pH prediction of a neutral leaching process using adaptive-network-based fuzzy inference system and reaction kinetics

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Abstract: PH value is an important index to measure the quality of product in neutral leaching process (NLP). However, due to the harsh production environment, there is almost no pH measuring device that can be applied to the site for a long time. To solve this problem, an effective pH prediction method for NLP is proposed in this paper. Firstly, the reaction kinetics of the NLP was researched, and the mechanism models under different running conditions were established. Secondly, ANFIS (Adaptive-Network-Based Fuzzy Inference System) is used to establish the data models of the process based on the idea of fuzzy training. Finally, according to the characteristics of two models and the "model mismatch" phenomenon in NLP, an effective model integration method based on fuzzy membership of running conditions is proposed, and the optimal integration was realized. Data show that the integrated model has better predictive performance than a single one, and pH predictive output of the model can also provide effective guidance for NLP.

Keywords: hydrometallurgy, neutral leaching, mechanism model, ANFIS, running condition, fuzzy membership

1. INTRODUCTION

Zinc is an important strategic resource and also an important raw material for national production. Although there are multiple zinc preparation methods at present, zinc hydrometallurgy technology still occupies the mainstream position (Coelho et al. (2018), Xie, S et al. (2018)). Zinc hydrometallurgy is mainly composed of roasting, leaching, purification, electrolysis and some others secondary processes. Leaching process is a major process that plays a key role in zinc hydrometallurgy technology, while neutral leaching is the most important sub-process in leaching process.

Neutral leaching process (NLP) refers to a reaction control process in which zinc calcine, spent electrolyte, acid leaching supernatant, mixture, pyrolusite slurry and other reactive substances are added to a number of Continuous Stirring Reactor (CSTR), which act as reaction containers, to achieve the high degree of dissolution of zinc calcine. NLP requires a stable pH value at the outlet, too high pH value or too low pH value will affect the production efficiency of the process. The industrial site conditions of NLP are extremely harsh, pH detection device is difficult to be used for a long time. The pH meter which designed with automatic cleaning and maintenance function is not only more expensive, but also more difficult to maintain. At the same time, the NLP has a greater time delay, that is, the pH value at the outlet of the current reactor does not reflect the rationality of the operation, which also greatly increases the difficulty of the operation on site.

Process modeling is an important way to realize the prediction of parameters (Sun, B et al. (2013), Zhang, B et al. (2016)). At present, there are abundant researches on process modeling. The methods of complex industrial process modeling mainly include mechanism modeling, data-driven modeling and data-mechanism fusion modeling (Sun, B et al. (2018), Sun, B et al. (2015)). The mechanism model reflects the general internal rules of the object and it is a relatively reliable modeling method (Shi, B et al. (2014). But, for complex objects, a precise mechanism model is difficult to establish. Data model is established based on the accumulated data of the process operation, mining the internal relations of the object and approximating the real internal relations of the object in a deep level, such as neural network model, autoregressive model and so on (Carlos Alberto et al. (2017), Xie, S et al. (2018)). Data-mechanism fusion model is a method to combine the advantages of the two models. Many experiments have proved that the data-mechanism fusion model has better dynamic performance than a single one (Zurita, D et al. (2017)).

At present, there are few studies on the NLP, which are old or in the initial stage, and the research results are difficult to be applied to the actual production. The purpose of this paper is to establish a reliable pH prediction model for NLP and provide effective guidance for process production. In view of this and the problems in NLP, on the premise of in-depth study of the process mechanism, the mechanism models of the reaction tanks were established. Based on the process data and facts, the running conditions were classified and the mechanism models under different running conditions were identified. According to the idea of fuzzy training, the process data is input to *Adaptive-Network-Based Fuzzy*

Inference System (ANFIS) for training, and the fuzzy neural network model with certain generalization ability is obtained. Finally, an effective method of fuzzy integration under different running conditions was proposed. The fuzzy weighted integration of the two models under different running conditions was obtained to realize the online prediction of the pH value at the outlet of the reactor.

