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Intelligent setting of ORP under multiple operation modes

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Abstract: In the cobalt removal process, the setting value of oxidation-reduction potential (ORP) directly affects cobalt remioval performance. Consider the multimode feature, an intelligent setting strategy for ORP is proposed. First, a basic ORP setting model is obtained based on mechanism knowledge. Then, an adaptive operation mode division method based on the parameter similarity is proposed. The model parameters for each mode are identified by an intelligent optimization algorithm. Finally, an intelligent online setting framework for ORP is constructed. The experiments verify the validity of the model.

Keywords: Cobalt removal process, Parameter similarity, Operation mode division, Oxidation–reduction potential, Intelligent setting.

1. INTRODUCTION

Zinc hydrometallurgy mainly consists of roasting, leaching, purification, electrolysis, and other processes, as shown in Fig. 1. Purification is a vital part of zinc hydrometallurgy. Impurity ions such as cobalt, nickel, copper, and cadmium in the solution have a significant impact on the efficiency of electrolysis (Bøckman and Østvold (2000), Tozawa et al. (1992)). The complexity of the cobalt removal reaction makes it difficult to remove cobalt ions (Nelson et al. (2000)). It is known from production experience that the oxidation-reduction potential (ORP) is a comprehensive characteristic reflecting the state of the cobalt removal reaction. Its value can be detected online by a testing instrument, which is called ORP detection value (ORP_det). Meanwhile, the operator determines a reference value of ORP in advance, which is called ORP setting value (ORP_set). To remove cobalt ions, the cobalt removal strategy is for the operator to make the ORP_det value as close as possible to the ORP_set value by adjusting the amount of zinc powder added. Therefore, the ORP_set value is the primary basis for the operator to control the cobalt removal process (CRP).

The complexity of CRP increases the difficulty of determining a reasonable ORP_set value. An unreasonable ORP_set value will bring the wrong guidance to operators, resulting in substandard outlet cobalt ion concentration and wasting zinc powder. Some scholars have done some studies related to the difficulty of purification. Dreher et al. (2001) studied the effects of solution temperature and additive concentration on CRP, from which the optimal solution temperature, additive concentration, and zinc powder addition in CRP were obtained. Sun et al. (2013) introduced the ORP_det value into the kinetic model to achieve online prediction of the outlet cobalt ion concentration. Sun et al. (2014) studied the application of case retrieval in the abnormal state of CRP and obtained the optimal zinc powder addition and ORP_set value in CRP. In the copper removal process, Zhang et al. (2016) established a kinetic model to ensure stable production of the process by analyzing and evaluating the levels of ORP_det level. These research results provided the basis for parameter optimization of CRP. (Polcaro et al. (1995), Van der Pas and Dreisinger (1996), Dib and Makhloufi (2006)). Many scholars have studied how to optimize or predict other parameters based on the ORP_det value, but only a few studied the ORP_set value. This paper proposes an intelligent strategy for ORP settings based on previous studies by scholars.

The rest of the paper is organized as follows. Section 2 introduces the CRP and identifies the challenges of strategy. Section 3 details the critical steps of the strategy.

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Section 4 verifies the validity of the model. Finally, the conclusion is given in Section 5.

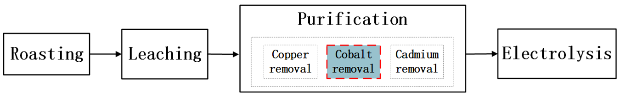

Fig. 1. Zinc hydrometallurgy process

2. PROCESS ANALYSIS

2.1 Process description

A brief flow chart of CRP is shown in Fig. 2. The CRP consists of five continuously stirred reactors and a thickener. Under high temperature and acidic conditions, zinc powder and arsenic salts continuously added to the reactor undergo a complex oxidation-reduction reaction with cobalt ions and copper ions in solution. The reaction produces an alloy of cobalt, arsenic, copper, zinc and other metals, thus achieving the purification purpose.

