\delta^-_{x_1}}{0.1} + \frac{\delta^+_{x_1}}{0.1} = 1,$$

$$0 \le \delta^-_{x_1} \le 0.1, \quad 0 \le \delta^+_{x_1} \le 0.1.$$

The MFs with their corresponding constraints for x_1 and x_2 are formulated in a similar manner.

Step 3: The objective function of the weighted additive GP model is to minimize the sum of the weighted deviations of responses and process variables.

Accordingly, the objective function is to minimize

$$Z = w_{y1}\left(\frac{\delta^+_{y1}}{\Delta^+_{y1}}\right) + w_{y2}\left(\frac{\delta^+_{y2}}{\Delta^+_{y2}} + \frac{\delta^-_{y2}}{\Delta^-_{y2}}\right)$$

$$+ \sum_{j=1}^{3} w_{x_j}\left(\frac{\delta^+_{x_j}}{\Delta^+_{x_j}} + \frac{\delta^-_{x_j}}{\Delta^-_{x_j}}\right). \qquad (10)$$

Substituting the values of deviations weights, Eq. (10) is formulated as

$$Z = 0.2\left(\frac{\delta_{y_1}^+}{15}\right) + 0.2\left(\frac{\delta_{y_2}^+}{20} + \frac{\delta_{y_2}^-}{30}\right) + 0.2\sum_{j=1}^{3}\left(\frac{\delta_{x_j}^+}{0.1} + \frac{\delta_{x_j}^-}{0.1}\right).$$

Step 4: A linear programming model is formulated using steps 1–4 as follows:

Minimize Z subject to:

$$y_1 = -84.6 + 4.71x_1 + 1.83x_2 + 12.8x_3 - 0.0962x_1x_2$$
$$- 0.716x_1x_3 - 0.184x_2x_3 + 0.011x_1x_2x_3,$$

$$y_2 = 31.5 + 0.28x_1 + 1.24x_2 + 7.11x_3 - 0.0363x_1x_2$$
$$- 0.241x_1x_3 - 0.0631x_2x_3 + 0.00424x_1x_2x_3,$$

$$y_1 - \delta_{y_1}^+ \leq 15, \quad \mu_{y_1} + \frac{\delta_{y_1}^+}{15} = 1, \quad 0 \leq \delta_{y_1}^+ \leq 15,$$

$$y_2 + \delta_{y_2}^- - \delta_{y_2}^+ = 70, \quad \mu_{y_2} + \frac{\delta_{y_2}^-}{20} + \frac{\delta_{y_2}^+}{30} = 1,$$

$$0 \leq \delta_{y_2}^- \leq 20, \quad 0 \leq \delta_{y_2}^+ \leq 30,$$

$$x_1 + \delta_{x_1}^- \geq 6, \quad x_1 - \delta_{x_1}^+ \leq 18,$$

$$x_2 + \delta_{x_2}^- \geq 40, \quad x_2 - \delta_{x_2}^+ \leq 80,$$

$$x_3 + \delta_{x_3}^- \geq 4, \quad x_3 - \delta_{x_3}^+ \leq 8,$$

$$x_2 + \delta_{x_2}^- \geq 40, \quad x_2 - \delta_{x_2}^+ \leq 80,$$

$$x_3 + \delta_{x_3}^- \geq 4, \quad x_3 - \delta_{x_3}^+ \leq 8,$$

$$\mu_{x_j} + 10(\delta_{x_j}^- + \delta_{x_j}^+) = 1, \quad j = 1, 2, 3,$$

$$0 \leq \delta_{x_j}^- \leq 0.1, \quad j = 1, 2, 3,$$

$$0 \leq \delta_{x_j}^+ \leq 0.1, \quad j = 1, 2, 3,$$

$$\mu_{x_j}, \mu_{y_k}, y_k, x_j \geq 0, \quad j = 1, 2, 3, \ k = 1, 2.$$

Solving the model, the obtained optimal process conditions are: flocculant dose, x_1, of 18 mg/L, co-agulant dose, x_2, of 40.0 mg/L, and pH, x_3, of 4.0.

These parameters yield optimal values of turbidity and SVI of 6.184 NTU and 73.21 ml/g, respectively. The membership values for y_1 and y_2 are 100% and 89.3%, respectively. Moreover, all the process preferences are completely satisfied (=100%).

2.5 Online sampling

The last step in the PAT framework includes conducting confirmation experiments utilizing the optimal factors obtained by the weighted additive model. For this purpose, the MA and MR control charts are constructed for validation of optimal process performance and to monitor future production. The MA and MR control charts at the combination of optimal factor settings are shown in Figs. 8 and 9 for turbidity and SVI, respectively.