2. MECHANISM MODEL OF NEUTRAL LEACHING PROCESS

2.1 Neutral Leaching Process

The process of neutral leaching in a smelting plant can be summarized and simplified as shown in Fig. 1. It can be seen that the NLP is mainly composed of multiple cascaded CSTR, and the reactor presents high-low order arrangement. The process inputs include zinc calcine, spent electrolyte, acid leaching supernatant, mixture, pyrolusite slurry, etc. The outlet of each reaction tank is setting in the middle and lower part of the tank, and the input port is in the upper part of the tank. In order to monitor the pH value in the tank, a pH detection device is often installed at the outlet of the reaction tank. However, due to the unreliability of pH detection device, the actual pH value is mainly determined by manual timing detection on site. The stable temperature environment required for leaching is ensured by injecting heated steam into the system. Oxygen is also pumped into tanks 4# and 5# to further oxidize Fe^{2+} in solution. The ultimate control goal is to maintain a pH value between 4.8 and 5.2 at the outlet of the 5# reactor, so that the impurity ions in the solution have a stable precipitation environment, which provides necessary conditions for the subsequent purification process.

In the actual production process, the adjustable amount of the NLP is very little. In the input variables of the system, zinc calcine is determined by scheduling, the total amount of input is limited and cannot be changed easily. And in order to ensure the adequacy of leaching degree, zinc calcine is generally added in tank 1# or tank 2#, the zinc calcine in subsequent tanks is only reserved to deal with the abnormal running conditions of the system. The acidity of mixture and pyrolusite slurry is much lower than that of spent electrolyte. Therefore, the adjustable quantity on site is usually only spent electrolyte, and the waste acid addition pipeline and device are generally only set up in the 1# tank and 3# tank.

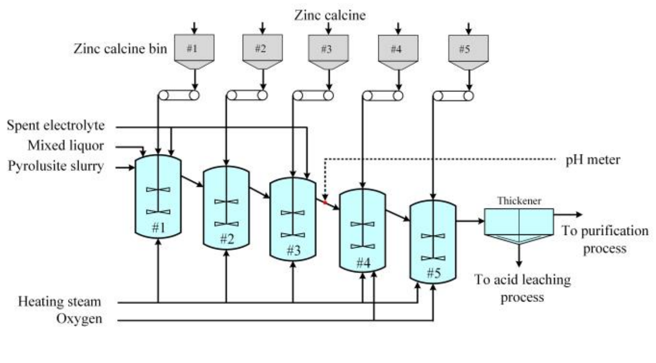

Fig. 1. A neutral leaching process

Through investigation and data analysis, it is found that the pH value at the outlet of each reaction tank tends to show a gradient upward trend for the field production conditions. In addition, under normal running conditions, no control operation is carried out after the 3# tank. If the pH value at the outlet of 3# tank can be controlled within a stable range, the final pH value at the outlet can be guaranteed.

2.2 System Mechanism Modeling

As shown in Fig. 1, all three reaction tanks have Zinc calcine outlets, waste acid is added in the 1# and 3# tanks, and pyrolusite slurry mixture is added in the 1# tank. So, the solid reactants that affect pH value mainly come from the solid substance in ore flushes such as zinc calcine and pyrolusite slurry.

For 1# reactor, we can set leaching process reaction surface area as $A^{1\#}$ in 1# reactor. As shown in Fig. 1, since only pH value is considered here, there is the following material balance equation for hydrogen ions

$$V\frac{dc^{1\#}}{dt} = F_{Mn}c_{Mn} + F_{mix}c_{mix} + F_{acid}^{1\#}c_{acid} - F_{out}^{1\#}c^{1\#} - VkA^{1\#}c^{1\#}. \quad (1)$$