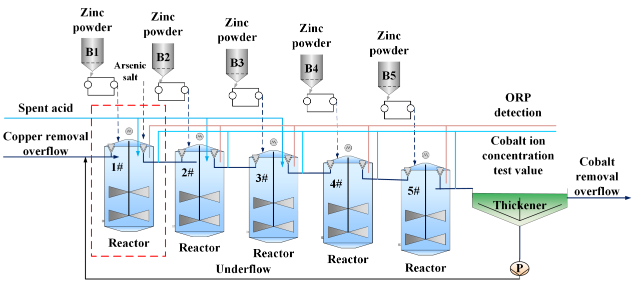

Fig. 2. Cobalt removal process

The main chemical reactions of CRP are as follows:

$$As_2O_3{+}2NaOH = 2NaAsO_2{+}H_2O \tag{1}$$

$$AsO_2^-{+}H^+{=}\ HAsO_2 \tag{2}$$

$$Cu^{2+}{+}Zn = Zn^{2+}{+}Cu \tag{3}$$

$$HAsO_2{+}3Cu^{2+}{+}3H^+{+}4.5Zn = $$
$$Cu_3As + 4.5Zn^{2+}{+}2H_2O \tag{4}$$

$$HAsO_2{+}Co^{2+}{+}3H^+{+}2.5Zn = $$
$$CoAs + 2.5Zn^{2+}{+}2H_2O \tag{5}$$

$$HAsO_2{+}Ni^{2+}{+}3H^+{+}2.5Zn = $$
$$NiAs + 2.5Zn^{2+}{+}2H_2O \tag{6}$$

where equations (3) to (6) are the main reactions of CRP, and equations (1) and (2) describe the preparation of arsenic salt.

In CRP, zinc powder is the reactant of the impurity removal reaction, and its addition directly affects the state of the reaction and the outlet cobalt ion concentration. The ORP_set value is an important basis for the operator to control zinc powder addition. The more negative the value is, and the more zinc powder the operator adds. Conversely, the less. In this way, the ORP_set value is particularly important.

2.2 *The challenges of setting* ORP_set *value*

In production, the operator determines the ORP_set value of the reactor based on personal experience and production planning, which is subjective. By studying the reaction mechanism of CRP and establishing the relationship model between operating parameters and process indexes, we can not only avoid the subjectivity of operator experience but also provide an effective way to set the ORP_set value. The challenges of setting the ORP_set value are as follows:

(1) Complex reaction mechanism and process time lag. CRP involves multiple reactions, and some of them have both competitive and facilitative relationships with each other. In addition, due to the large volume of the reactor, the adjustment of the operating parameters cannot immediately affect the outlet cobalt ion concentration. That is, the CRP shows a significant time lag.

(2) Unstable process parameters. Due to the unstable composition of the zinc concentrate and its own process defects, it causes fluctuations in the inlet cobalt and copper ion concentrations in the 1# reactor, as shown in Fig. 3. In addition, due to the production conditions, the solution flow rate and ORP_det value of the 1# reactor are also unstable, as shown in Fig. 4.

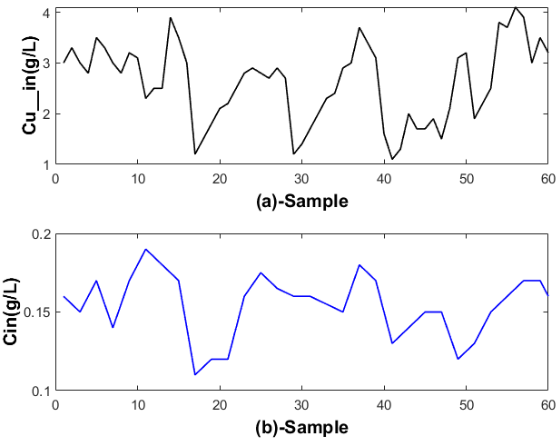

Fig. 3. Some parameters of the 1# reactor: (a) Inlet copper ion concentration, (b) Inlet cobalt ion concentration concentration

(3) Coupling and nonlinearity between parameters. Many parameters in CRP are strongly nonlinear and strongly coupled with each other. There is a strong correlation between the front and rear reactors. There is a strong correlation between the pre-reactors and post-reactors. Even with the same amount of zinc powder addition, the ORP_set value of each reactor is different, and the effect of cobalt removal can be significantly different.