In Fig. 8 for turbidity, the CL values of the MA and MR control charts are 6.42 and 1.433 NTU, respectively. The corresponding values of the UCL and LCL for the MA (MR) control charts are 9.116 (4.683) and 3.724 (0.0) NTU, respectively. On the other hand, in Fig. 9 for SVI, the UCL, CL and LCL values of MA (MR) are 79.34 (12.48), 72.15 (3.82), and 64.97 (0) ml/g, respectively. Finally, Figs. 8 and 9 reveal no unusual pattern or any point outside the control
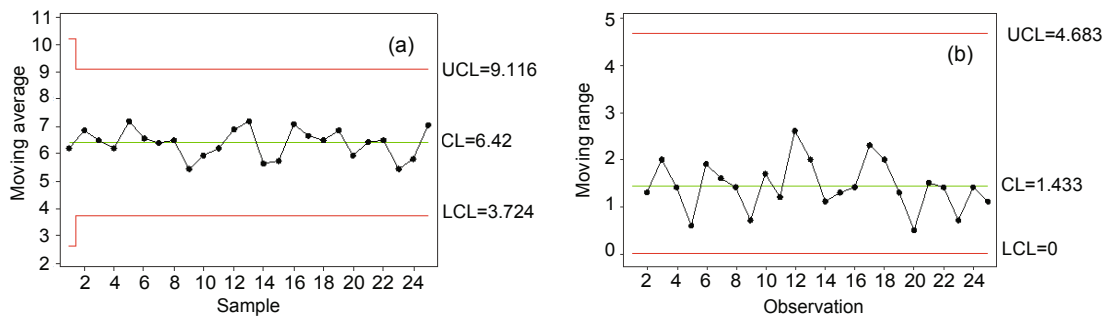

Fig. 8 MA control chart (a) and MR chart (b) for turbidity at optimal factor settings
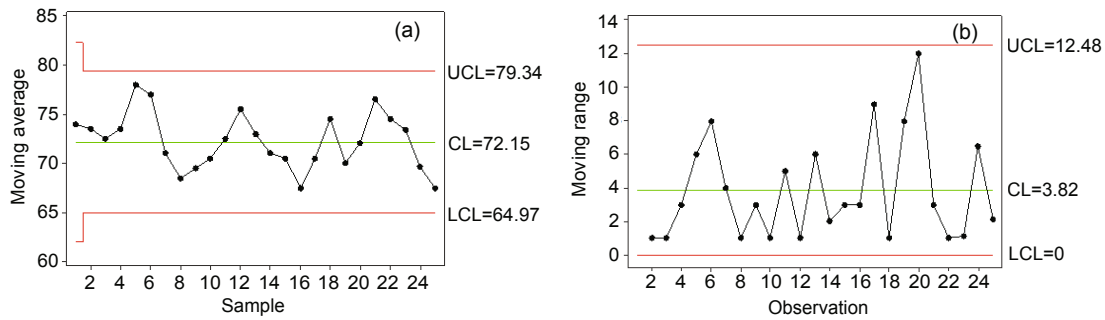
Fig. 9 MA control chart (a) and MR chart (b) for SVI at optimal factor settings

limits or any significant pattern within the control limits. Consequently, the MA (MR) control charts are concluded to be in-control state for both responses, and hence they can be used for monitoring and controlling future treatment process.

3 Improvement analysis

The summary of the control chart results are displayed in Table 5. Process capability analysis will be utilized to measure the performance of the wastewater treatment process before and after the PAT framework. The most widely used process capability index is $\hat{C}_p$. However, this ratio does not take into account where the process mean is located relative to specifications. As a consequence, a new process capability ratio, $\hat{C}_{pk}$, which takes process centering into account, will be used for measuring performance. $\hat{C}_{pk}$ is a function of $\hat{C}_{pu}$ and $\hat{C}_{pl}$ which are the one-sided process capability ratios. $\hat{C}_{pk}$ is expressed as follows:

$$\hat{C}_{pk} = \min\{\hat{C}_{pl}, \hat{C}_{pu}\}, \qquad (11)$$

where

$$\hat{C}_{pl} = \frac{\hat{\mu} - \text{LSL}}{3\hat{\sigma}}, \qquad (12)$$

$$\hat{C}_{pl} = \frac{\text{USL} - \hat{\mu}}{3\hat{\sigma}}, \qquad (13)$$

where $\hat{\mu}$ and $\hat{\sigma}$ are the estimated process mean and

standard deviation, respectively. Furthermore, the multiple capability index, MC_{pk}, for a complete process of Q responses considered concurrently is calculated as

$$MC_{pk} = \left(\prod_{q=1}^{Q} C_{pk_q}\right)^{1/Q}. \qquad (14)$$

The $\hat{C}_{pk}$ values for turbidity and SVI are compared at the initial and optimal factor settings, and then the results and improvement analysis are displayed in Table 6.