Where, F_{Mn} , F_{mix} , $F_{acid}^{1\#}$ and $F_{out}^{1\#}$ are respectively the flow rate of manganese ore slurry, the flow rate of mixed liquid, the flow rate of waste acid into tank 1# and the flow rate at the outlet of tank 1#. c_{Mn} , c_{mix} , c_{acid} and $c^{1\#}$ represent respectively the acidity of manganese ore slurry, the acidity of mixed solution, the concentration of waste acid, and the concentration of hydrogen ion in 1# reactor. According to *Arrhenius Equation*, we can get

$$k = A_0 \exp\left(-E/RT\right). \quad (2)$$

At the same time, for the surface area of the effective reaction substance, it can be deduced as follows. The solids entering the reactor are mainly zinc calcine and other solids in solution (Sun, B et al. (2016)). If the quality of zinc calcine in 1# tank is $m^{1\#}$, then

$$A_1^{1\#} = \alpha^{1\#} \cdot [(m^{1\#}/\rho)/(4\pi r_0^3/3)] \cdot 4\pi r_0^2 = \alpha^{1\#} \cdot 3m^{1\#}/(\rho r_0). \quad (3)$$

Where $A_1^{1\#}$ is the effective substrate surface area of zinc calcine, ρ is the density of zinc calcine, r_0 is the particle radius of zinc calcine, and $\alpha^{1\#}$ is the proportion of the effective substrate surface area of zinc calcine in 1# reactor. Suppose the effective surface area of solid substance in pyrolusite slurry and other solutions in 1# reactor is $A_2^{1\#}$, then

$$A^{1\#} = A_1^{1\#} + A_2^{1\#}. \quad (4)$$

By combining (1) (2) (3) (4), we can get

$$V\frac{dc^{1\#}}{dt} = F_{Mn}c_{Mn} + F_{mix}c_{mix} + F_{acid}^{1\#}c_{acid} - F_{out}^{1\#}c^{1\#} \\ -VA_0\exp\left(-E/RT\right)\left(a^{1\#}\cdot 3m^{1\#}/(\rho r_0) + A_2^{1\#}\right)c^{1\#}. \tag{5}$$

Let's set

$$\zeta\left(\theta_{id}^{1\#}, \theta_m^{1\#}\right) = -\left(F_{out}^{1\#}/V + a^{1\#}m^{1\#} + b^{1\#}\right) \tag{6}$$

$$\zeta\left(F_{acid}^{1\#}, F_{mix}, F_{Mn}\right) = (F_{Mn}c_{Mn} + F_{mix}c_{mix} + F_{acid}^{1\#}c_{acid})/V. \tag{7}$$

Where

$$a^{1\#} = A_0\exp\left(-E/RT\right)3\alpha^{1\#}/\rho r_0 \quad ,b^{1\#} = A_0\exp\left(-E/RT\right)A_2^{1\#}$$

$\theta_{id}^{1\#} = \left(a^{1\#}, b^{1\#}\right)$ is the unknown parameter to be identified, $\theta_m^{1\#} = \left(F_{out}^{1\#}, m^{1\#}\right)$ is the parameter that can be detected online. Therefore, (5) can be written into

$$\frac{dc^{1\#}}{dt} = \zeta\left(F_{acid}^{1\#}, F_{mix}, F_{Mn}\right) + \zeta\left(\theta_{id}^{1\#}, \theta_m^{1\#}\right)c^{1\#}. \tag{8}$$

Solve this first-order linear non-homogeneous differential equation, result can be found:

$$c^{1\#} = \exp(\zeta\left(\theta_{id}^{1\#}, \theta_m^{1\#}\right)(t - t_0))c_0^{1\#} + \exp(\zeta\left(\theta_{id}^{1\#}, \theta_m^{1\#}\right)t) \\ \cdot\int_{t_0}^{t}\exp(-\zeta\left(\theta_{id}^{1\#}, \theta_m^{1\#}\right)\tau)\zeta\left(F_{acid}^{1\#}, F_{mix}, F_{Mn}\right)d\tau. \tag{9}$$

Where t_0 is the initial time and $c_0^{1\#}$ is the acidity at the exit of the 1# reactor at the initial time.

It can be seen from Fig. 1 that the neutral leaching is cascaded by multiple reactors. So the modeling methods for another two reactors are similar to the 1# reactor.

