Study on the changing characteristics and model of oxidation-reduction potential in wet desulfurization under experimental conditions

Shuangchen Ma[a,b,*], Fang Xu[a], Yanfei Yu[a], Zixuan Fan[a], ShuaiJun Fan[a]

[a]*Hebei Key Lab of Power Plant Flue Gas Multi-Pollutants Control, Department of Environmental Science and Engineering, North China Electric Power University, Baoding 071003, China, emails: msc1225@163.com (S. Ma), dawnxufang@163.com (F. Xu), 1255374172@qq.com (Y. Yu), 1023740156@qq.com (Z. Fan), 1169146212@qq.com (S.J. Fan)*
[b]*MOE Key Laboratory of Resources and Environmental Systems Optimization, College of Environmental Science and Engineering, North China Electric Power University, Beijing 102206, China*

ABSTRACT

The pH is commonly used to control the acid–base reaction process in existing wet desulfurization systems, but the redox process has not been reasonably controlled. In this study, the oxidation-reduction potential (ORP) is included as a control index of redox reaction in desulfurization system, which is important for scientific adjustment of desulfurization system. Under the experimental conditions, the redox reaction in the process of wet desulfurization was simulated. Through a large amount of experimental data combined with the classical Nernst equation, a semi-empirical formula of ORP suitable for redox reaction of wet desulfurization system was established and its accuracy was evaluated. In the fitting results, the correlation coefficient is 0.98 and the maximum error is less than 8 mV. The experimental research and model give the main parameters that affect ORP changes. The established model can play the role in correcting the on-site ORP measurement value. At the same time, it also provides theoretical support for the introduction of ORP as a wet desulfurization control system.

Keywords: WFGD; ORP; Control indicator; Model; Experimental study

1. Research background

Limestone–gypsum wet desulfurization is the most important means of flue gas desulfurization in China [1]. The reaction that occurs after sulfur dioxide enters the absorption tower can be divided into two parts: acid-base reaction and redox reaction [2,3]. The acid–base reaction is the main reaction affecting the desulfurization rate. The pH value of the slurry in the absorption tower is measured by an online pH monitor, and it is in the proper range by adjusting the amount of limestone slurry. The proper pH range helps to ensure SO_2 absorption and high desulfurization efficiency. After the SO_2 is absorbed by the desulfurization slurry, the redox process of $Ca(HSO_3)_2$ and $CaSO_3$ to

$CaSO_4$ cannot be controlled due to the lack of corresponding indicators. The current redox process intervention mode is to set the amount of oxidizing air blown into the absorption tower to be 1.8–2.5 times the amount of oxygen required to completely oxidize SO_2 under theoretical conditions, which may lead to over-oxidation or under-oxidation due to untimely adjustment [4]. To achieve accurate control of the desulfurization system, the addition of oxidation-reduction potential (ORP) to the limestone–gypsum wet desulfurization system is of great significance for the adjustment of the redox process [5,6].

The essence of ORP is an indicator for the macroscopic oxidation–reduction of all substances in an aqueous

* Corresponding author.

chemical reaction. Most chemical reactions belong to redox reactions. ORP has been successfully applied in many fields as a comprehensive indicator, including drinking water treatment, sewage treatment, metal corrosion protection, heavy metal removal and so on [7–10]. By measuring ORP, you can more accurately understand the redox state of the measured environment. In order to control the product form of the system, adjust the reaction process and factors that play a major role in the redox state of the system. Applying ORP to a wet desulfurization system can solve the following problems:

- Heavy metal form is difficult to control: coal and limestone contain trace amounts of heavy metal elements, which are dissolved in the slurry and repeatedly circulated in the desulfurization slurry to produce a large enrichment effect. This process leads to excessively high concentrations of heavy metal ions and corresponding oxygen acid ions in the wastewater. Desulfurization wastewater cannot be effectively treated, and it is seriously polluted after discharge. Brown et al. [11] pointed out that when the ORP value does not exceed 300 mV, it can play a significant role in controlling the interconversion between selenate and selenite, making it easier for selenite to precipitate as a solid. In addition, Hg^{2+} can be reduced to Hg by HSO_3^-, which is insoluble in water. At low ORP levels (<150 mV), Hg is mainly present in solid form in the slurry. When at high ORP levels (>650 mV), most of the Hg will dissolve, so that Hg may enter the atmosphere with the emission of flue gas, resulting in Hg is re-discharged [12–17].
- The relationship between redox reaction and acid–base reaction does not match: after the SO_2 in the flue gas is dissolved in the absorption tower, it will exist in the form of HSO_3^-, SO_3^{2-}, and the redox reaction process is to convert HSO_3^-, SO_3^{2-} into sulfate precipitation and discharge. When the redox reaction rate is too slow, the consumption rate is lower than the production rate, which will cause HSO_3^-, SO_3^{2-} accumulation. When the sulfite content is high, it will become a crystalline pollutant and cause system fouling. In addition, calcium sulfite with a small particle size deteriorates the quality of gypsum. Because gypsum dehydration is more difficult. The high concentration of sulfite covers the surface of the limestone particles, which will prevent it from dissolving and cause limestone blockage. This results in the reduction of limestone utilization and desulfurization efficiency, which seriously endangers the normal operation of the desulfurization system [18–20].
- Energy waste and potential safety hazards: to give priority to the complete oxidation of SO_2, an oxidation air volume is designed according to the amount of oxygen required for complete oxidation of SO_2 under theoretical conditions. The design value is 1.8–2.5 times the theoretical calculation result obtained through experience. Excessive oxygen increases the overall ORP of the slurry, which increases the energy of the oxidation fan and the operating cost of the power plant's desulfurization system. This model is not conducive to the energy saving and emission reduction of power plants. At the same time, the excess air in the desulfurized slurry diffuses

from the bottom of the oxidation tower to the surface of the slurry in the form of bubbles, and a large amount of foam is formed on the desulfurized slurry. The change of the foam thickness on the surface of the absorption tower slurry causes the inaccurate level measurement of the desulfurization tower slurry and the overflow of the absorption tower slurry. When the phenomenon occurs, it will not only lead to deterioration of the slurry quality, but also lead to safety accidents in serious cases. Adding ORP as a control indicator to the existing wet desulfurization system is of great significance for optimizing the existing desulfurization system. However, the theoretical guidance of ORP in the application process can be further improved. The measurement principle of ORP is based on the Nernst equation, but the influence of relevant factors on the redox reaction in the wet desulfurization system cannot be accurately obtained only based on the Nernst equation. Therefore, it is of great theoretical and practical value to establish a practical ORP model with desulfurization system as the research object.

2. Experimental and semi-empirical formula

2.1. Experimental device and method

In practical engineering applications, the flue gas enters the absorption tower for redox reaction after being treated by an electrostatic precipitator. During the simulation experiment in the laboratory, the redox reaction in the absorption tower was mainly studied. Calcium sulfite solids are added to the reaction vessel to replace the process of introducing flue gas into the limestone slurry. The experimental device is shown in Fig. 1.

During the experiment, the temperature of the reaction system was adjusted by controlling the temperature of the water bath heater; the initial concentration of the experiment was changed by changing the solid content of calcium sulfite; the amount of oxidizing air was controlled by a gas flow meter; and the stirring speed was adjusted by controlling the stirring rate.

The experiment was carried out to monitor the dissolved oxygen (DO), pH, and ORP values during the reaction by DO electrode (Shanghai Sanxin SX751), pH electrode (Luheng Bio-DG160), and ORP electrode (E+H CPM253). The experiment process is recorded every 20 min. The concentration of sulfite ions during the reaction is measured by titration. In the acidic solution, the sulfite is subjected to an oxidation–reduction reaction with iodine, and the excess iodine is titrated with a sodium thiosulfate standard solution. Its reaction formulas are:

$$SO_3^{2-} + I_2 + H_2O \rightarrow SO_4^{2-} + 2HI \tag{1}$$

$$2S_2O_3^{2-} + I_2 \rightarrow S_4O_6^{2-} + 2I^- \tag{2}$$

2.2. Semi-empirical formula

2.2.1. Nernst equation

The classical Nernst equation quantitatively describes the diffusion potential formed between the two systems
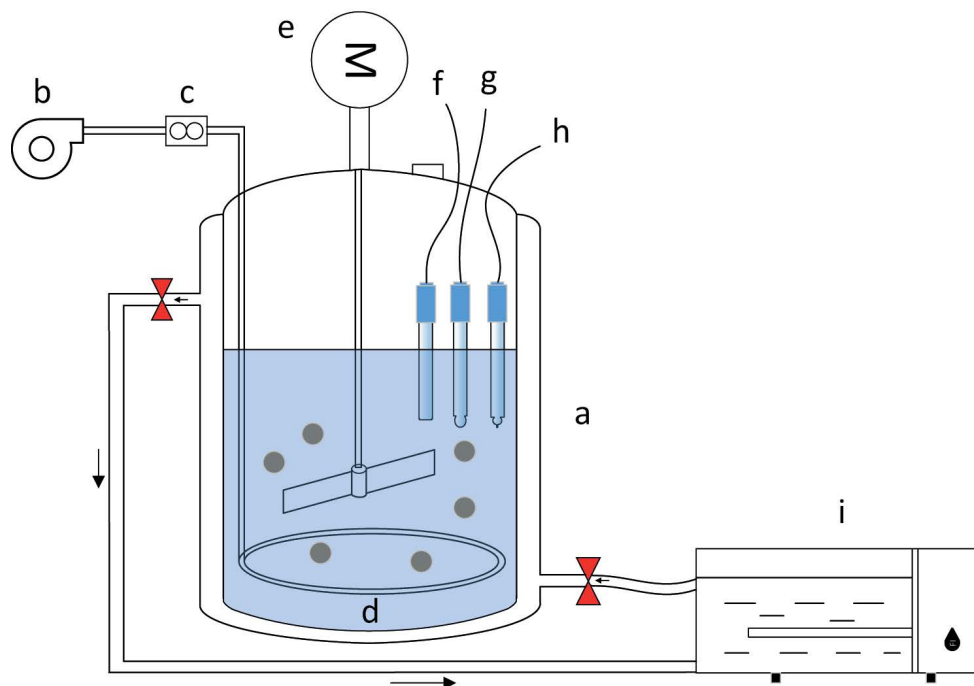
Fig. 1. Experimental device: a, glass reactor; b, oxidation fan; c, flow meter; d, oxidation duct; e, stirrer; f, dissolved oxygen electrode; g, pH electrode; h, ORP electrode; and i, water bath heater.

of oxidation and reduction in the system. The equation expression is:

$$\text{ORP} = E^{\theta} + K \times \ln \frac{[\text{OX}]}{[\text{RED}]} \tag{3}$$

In the formula, E^{θ} is the standard ORP; [OX] is the concentration of oxidizing substances; [RED] is the concentration of reducing substances; K is the constant, i.e., RT/nF.

In the wet desulfurization system, the redox process occurring in the slurry is not a specific reaction of an oxidizing substance and a reducing substance in the same phase. In this process, the reaction of +4 valence sulfur to oxidize to +6 valence sulfur take place under the action of oxygen. At the same time, there will be valence changes in other variable elements.

To obtain a formula that is more consistent with the change in ORP during the wet desulfurization redox reaction process, it is used to calculate the SO_3^{2-} concentration in the slurry. Therefore, it is necessary to perform fitting based on the classic Nernst equation to ensure the normal operation of the desulfurization system.

2.2.2. Influencing factors of ORP

To establish a semi-empirical formula that is more suitable for wet desulfurization systems, the influencing factors of ORP in the wet desulfurization system need to be determined. Through the redox reaction, it can be determined that the DO and SO_3^{2-} concentration have a high correlation with the ORP value. They belong to the reaction system in the main oxidizing substances and reducing substances. However, pH is a variable during the reaction, so

it is necessary to incorporate the effect of pH on the ORP value into a semi-empirical formula to increase its accuracy. To this end, a representative experiment was carried out. The correlation between each factor and ORP was observed under the conditions of calcium sulfite concentration of 2 g/L, agitator of 100 r/min, and oxidizing air volume of 100 L/h. The stabilization phase is shown in Table 1.