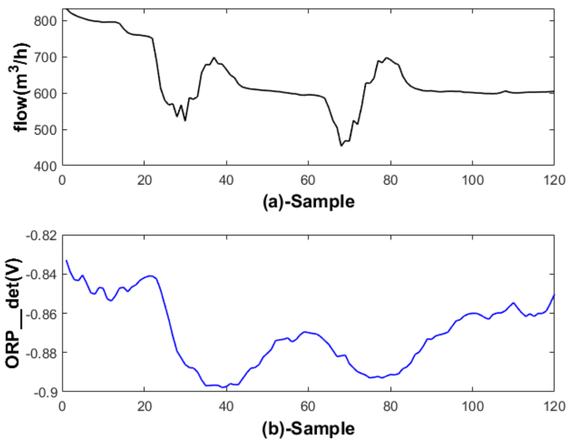

Fig. 4. The 1# reactor: (a)Flow, (b)ORP_det value

In response to the above challenges, considering both the complex reaction mechanism and the state-variable process, an intelligent setting strategy based on parameter similarity for multiple operation modes for ORP_set value in the cobalt removal process is proposed in this paper.

3. AN INTELLIGENT SETTING STRATEGY FOR ORP

3.1 Establish a basic ORP_set *value model*

In CRP, the cobalt removal reaction can be described by a first-order kinetic model as follows:

$$\frac{dc}{dt} = -kc \tag{7}$$

where c represents the current outlet cobalt ion concentration and k represents the cobalt removal reaction rate.

k can be expressed by the Arrhenius formula as follows:

$$k = A_0 \exp(-\frac{E_a}{RT}) \tag{8}$$

where A_0 is the pre-exponential factor, E_a is the reaction activation energy, R is the ideal gas constant, and T is the reaction temperature.

Assuming that the material in the reactor is completely mixed and uniformly distributed in all positions and directions, the first-order kinetic model for the cobalt removal reaction can be rewritten based on material equilibrium, as follows:

$$\frac{dc}{dt} = \frac{f_{in}}{V} c_{in} - \frac{f_{out}}{V} c - kc \tag{9}$$

Where V represents the reactor volume, f_{in} and f_{out} represent the inlet and outlet flow rates of the reactor, respectively, c_{in} represents the inlet cobalt ion concentration.

The oxidation-reduction reaction occurring in the reactor can be divided into multiple parallel electrode reactions as follows:

Anode reaction:

$$\mathrm{Zn} = \mathrm{Zn}^{2+} + 2\mathrm{e}^{-} \tag{10}$$

Cathodic reaction:

$$\mathrm{Cu}^{2+} + 2\mathrm{e}^{-} = \mathrm{Cu} \tag{11}$$

$$\mathrm{Co}^{2+} + 2\mathrm{e}^{-} = \mathrm{Co} \tag{12}$$

$$\mathrm{Ni}^{2+} + 2\mathrm{e}^{-} = \mathrm{Ni} \tag{13}$$

$$2\mathrm{H}^{+} + 2\mathrm{e}^{-} = \mathrm{H}_2 \tag{14}$$

The complex electrode reactions follow independent and parallel reaction principles (Antropov (1972)), and the only common feature of the parallel reactions is the detectable mixing potential, i.e., ORP, in solution. The change in ORP will affect the reaction activation energy, thereby affecting the reaction rate k.

According to the electrode reaction kinetics, the cathodic reaction is shown in equation (15).

$$O + n\, e^{-} \rightarrow R \tag{15}$$

where O represents the substance before reduction, and R represents the substance after reduction, e^{-} represents electrons, and n represents the number of transferred electrons. When 1 mol of new material is generated, the nF Coulomb's positive charge (F is the Faraday constant) is moved to the electrode. If the electrode potential increases by $\Delta\varphi$, the total potential energy of the reaction product will increase by $nF\Delta\varphi$, and the activation energy of the cathodic reaction increases by $\gamma nF\Delta\varphi$ $(0 < \gamma < 1)$. The activation energy of the cathodic reaction is as follows:

$$E_a = E_{a0} + \gamma nF\Delta\varphi \tag{16}$$

where γ characterizes the effect of the electrode potential change on the reaction activation energy, which is called the cathode transfer coefficient, E_{a0} is the standard reaction activation energy.