2.3 Parameter identification and Model validation

Due to the varying conditions of the NLP, the parameters of the model will drift with different acidity. Then, a single mechanism model cannot reflect the running states of the system under all running conditions, nor can it achieve good model accuracy. At the same time, it is difficult to implement the online identification of the model because of the difficulty in obtaining enough online data in the site (Zhang, B et al. (2017)). Therefore, this paper classifies the running conditions according to the actual field operation, and obtains the dynamic mathematical models under different running conditions by offline identification. The specific running condition classification and method can be expressed as Fig. 2.

Where $F_{acid}^{1\#}$ and $F_{acid}^{3\#}$ are the amount of waste acid entering the 1# and 3# reactor, m_i is the quality of calcine entering the *i*# reactor. Electrolytic cutting is a special condition of neutral leaching, which means cleaning the electrolytic cell in the electrolytic process. For a smelter in China, pyrolusite slurry is directly prepared by using electrolytic waste liquid in the electrolytic process. When electrolytic cutting is carried out, the acidity of pyrolusite slurry will greatly increase, which has a great impact on the control of pH value. Electrolytic cutting signal is generally obtained offline, and there will be relevant reminders before cutting.

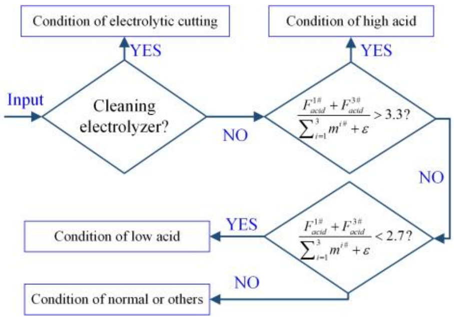

Fig. 2. Classification of running conditions

By manually measuring the pH value in the factory, we obtained several sets of data at intervals of 10 minutes. After running condition classification, data processing and delay correction, 400 groups of data were taken for parameter identification, there are 100 groups of data each running condition, 100 groups of data were taken for result verification, and 25 groups of data were taken for each running condition. Based on *least square method, State Transition Algorithm* (STA) is used for parameter identification (Zhou, X et al. (2012)). The final smoothing results, the data verification results after processing in different running conditions and error analysis are shown in Fig. 4 and Table 1.

3. PH PREDICTION BASED ON ANFIS

The mechanism of the NLP was analyzed above, and the running conditions were divided based on the actual operation, so as to obtain the mechanism model of the system under different running conditions. However, the accuracy of pH value measured by pH meter or pH test paper in the NLP is uncertain. Therefore, the traditional quantitative analysis method based on numerical value has certain attribute defects. Based on this, the method based on qualitative analysis can be further used to improve the robustness of the modeling results.

ANFIS (Adaptive-Network-Based Fuzzy Inference System) is a fuzzy inference system based on T-S model, which was proposed by Jang (Jang, J. S. (1993)). It combines the reasoning ability of the fuzzy system with the approximation ability of the neural network, and uses the self-learning ability of the neural network to adjust the parameters of the former and the latter parts of the fuzzy reasoning system, so that the system has higher fuzzy reasoning accuracy and better generalization performance (Mashallah Rezakazemi et al. (2017), Poul, A. K et al. (2019)). It has been proved that ANFIS is capable of approximating nonlinear functions with arbitrary precision (Walia, N et al. (2015)). Therefore, aiming at the above problems, ANFIS is used to build the systematic prediction model based on the idea of fuzzy training.

The typical ANFIS structure is shown in Fig. 3, which mainly consists of five layers. Here, the definition and setting of each layer are as follows for the NLP.