During the experiment, the parameters were measured at intervals of 20 min. The curves of various parameters with time in the steady phase of the experiment are shown in Fig. 2.

According to the variation law of various parameters in the experiment of influencing factors. It can be concluded that the pH, DO, and the concentration of SO_3^{2-} have a high correlation with the ORP value, so the concentration of SO_3^{2-}, DO content, and pH were included in the model.

2.2.3. ORP semi-empirical formula

In order to establish an ORP semi-empirical formula, an oxidation process experiment was performed. First,

Table 1
Influence factor experiments

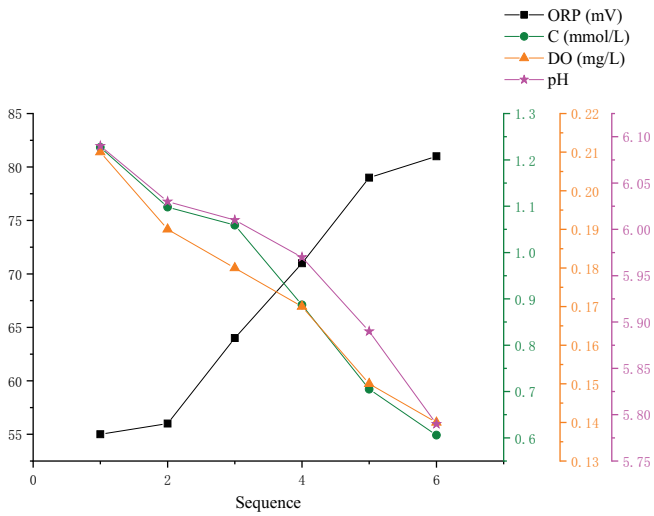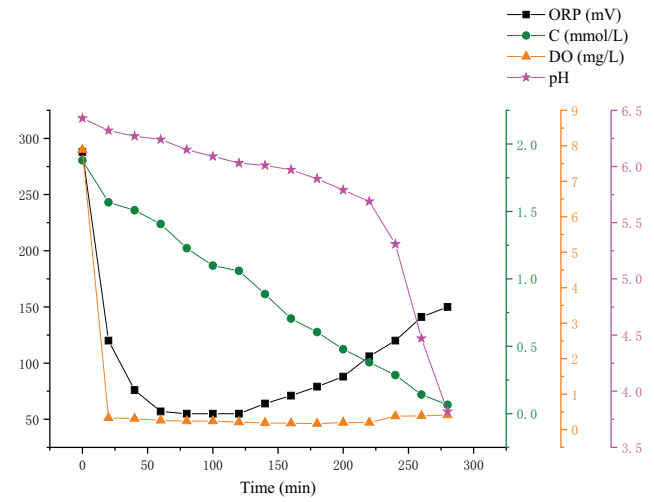| Sequence	pH	ORP (mV)	DO (mg/L)	C (mmol/L)
1	6.09	55	0.21	1.227
2	6.03	56	0.19	1.098
3	6.01	64	0.18	1.059
4	5.98	71	0.17	0.887
5	5.89	79	0.15	0.705
6	5.79	81	0.14	0.606

Fig. 2. Parameter curve.

Fig. 3. Oxidation curve using calcium sulfite of 2 g/L.

the experiment measures calcium sulfite data by titrating every 20 min. During the experiment, the ORP value, pH value and DO value were continuously recorded until the sulfite oxidation was complete. Through the experiment of the concentration variable and the air volume variable, the characteristics of the oxidation process are basically obtained. The change trend of each parameter under the experimental conditions of calcium sulfite 2 g/L, oxidizing air volume 100 L/h, stirring rate 100 rpm and calcium sulfite 1 g/L, oxidizing air volume 100 L/h, stirring rate 100 rpm, respectively, is shown in Figs. 3 and 4.