$\Delta\varphi$ is related to the ORP and equilibrium potential of the solution, as follows:

$$\Delta\varphi = ORP - \varphi_{eq} \tag{17}$$

where φ_{eq} represents the equilibrium potential of cobalt ions.

Substituting equations (8), (11) and (12) into equation (9), the first-order kinetic model for the cobalt removal reaction can be rewritten as follows:

$$\begin{aligned} \tfrac{dc}{dt} &= \tfrac{f_{in}}{V} c_{in} - \tfrac{f_{out}}{V} c \\ &- A_0 \exp(-\tfrac{E_{a0} + \gamma nF(ORP - \varphi_{eq})}{RT}) c \end{aligned} \tag{18}$$

In CRP, taking $n = 2$, $f_{in} \approx f_{out} = f$ and $A = A_0 \exp(\frac{\gamma nF\varphi_{eq} - E_{a0}}{RT})$, equation (18) can be simplified as follows:

$$\frac{dc}{dt} = \frac{f}{V}(c_{in} - c) - A\exp(-\frac{2\gamma F}{RT} \bullet ORP)c \tag{19}$$

where both A and γ are unknown parameters that need to be identified.

The properties of each parameter in equation (19), such as physical meaning, acquisition method and so on, are shown in Table 1.

To simplify the description, the first-order kinetic model is abbreviated as $\boldsymbol{f}_K(t, \boldsymbol{P}_M, \boldsymbol{P}_K)$, where $\boldsymbol{P}_M$ represents the known parameters and $\boldsymbol{P}_K$ represents the unknown parameters to be identified.

On the time interval $[t_0, t]$, let:

$$dc = c_t - c \tag{20}$$

$$dt = t - t_0 \tag{21}$$

where c_t is the outlet cobalt ion concentration at time t.

Substituting equations (20) and (21) into equation (19), the ORP_set value model can be obtained:

$$ORP\_set = -\frac{RT}{2\gamma F} \bullet \ln \frac{f(c_{in} - c)/V - (c_t - c)/(t - t_0)}{Ac} \tag{22}$$

Table 1. Parameter properties

Parameter	Units	Meaning	Access
c	mg/L	Outlet cobalt ion	Testing
c_{in}	mg/L	Inlet cobalt ion	Testing
f	m^3/h	Flow	Detection
ORP	V	Potential	Detection
T	K	Temperature	Detection
F	C/mol	Faraday constant	Constant
R	J/(mol $*$ K)	Ideal gas constant	Constant
V	m^3	Reactor volume	Constant
γ	1	Transfer coefficient	Identify
A	1		Identify

3.2 Operation mode division and parameter identification

The model parameters vary with the reaction state, so a model with fixed parameters cannot accurately describe a variable process. Therefore, the model parameters are optimized separately for different operation modes. Under a specific operation mode, the model parameters vary very little and can even be considered constant (Zhang et al. (2013)). To better describe the difference of parameters under different operation modes, the concept of model parameter similarity (PS) is proposed in this paper, which is defined as follows:

$$PS = 1 - e \tag{23}$$

where $e = \left|c^{\boldsymbol{f}_K} - c^{real}\right| / c^{real}$, $c^{\boldsymbol{f}_K}$ is the outlet cobalt ion concentration under the ORP_set value model and c^{real} is the outlet cobalt ion concentration under the operator. The larger the PS value of each sample is, the more similar the corresponding cobalt removal reaction state of each sample is, then these samples belong to a certain operation mode.

In view of the difficulty for operators to classify industrial data into the different operation modes, a method based on the PS to adaptively classify operation modes is proposed in this paper, as shown in Fig. 5. The specific steps are as follows:

Step 1: Production data containing various operation modes are randomly selected to form the initial sample set $\mathbf{S}_0 = \left\{\boldsymbol{s}_i^0 | i = 1, ..., n\right\}$, where $\boldsymbol{s}_i^0 = \left\{s_{i,j}^0 | j = 1, ..., m\right\}$ is the i-th sample and m is the sample dimension. Set the PS threshold θ_{PS}, the working condition sample size θ_{CS} and the optimization range $\{A, \gamma | A_{\min} \le A \le A_{\max}, \gamma_{\min} \le \gamma \le \gamma_{\max}\}$.