Table 5 Summary of results from control charts

Response		MA			MR		
		UCL	CL	LCL	UCL	CL	LCL
y_1	Before	24.81	20.22	15.64	7.964	2.438	0.0
	After	9.116	6.42	3.724	4.683	1.433	0.0
y_2	Before	92.69	79.92	67.15	22.19	6.79	0.0
	After	79.34	72.15	64.97	12.48	3.82	0.0

Table 6 Improvement analysis*

Response	LSL	USL	$\hat{\mu}$	$\hat{\sigma}$	$\hat{C}_{pk}$	MC_{pk}
y_1	0	30	20.22 (6.42)	2.16 (1.27)	1.34 (5.5)	1.95
y_2	50	100	79.69 (72.15)	6.02 (3.39)	1.46 (1.93)	(10.6)

* Values at optimal settings are in parentheses

From Table 6 it is noted that:

1. For turbidity and SVI, the estimated process means at initial (optimal) factor settings are 20.22 (6.42) NTU and 79.69 (72.15) ml/g, respectively. The

process means at optimal factor settings are improved significantly as they are closer to the desired values of less than 15 NTU and 70 ml/g, respectively.

2. The process standard deviations at initial (optimal) factor settings are 2.16 (1.27) NTU and 6.02 (3.39) ml/g for turbidity and SVI, respectively. Clearly, optimal factor settings results in significant variability reductions are 41.22% and 77.75% for turbidity and SVI, respectively.

3. The $\hat{C}_{pk}$ values are improved significantly due to setting factor levels at optimal settings. That is, the $\hat{C}_{pk}$ value for turbidity is improved from 1.34 to 5.5, while the $\hat{C}_{pk}$ value for SVI is enhanced from 1.46 to 1.93. These results indicate that the wastewater treatment process becomes highly efficient for both responses due to setting the factor at optimal levels.

4. The MC_{pk} is improved significantly from 1.95 to 10.6, which also indicates that the process become highly capable.

4 Conclusions

The PAT framework is successfully implemented to improve the performance of the wastewater treatment process for two main quality characteristics: turbidity and SVI. In this framework, the MA and MR control charts are used to assess the performance of the process at initial factor settings, where the control charts indicate in-control states for both responses. The 3^3 full factorial design is used for conducting experimental work. The weighted additive model is proposed, and then used for optimizing process performance while considering engineers' preferences for factor settings and quality responses. Each response and process factor is described by suitable MF. The corresponding goal constraints are then defined. The objective function is used to minimize the weighted sum of the positive and negative deviations. Results show that the optimal process conditions found are flocculant dose of 18 mg/L, coagulant dose of 40.0 mg/L, and pH of 4.0. These parameters yield optimal values of turbidity and SVI of 6.184 NTU and 73.21 ml/g, respectively. Confirmation experiments are conducted at optimal factor settings. It is found

that: (1) the process means for turbidity and SVI at optimal factor settings are closer to the desired values of less than 15 NTU and 70 ml/g, respectively; (2) process variability is significantly reduced by 41.22% and 77.75% for turbidity and SVI, respectively, and the MC_{pk} is improved significantly from 1.95 to 10.6, which indicates that the process become highly capable. In conclusion, the weighted additive model is found to be an efficient technique for optimizing the performance of processes with multiple responses, taking into consideration the engineers' preferences about process settings.

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中文概要：

本文题目：在废水处理过程中基于过程分析技术的多重响应优化

Applying process analytical technology framework to optimize multiple responses in wastewater treatment process

研究目的： 基于过程分析技术框架，优化家禽业的废水处理工艺的性能。

研究方法： 生产商主要关注浊度和污泥容积指数（SVI）这两个响应。首先为每个响应建立移动平均（MA）和移动范围（MR）控制图，并在实验工作中重复两次 3^3 全因子设计。基于模糊目标规划建立加权相加模型，并用于确定最佳的因子设置组合。最后，在最佳的因子设置组合条件下，对实验结果进行跟踪确认。

重要结论： 在絮凝剂为18 mg/L、凝结剂为40.0 mg/L和pH=4.0的最佳工艺条件下，得到的浊度和污泥体积指数的最佳值分别为6.184 NTU和73.21 ml/g。在此条件下得到的浊度和污泥沉降指数能够满足设计要求，它们的过程变异性分别显著下降了41.22%和77.75%，且多重能力指数从1.95显著增加至10.6。这表明此过程可行性很高。总之，加权相加模型是一种用于优化多个响应流程性能的有效技术，并且可以考虑工程师的首选设置流程。