By analyzing the data obtained during the experimental process, the following rules can be drawn:

- The influence of SO_3^{2-} concentration: the concentration of SO_3^{2-} continued to decrease throughout the progress of the reaction. When the DO content and pH change slowly, it can be found that as the redox reaction progresses slowly, the concentration of SO_3^{2-} continues to decrease while the ORP value was also gradually increasing, at which time sulfite was the main factor leading to ORP change.

- The influence of DO: it can be clearly found that in the whole process, the change of DO is obvious. The whole process is to keep a low value for a period after a rapid decline, and finally rise to a medium value. At the beginning of the reaction, due to the addition of calcium sulfite, a large amount of DO in the water was consumed. This process can be called rapid oxidation. Sulfite concentration is the main rate controlling factor. Since the dissolution rate of the air blown by the fan is lower than the rate of DO consumed by the redox reaction, the DO remains at a low level. After the sulfite concentration drops to a certain concentration, the amount of DO converted by oxygen exceeds the consumption of DO, so the concentration of DO gradually increases. In the final process, when the pH and sulfite concentration are basically unchanged, ORP directly reflects the increase in DO.

- The influence of pH: both ORP and pH were detected by online instrument. Observing the ORP value and pH value, ORP has a very sensitive reaction to pH. Therefore,
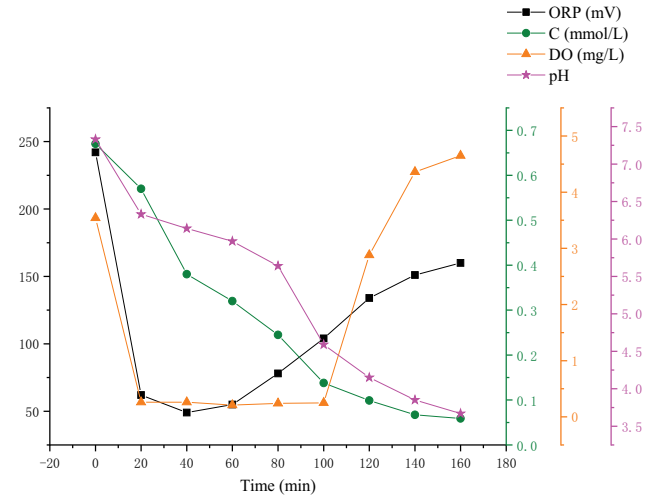

Fig. 4. Oxidation curve using calcium sulfite of 1 g/L.

when judging the oxidation status of sulfite, the effect of pH should be considered first.

In summary, ORP can directly reflect the extent of oxidation of flue gas desulfurization slurry. If the detected value of the reaction oxidation state, such as pH value, DO value and sulfite concentration, can be correlated with the ORP value, the oxidation state of the slurry system can be reflected very well. The most intuitive method is to use the ORP value to directly calculate the sulfite concentration to evaluate the oxidation state of the absorption tower. The titration step can be omitted to determine the sulfite content accurately and quickly. The theoretical ORP value can also be calculated from the measured sulfite concentration and compared to the measured value for calibration.

Analysis of the entire oxidation process can be roughly divided into four phases:

- The first phase was a rapid consumption of the original DO at the beginning of the reaction. The DO was

much higher than the dissolved sulfite concentration. During this short period, the main controlling factor is the sulfite concentration.

- The second period is the slow oxidation period after the original DO is rapidly consumed. Since the reaction rate controlling factor becomes the dissolution rate of oxygen, the DO content during this period is stable at a low level.
- The third period is when the sulfite decreases to a lower concentration, the oxygen content required for the reaction decreases, so the DO content begins to increase. The length of this period depends on the air flow, the greater the flow, the shorter the time.
- The fourth period is after the sulfite is completely consumed; the concentration of DO gradually increases to saturated DO.

After analysis, to accurately establish a semi-empirical equation, a large number of experiments under different conditions have been carried out. Therefore, the data obtained is more representative and the model is easier to build. After categorizing the results of the experiment, 105 experimental result points are selected in the first part to fit the semi-empirical formula. The second part selects another 100 experimental results points and integrates the first part data to evaluate the established model.