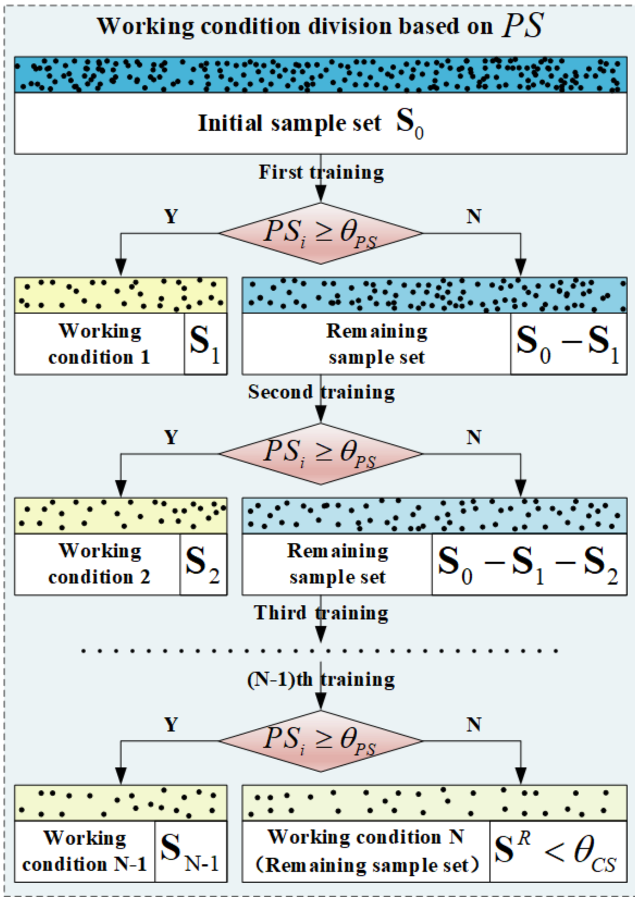

Fig. 5. Operation mode division diagram

Step 2: By training a first-order dynamics model on $\mathbf{S}_0$ using an intelligent optimization algorithm, $\boldsymbol{P}_K$ and $\boldsymbol{f}_K^{S_0}(t, \boldsymbol{P}_M, \boldsymbol{P}_K)$ are obtained. The parameter similarity corresponding to each sample is calculated: $PS_i = 1 - \left|c_i^{\boldsymbol{f}_K} - c_i^{real}\right| / c_i^{real}$.

Step 3: Select samples whose PS_i are greater than θ_{PS} from $\mathbf{S}_0$ to form the first type of operation mode sample set $\mathbf{S}_1 = \left\{\boldsymbol{s}_i^1 | i = 1, ..., n_1, PS(\boldsymbol{f}_K^{S_0}(s_i^1)) \ge \theta_{PS}\right\}$. The remaining samples are formed a new training sample set $\mathbf{S}_1^{\mathrm{R}} = \mathbf{S}_0 - \mathbf{S}_1$.

Step 4: Whether the sample size of $\mathbf{S}_1^{\mathrm{R}}$ is smaller than θ_{CS} or not.

Step 5: If the sample size of $\mathbf{S}_1^{\mathrm{R}}$ is larger than θ_{CS}, repeat steps 2 to 4 on $\mathbf{S}_1^{\mathrm{R}}$ to obtain sample sets of various operation mode until the sample size of $\mathbf{S}_1^{\mathrm{R}}$ is smaller than θ_{CS}. If the sample size of $\mathbf{S}_1^{\mathrm{R}}$ is smaller than θ_{CS}, stop classifying the operation mode and use $\mathbf{S}_1^{\mathrm{R}}$ as the last type of operation mode sample set. Finally, $\mathbf{S}_0$ is divided into operation mode sample set $\{\mathbf{S}_1, \mathbf{S}_2, ..., \mathbf{S}_{\mathrm{N}}\}$.