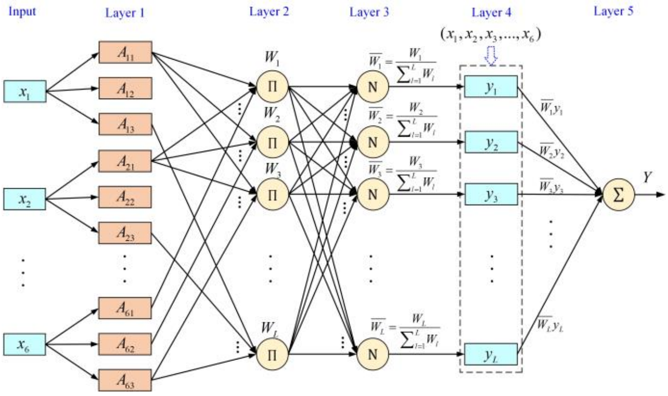

Fig. 3. Structure of ANFIS for pH prediction in NLP

As before, 500 sets of data were collected in the NLP, of which 400 were used for training and the remaining 100 were used for verification. Among the 400 sets of training data, each running condition was 100 sets and 25 groups in each running condition in 100 groups of verification data. Set 400 sets of training data as

$$\mathbf{X}=\left[\mathbf{X_1},\mathbf{X_2},\mathbf{X_3},\ldots,\mathbf{X_n}\right],n=400. \tag{10}$$

Where $\mathbf{X_i}=\left[x_1,x_2,x_3,x_4,x_5,x_6,y\right]$. Due to the large time delay of the CSTR, the accumulated amount of the past period of time was used as the input for training. $x_1 \sim x_6$ are respectively the accumulated value of calcine added in 1# reactor, calcine added in 3# reactor, mixture, pyrolusite slurry, spent electrolyte added in 1# reactor and spent electrolyte added in 2# reactor in the first half hour of pH detection, y indicating the pH value at the outlet of 3# reactor. Hybrid method was used for data training and the final training models were obtained after 100 iterations for each running condition. The training results and error analysis are shown in Fig. 4 and Table 1.

4. MODEL INTEGRATION

The system mechanism model and the fuzzy neural network model based on ANFIS were obtained. However, in the actual industrial process, a single model has high risks in operation, so the method of model integration is often adopted to improve the reliability of the model. At the same time, in the actual industrial process, the quality of the model is not determined by a single index. Model integration can often make up for the shortcomings of some performance of a single model, so that the model can better adapt to the needs of the industrial site. Then, we discuss the feasibility of model integration for the NLP.

Let the mechanism models under each running condition be $f_i(\mathbf{X})$ and the ANFIS models be $h_i(\mathbf{X})$, then the *Weighted Integration Model*(WIM) under each running condition and the objective function of the solution can be expressed as follows

$$\begin{cases} \mathrm{Y}_i=\omega_i f_i(\mathbf{X})+\left(1-\omega_i\right)h_i(\mathbf{X}), \ \ i=1,2,3,4 \\ \min J=\min\sum_{i=1}^{m}(\mathrm{Y}_i-\mathrm{Y})^2, \ 0\le\omega_i\le 1. \end{cases} \tag{11}$$

Where i is the label of running condition, Y_i is the output of the WIM, ω_i is the weight value of corresponding running condition, $\mathbf{X}$ is the input variable, Y is the measured value of system output after normalization.

In order to obtain the models of the NLP, the running conditions were simply divided based on actual operations above. However, due to the strong non-linearity of the NLP and the coupling of running conditions, the simple ratio relation cannot accurately describe the condition migration of the system. At the same time, the division of running conditions is not invariable, and the diversity of division methods also makes the accuracy of the model greatly different.

Generally, the system can be divided into N running conditions, and the change of running conditions is also a continuous and smooth process, without sudden jump. The current classification of running conditions is based on the assumption that the model under each running condition is a complete expression of the respective operating conditions. However, under a certain running condition, the obtained model often cannot well or completely express all the information of the running condition, which leads to a distinct boundary between the models, that is, the "*model isolation*" problem caused by "*model mismatch*".