Many experiments have been carried out on different initial calcium sulfite concentrations and blast volumes. After extensive data analysis and mapping analysis, it can be found that there is a certain relationship between ORP value and pH value, DO, and sulfite concentration. When the other two quantities remain constant, sulfite concentration is negatively correlated with ORP value, oxygen content is positively correlated with ORP value, and pH value is negatively correlated with ORP value. Combining the classic Nernst equation and fitting through a large amount of experimental data, the ORP semi-empirical Eq. (4) under experimental conditions is obtained:

$$ORP = b + k \times \log\left(\frac{DO \times 10^7}{C_{(SO_3^{2-})} \times 10^{pH}}\right) \qquad (4)$$

3. Results and discussion

3.1. Semi-empirical formula parameter determination and evaluation

Based on the semi-empirical formula model, the data recorded in the experimental process is substituted into Eq. (4), the parameters are determined and evaluated.

The concentrations of 0.5, 1.0, 1.5, and 2 g/L calcium sulfite were, respectively, passed to different air volumes. After recording the experimental data, 105 items were selected to participate in the semi-empirical formula fitting, and the fitted curve is shown in Fig. 5.

In the fitted curve, the abscissa is $\log\left(\dfrac{DO \times 10^7}{C_{(SO_3^{2-})} \times 10^{pH}}\right)$ and the ordinate is the measured ORP value. The fitted curve
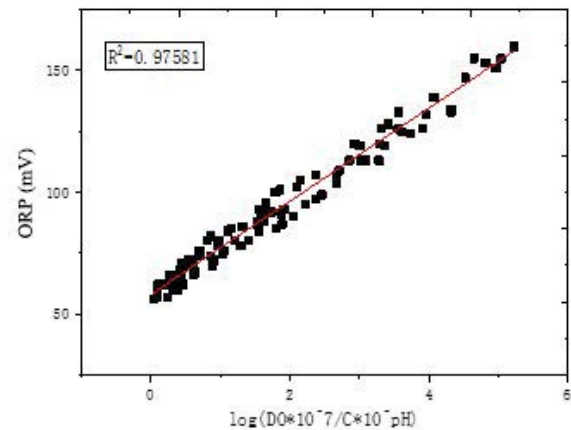

Fig. 5. Fitting curve.

shows a higher accuracy. It is found that the parameter b in the semi-empirical formula is 58.13, and k is 19.13. The semi-empirical Eq. (5) after the parameters determined is as follows:

$$ORP = 58.13 + 19.13 \times \log\left(\frac{DO \times 10^7}{C_{(SO_3^{2-})} \times 10^{pH}}\right) \qquad (5)$$

The correlation coefficient of the semi-empirical formula was $R^2 = 0.9758$. The actual measured values of 205 data obtained under different experimental conditions were compared with the results calculated by semi-empirical formulas. The calibration curve is shown in Fig. 6.

In the measurement and fitting comparison chart, the abscissa is the data number, and the ordinate is the ORP value. The points in the graph are the actual measured values and the semi-empirical formula calculated during the experiment. The accuracy is not as high as the fitting after the number of samples is expanded, and the actual measurement point distribution has a larger range on both sides of the fitting curve, but the overall change trend is consistent with the actual measurement. To further evaluate the established semi-empirical formula, the regression analysis is used to evaluate the prediction accuracy of the equation, including the determination coefficient (R^2), the root mean square error (RMSE), and the normal root mean square error (NRMSE), which are defined as follows:

$$R^2 = 1 - \frac{\sum_{i=1}^{n}\left(y_i - \hat{y}_1\right)^2}{\sum_{i=1}^{n}\left(y_i - \overline{y}_1\right)^2} \qquad (6)$$

$$RMSE = \sqrt{\frac{1}{n}\sum_{i=1}^{n}\left(y_i - \hat{y}_1\right)^2} \qquad (7)$$

$$NRMSE = \frac{RMSE}{y_{max} - y_{min}} \times 100\% \qquad (8)$$

Table 2
Regression analysis evaluation results

	R^2	RMSE (mV)	NRMSE (%)
Fitting group (105)	0.98	4.2	4.1
Integral group (205)	0.88	9.0	7.6

where n is the sample number, y_i and $\hat{y}_1$ are the actual measured ORP values and the formula calculates the ORP value, $\hat{y}_1$ is the average value of y_i, y_{max} and y_{min} are the maximum and minimum values of y_i, respectively. The evaluation results are shown in Table 2.