Selecting $n_{identify}$ sets of identification samples, the goal of parameter identification is to find a set of optimal $\boldsymbol{P}_K$ such that the sum of PS for each sample is the largest:

$$\max\ J_{PS}(\boldsymbol{P}_K) = \sum_{i=1}^{n_{identify}} \left(1 - \left|c_i^{f_K} - c_i^{real}\right| \Big/ c_i^{real}\right) \quad (24)$$

Since the first-order dynamics model is nonlinear for $\boldsymbol{P}_K$, the particle swarm algorithm based on group activity perception (Kennedy and Eberhart (1995), Liang et al. (2006), Li (2009), Zhan et al. (2009)) is used to identify the unknown parameters. After identification, the optimal parameter matrix is obtained: $\{\boldsymbol{P}_{K,1}, \boldsymbol{P}_{K,2}, ..., \boldsymbol{P}_{K,N}\}$.

3.3 An intelligent online setting framework for ORP

On the time interval $[t_0, t]$, the expected value of the outlet cobalt ion concentration at time t is set as c_{t_target}. Combining c_{in}, c, and f, the field data is abbreviated as $data(f, c_{in}, c, c_{t\_target})$. To determine the ORP_set value online, an intelligent online setting framework for the ORP_set value is designed, which is composed of a working condition sensor, a parameter selector and the ORP_set value model, as shown in Fig. 6.

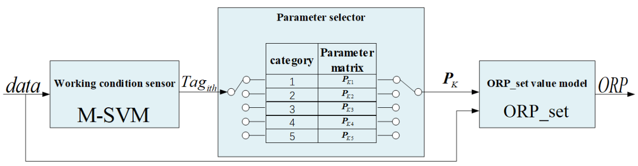

Fig. 6. Intelligent online setting framework for ORP

In order to obtain a operation mode sensor with $data(f, c_{in}, c, c_{t.}$ as the input and Tag_{ith} as the only output, the multi_class support vector machine algorithm (M-SVM)(Weston and Watkins (1998)) is used to train the sample set $\{\mathbf{S}_1,\mathbf{S}_2,...,\mathbf{S}_\mathrm{N}\}$. The parameter selector selects the corresponding parameters P_K from the parameter matrix according to Tag_{ith} and sends them to the ORP_set value model. The ORP_set value model receives P_K and $data$ to calculate the ORP_set value.

4. EXPERIMENTAL STUDY

The 1# reactor was selected as the experimental object, and 900 sets of production data were randomly selected as the sample set S_0, which contained the solution flow rate f, ORP_det value, inlet cobalt ion concentration c_{in}, and outlet cobalt ion concentration c (The data is desensitized for confidentiality reasons. In the result figures, the vertical axis values are scaled.). In the experiment, let $\theta_{PS} = 0.9$ and $\theta_{CS} = 200$. Through the method introduced in Section 3.2, the sample set S_0 is divided into five types of operation modes, and the model parameters under each operation mode are identified. The results are shown in Table 2.

To verify the validity of the ORP_set value model, 36 sets of production data (3 days in duration) without breakpoints were randomly selected as validation samples. The ORP_set values determined by the model and operator, respectively, are shown in Fig. 7. The outlet cobalt ion concentration of 1# reactor is the key technical index to evaluate the superiority of ORP_set value, which is lower than the production index ($0.045g/L$). Under the two ways of determining the ORP_set value, the outlet cobalt ion concentration of the 1# reactor is shown in Fig. 8.

Table 2. Parameter $\boldsymbol{P}_K$ identification results

Category	A	γ
Condition 1	6.9680	0.00337
Condition 2	8.5145	0.00325
Condition 3	9.8935	0.00193
Condition 4	10.8095	0.00294
Condition 5	6.1385	0.00274