Therefore, the traditional integration method based on the division of running conditions is not reasonable, nor can it solve the "*model isolation*" problem which caused by the coupling of running conditions. In view of this, a method of condition division based on fuzzy membership is proposed. Since the cutting condition is a special and distinct running condition from others, so we can first express the *Fuzzy Weighted Integration Model* (FWIM) under non-cutting running conditions as follows

$$\mathrm{Y}=\omega_1\left[\sum_{i=1}^{3}g_i(p)f_i(\mathbf{X})\right]+\omega_2\left[\sum_{i=1}^{3}g_i(p)h_i(\mathbf{X})\right], \quad 0\le\omega_1,\omega_2\le 1, i=1,2,3 \tag{12}$$

A simple weighted integration is used for the cutting condition, that is:

$$\mathrm{Y}_2=\omega_3 f_4\left(\mathbf{X}\right)+\omega_4 h_4\left(\mathbf{X}\right), 0\le\omega_3,\omega_4\le 1 \tag{13}$$

In (12) and (13), Y and Y_2 are the output of the integrated models, $p=(F_{acid}^{1\#}+F_{acid}^{3\#})\Big/(\sum_{i=1}^{3}m^{i\#}+\varepsilon)$ is the acid-calcine ratio used to divide the system into different running conditions in Fig 3, $f_i\left(\mathbf{X}\right)$ and $h_i\left(\mathbf{X}\right)$ are the model outputs of the mechanism models and ANFIS models under different running conditions, $\omega_1,\omega_2,\omega_3,\omega_4$ are the weight coefficient,

$g_i(p)$ represents the expressible degree of the current state corresponding to the ith model, namely the fuzzy membership degree. As the ratio of acid materials has only a lower bound without an upper one, then the following membership function can be selected for non-cutting conditions:

$$g_1(p;\sigma_1) = \exp\left[-(p/\sigma_1)^2/2\right],$$

$$g_2(p;\sigma_2) = \exp\left[-(p-3/\sigma_2)^2/2\right],$$

$$g_3(p;\sigma_3) = 1\Big/\left\{1+\exp\left[-\sigma_3(p-3)\right]\right\}.$$

Then, (12) can be converted into the following regression form

$$\mathrm{Y} = w^T y \tag{14}$$

In (14)

$$w^T = \begin{bmatrix} \omega_1 g_1(p), \omega_1 g_2(p), \omega_1 g_3(p), \omega_2 g_1(p) \\ , \omega_2 g_2(p), \omega_2 g_3(p) \end{bmatrix} \tag{15}$$

$$y = \left[f_1(\mathbf{X}), f_2(\mathbf{X}), f_3(\mathbf{X}), h_1(\mathbf{X}), h_2(\mathbf{X}), h_3(\mathbf{X})\right]^{\mathrm{T}}. \tag{16}$$

The Mean Square Error (MSE) is selected as the cost function. In order to improve the generalization performance of the integrated model, *L2 regularization* is introduced. The objective function can be expressed as

$$\min \sum_{i=1}^{m} (\mathrm{Y}-\mathrm{Y})^2 + \lambda \|w\|_2^2, m = 75. \tag{17}$$

Specific as follows

$$\begin{cases} \min \sum_{i=1}^{m} \begin{bmatrix} \omega_1 \left[\sum_{i=1}^{3} g_i(p) f_i(\mathbf{X})\right] + \omega_2 \\ \cdot \left[\sum_{i=1}^{3} g_i(p) h_i(\mathbf{X})\right] - \mathrm{Y} \end{bmatrix}^2 + \lambda \sum_{i=1}^{3} g_i(p) \\ p = (F_{acid}^{1\#} + F_{acid}^{3\#}) \Big/ (\sum_{i=1}^{3} m^{i\#} + \varepsilon) \geq 0, \ 0 \leq \omega_i \leq 1. \end{cases} \tag{18}$$

The parameters to be solved are $\{w_1, w_2, \sigma_1, \sigma_2, \sigma_3\}$, let's use GCV (Generalized Cross-Validation) to determine the value of λ. For the regression equation (14), we can get

$$V(\lambda) = (\|(\mathbf{I}-\mathbf{A}(\lambda))\hat{Y}\|^2/m) \Big/ \left[\mathrm{Trace}(\mathbf{I}-\mathbf{A}(\lambda))/m\right]^2 \tag{19}$$

Where $\mathbf{A}(\lambda) = y\left(y^T y + m\lambda\mathbf{I}\right)^{-1} y^T$, then

$$\lambda = \arg\min_{\lambda} V(\lambda) \tag{20}$$

For (17) and (20), we can also use *State Transition Algorithm* to solve it. The final results are shown in Table 1.