The fitting group is the comparison between the experimental data points participating in the fitting of semi-empirical formulas and the model calculation results. The correlation coefficient of the model is 0.98, and the error is within 8 mV, indicating that the experimental data points selected to participate in the model establishment are highly correlated with the final model. The integral group is the comparison between all experimental data points and model calculation results. The coefficient of determination was reduced to 0.88 compared to the fitted group, and the error was still within 17 mV. The RMSE of the fitting groups and integral groups was 4.2 and 9.0 mV, respectively.

3.2. Discussion

In the semi-empirical formula, when the pH is greater than 7, $\log\left(\dfrac{DO \times 10^7}{C_{(SO_3^{2-})} \times 10^{pH}}\right)$ is less than 0, and the sulfite concentration cannot be measured. The actual operating environment of the wet desulfurization system is an acidic environment, so this setting conforms to the actual working conditions.

Under the condition of obtaining the system pH value, DO value, and sulfite concentration, the ORP value can be calculated by a semi-empirical formula to realize the accuracy evaluation and correction of the ORP measurement value. This can improve the reliability after ORP is added to the system.

The semi-empirical formula obtained by the laboratory simplifies the existing influencing factors. There will be more interference factors in the actual operation process; the model needs to be further improved if it is actually applied to the wet desulfurization system.

4. Conclusion

ORP can indicate the oxidation–reduction status of a specific environment. Adding it to the wet desulfurization system can optimize the system. It helps to control the form of harmful heavy metals in the absorption tower, reduce the difficulty of subsequent desulfurization wastewater treatment, establish the matching relationship between oxidation reaction and acid–base reaction, improve the quality of desulfurization by-products, reduce the energy consumption of the desulfurization system and ensure the safe operation of the desulfurization system. In this paper, the oxidation
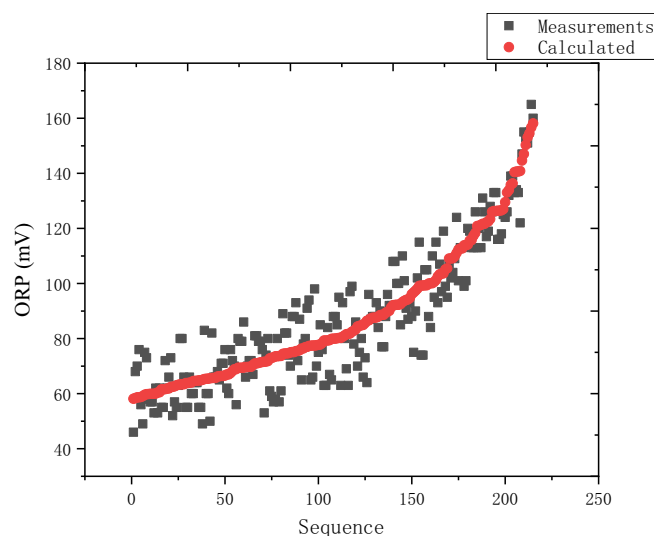

Fig. 6. Measurement and fitting comparison chart.

process in wet desulfurization was simulated under laboratory conditions. The experimental data and Nernst equation were combined to establish the ORP model. Model parameters include sulfite concentration ($C_{(SO_3^{2-})}$), DO content, and pH. The correlation coefficient between the model and the fitted experimental data points is 0.98. The correlation coefficient between the model and all experimental data points is 0.88. The error between the model and all experimental data point does not exceed 17 mV. The model established in the laboratory can play a role in correcting ORP measurements, which provide basic theoretical support for the introduction of ORP as a control index of wet desulfurization system.

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