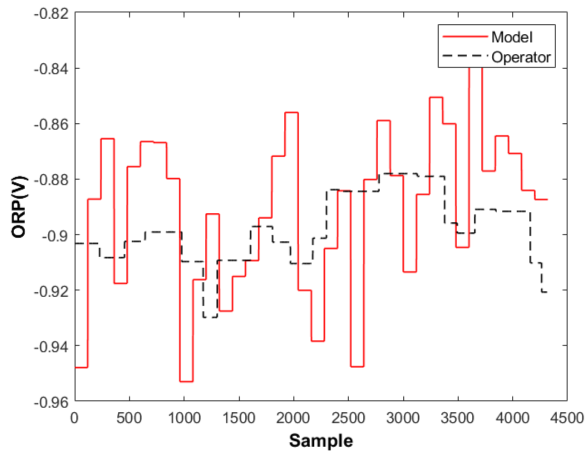

Fig. 7. ORP_set value comparison result

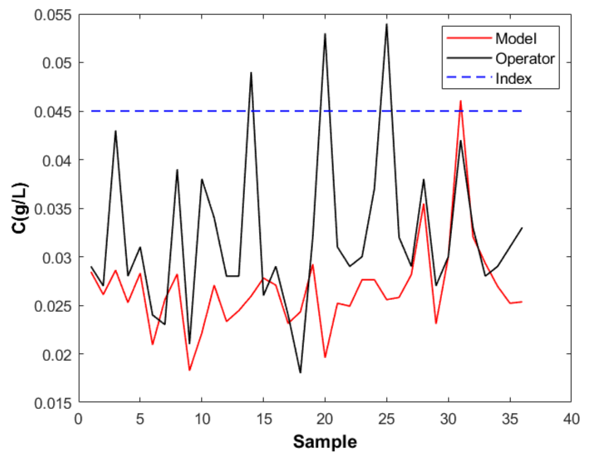

Fig. 8. The outlet cobalt ion concentration comparison result

To quantitatively evaluate the differences between the two ways of determining ORP_set values, four evaluation indicators are introduced: sensing sensitivity (SS), compliance rate (CR), standard deviation (σ) and high quality rate (HQR) of the outlet cobalt ion concentration. SS is the ratio of the effective adjustment times of the ORP_set value to the validation sample size, which is used to describe the accuracy of the ORP_set value sensing changes in operation modes, as follows:

$$SS = \frac{\sum sign_{SS}}{N_{samples}} \quad (25)$$

$$sign_{SS} = \begin{cases} 1 & if\,|\Delta ORP_{set}| \geq 0.005 \\ 0 & else \end{cases} \quad (26)$$

where $N_{samples}$ is the validation sample size, $|\Delta ORP_{set}|$ represents the absolute value of the difference between adjacent ORP_set values, and $sign_{SS} = 1$ represents an effective adjustment.

The expression of HQR is as follows:

$$HQR_{Model} = \frac{\sum sign_M}{N_{samples}} \tag{27}$$

$$HQR_{Operator} = \frac{\sum sign_O}{N_{samples}} \tag{28}$$

$$sign_M = \begin{cases} 1 \; if \; C_M \leq C_O \& C_M \leq 0.045 \\ 0 \;\; else \end{cases} \tag{29}$$

$$sign_O = \begin{cases} 1 \; if \; C_O \leq C_M \& C_O \leq 0.045 \\ 0 \;\; else \end{cases} \tag{30}$$

where C_M is the outlet cobalt ion concentration corresponding to the model, and C_O is the outlet cobalt ion concentration corresponding to the operator. The comparison results of different ways to determine the ORP_set value are shown in Table 3. The model is more sensitive than the operator to sensing changes in operation modes and makes effective adjustments. The cobalt ion concentration compliance rate (CR) corresponding to the model is higher than the operator's. The σ indicates that the outlet cobalt ion concentration corresponding to the model is less fluctuating, and therefore, the model provides a more stable inlet condition for the cadmium removal process. HQR_{Model} is more significant than $HQR_{Operator}$, indicating that the model way is more effective in removing cobalt.

Table 3. Parameter properties

Index category	Model	Operator
SS	91.67%	41.67%
CR	97.22%	91.67%
σ	0.00467	0.00814
HQR	86.11%	13.89%

5. CONCLUSIONS

To solve the problem of how to determine the ORP_set value of CRP, an ORP_set value model is established in this paper based on the mechanism of cobalt removal reaction. An adaptive method of dividing the operation modes based on parameter similarity is proposed for the variable cobalt removal reaction, and an optimization algorithm identifies the model's unknown parameters under different operation modes. Finally, an online operation framework for the ORP_set value model is designed, and the validity and superiority of the strategy for determining ORP_set value is verified by production samples.

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