5. RESULTS AND ANALYSIS OF THE EXPERIMENT

The experiment results are shown below.

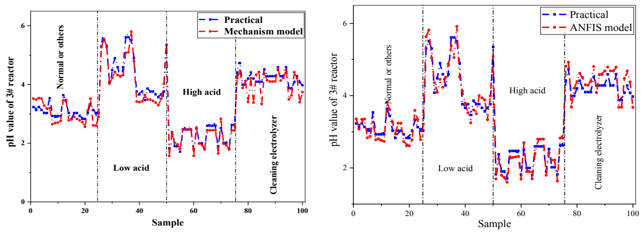

Fig. 4. Output validation of mechanism model and ANFIS model

In order to compare the performance of each model, MAE (Mean Absolute Error), MRE (Mean Relative Error), MaxAE (Max Absolute Error) and RMSE (Root Mean Square Error) were selected as the performance indicators. The specific results are in Table 1. Since the electrolytic cutting operation is carried out separately, the relevant results are not listed in the table.

As can be seen from Table 1, the mechanism model has a large error at some points, but the overall error is smaller. The overall performance of ANFIS model is relatively balanced, which is also related to the fuzzy induction ability of ANFIS. At the same time, the performances comparison of the two models under different running conditions are also different, which further illustrates the necessity of running condition division and model integration.

It can be concluded from the above simulation results that WIM under different running conditions has a greater improvement in overall performance than the single model. At the same time, FWIM based on the fuzzy condition division method also has certain advantages in the overall performance, which fully demonstrates the existence of the "*model mismatch*" phenomenon in the NLP and the effectiveness of the fuzzy condition division method.

6. CONCLUSIONS

In this paper, a method of integrated description based on mechanism model and data-driven model was proposed to predict and estimate the pH value at the outlet of 3# tank in NLP. Firstly, based on the process mechanism, the mechanism model of the system under different running conditions is established. Then according to the characteristics of the NLP, the ANFIS model is established based on the system operation data. Next, the fuzzy integration of the system was carried out for the "*model mismatch*" phenomenon in NLP, and the pH value at the outlet of 3# reactor in NLP was predicted more accurately, the feasibility of this scheme was verified by the data finally. The method described and verified in this paper can provide new ideas for the research on the control and pH value detection in NLP. Meanwhile, the method proposed can also provide effective guidance for the process.

Table 1. MaxRE, MRE, MAE and RMSE of four models

Conditions	Model	MAE	MRE	MaxRE	RMSE
Normal or others	Mechanism model	0.2125	6.88%	16.68%	0.2398
	ANFIS model	0.2195	7.12%	10.96%	0.2276
	WIM	0.1708	5.55%	10.12%	0.1869
	FWIM	0.1557	4.98%	9.35%	0.1836
Low acid	Mechanism model	0.2311	5.54%	18.96%	0.2884
	ANFIS model	0.2712	6.42%	9.75%	0.2865
	WIM	0.2081	4.96%	9.43%	0.2241
	FWIM	0.1732	4.18%	9.14%	0.1923
High acid	Mechanism model	0.1238	5.56%	13.89%	0.1500
	ANFIS model	0.1515	6.81%	9.94%	0.1590
	WIM	0.1070	4.93%	8.88%	0.1172
	FWIM	0.1063	4.85%	8.11%	0.1153
Ensemble	Mechanism model	0.2022	5.91%	18.96%	0.2476
	ANFIS model	0.2318	6.71%	10.96%	0.2495
	WIM	0.1729	5.01%	10.12%	0.1977
	FWIM	0.1604	4.65%	9.35%	0.1887

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