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STUDY ON MEASUREMENT MODEL OF LIGNIN CONTENT IN PULP AFTER ALKALINE EXTRACTION

Zhichao MA[1, 2], Shaoxu CHEN[1, 2], Yongjun YIN[1, 2*], Yanying ZHOU[2], Xiaodan LU[2], Tingting LIN[2]

[1]*Guangxi Key Laboratory of Clean Pulp & Papermaking and Pollution Control, Guangxi, P. R. China*
[2]*School of Light Industry and Food Engineering, Guangxi University, Guangxi, P. R. China*

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Highlights

- Analyzing the correlation between lignin content with operating conditions of alkaline extraction.
- Modeling of lignin content based on alkaline extraction conditions makes measurement more convenient and faster.
- The soft sensor model is helpful to control bleaching agent dosage and reduce the generation of AOX.

Abstract. Because kappa number cannot accurately represent the lignin content in the pulp after alkaline extraction, lead to the excessive dosage of bleaching chemicals added and the pollutant content increases. In order to accurately determine the dosage of bleaching agent, reduce pollutant emissions, a prediction model of lignin content of pulp was established by analyzing the correlation between lignin content and alkaline extraction conditions in this paper. The results show that the established soft sensor model can accurately measure lignin content, it is helpful to determine the amount of bleaching agent more accurately, reduce pollutant generation after pulp bleaching.

Keywords: adsorbable organic halogen (AOX), prediction model, lignin content, alkaline extraction.

Introduction

With the improvement of social demands on the environment, and the pollution of pulp bleaching wastewater becomes more and more serious which aroused wide attention of the whole society. Especially, adsorbable organic halogen (AOX) produced during pulp bleaching is lipophilic, can be accumulated in the organism, difficult to degrade, and has a strong carcinogenic and harm to biological endocrine system (Sow et al., 2014). Therefore, many countries impose strict limits on its emissions (Government of Canada, 1999; US EPA, 2018; Standardization Administration of China, 2008). How to reduce AOX content in wastewater becomes an important research direction in pulp and paper industry. As environmental requirements improve, ECF (Elemental Chlorine Free) bleaching technologies has developed rapidly in recent years in order to reduce AOX generation, researchers have done a lot of research on the mechanism of AOX generated by ECF bleaching and how to reduce AOX emission. Lehtimaa et al. studied the change of AOX of hardwood pulp under different pre-bleaching conditions and the effect of operation parameters on the formation of AOX by chlorine dioxide pre-bleaching of birch sulfate pulp. Research results indicate, AOX forms rapidly at the beginning of bleaching, the reaction temperature had little effect on the formation of AOX, and the increase of chlorine dioxide expedites the production of AOX in the D_0 stage (Lehtimaa et al., 2010a, 2010b). Shi et al. studied the effect of lignin structure on the generation of adsorbable organic halogens during chlorine dioxide bleaching and found the combination of phenolic and non-phenolic structures can promote the formation of AOX (Shi et al., 2019). The kinetic model of AOX degradation in bleaching sulfate pulp mill wastewater shows that half of the AOX degradation reactions are caused by light (Frisk & Bilaletdin, 1994). Sharma et al. found the concentration of AOX in bleaching wastewater decreased by 34% by xylanase treatment (Sharma et al., 2014). Also, the removal of hexuronic acid

*Corresponding author. E-mail: *yinyj@gxu.edu.cn*

can reduce AOX generation in chlorine dioxide bleaching (Zhang et al., 2018a). Isoaho et al. studied the degradation of AOX in the wastewater of cork sulfate pulp mill and found AOX removal rate was about 70% under certain Fenton reaction conditions (Isoaho et al., 2012). Some research about modeling, optimization of pulp bleaching had been done to reduce pollutant generation and bleaching costs in our previous work (Yin et al., 2018; Ma et al., 2020). At present, most of the studied focus on how to reduce AOX emissions theoretically in view of generation mechanism (Zhu et al., 2016, 2017; Dai et al., 2016), and there are few studies on controlling AOX emissions by mathematical model. People payed more attention to control the bleaching reaction for reducing AOX production (Kaur et al., 2018; Zhang et al., 2018b), without considering reducing the components that may produce AOX from the source. This paper intends to establish a measurement model of lignin content based on the relationship between alkaline extraction conditions and lignin residue contents in pulp. AOX emissions can be reduced because the amount of bleaching agent added are controlled based on accurate measurement of lignin content model.

Generally, researchers believe that the generation of AOX mainly comes from bleaching process in the pulp and paper industry, and the amount emitted is mainly affected by the residual lignin content in the pulp. AOX is mainly produced by the chemical reaction between lignin in pulp and bleaching agent. Usually, Kappa number was tested to estimate lignin residues in the pulp after alkaline extraction. Chlorine-containing bleaching agent widely used in pulp bleaching because of the low cost, the high bleaching efficiency and the strong applicability of equipment. A small amount of AOX will be generated when chlorine bleaching agent was used in the subsequent bleaching stage. Kappa number is related to carbohydrates (Li & Gellerstedt, 1997), which does not represent the lignin content of the pulp accurately. As a result, the amount of bleach agent used in the subsequent bleaching phase is higher than the actual demand, and the amount of contaminants increases. It is helpful to optimize the bleaching process to study the environment, economic cost and quality index of bleaching pulp. Lignin contents can be predicted timely after adjustment because the optimization of bleaching process implemented if measurement model of lignin content was established. The established measurement model can measure the lignin content quickly and help determine the appropriate amount of subsequent bleach accurately. Reasonable dosage of bleaching agent will contribute to reduce the generation of AOX.

1. Experiments and methods

1.1. Experiment

The unbleached pulp used in the experiment was from Guangxi hongyuan pulp paper Co., Ltd. The whiteness after D_0 bleaching was 43.64(% ISO), intrinsic viscosity was 890 (mg/L), the relative content of lignin was 2.26(%). $D_0E_PPD_1$ bleaching technology was selected in this study. In the alkaline extraction stage, the dosage of sodium hydroxide changes in range of 0.7 to-5.7%, the reaction time of alkaline extraction variate within 40 to 90 mins, the reaction temperature of was between 45–95 °C, and the dosage of hydrogen peroxide set to 0.7%. Single factor experiments with 3 factors and 6 levels were carried out in alkaline extraction of ECF bleaching sequence. The experimental results are shown in Table 1.

Table1. Results of single factor experiment

Serial	Sodium Hydroxide Dosage (%)	Temperature (°)	Time (min)	Lignin Relative Content (%)		
1	.55	60.00	55.00	0.78	0.75	0.76
2	1.05	60.00	55.00	0.62	0.65	0.60
3	1.55	60.00	55.00	0.50	0.53	0.49
4	2.05	60.00	55.00	0.48	0.47	0.45
5	2.55	60.00	55.00	0.36	0.34	0.37
6	3.05	60.00	55.00	0.22	0.24	0.25
7	1.55	60.00	35.00	0.68	0.71	0.73
8	1.55	60.00	45.00	0.60	0.60	0.59
9	1.55	60.00	55.00	0.50	0.52	0.55
10	1.55	60.00	65.00	0.42	0.41	0.44
11	1.55	60.00	75.00	0.34	0.33	0.30
12	1.55	60.00	85.00	0.28	0.27	0.26
13	1.55	40.00	55.00	0.66	0.65	0.64
14	1.55	50.00	55.00	0.60	0.58	0.61
15	1.55	60.00	55.00	0.55	0.54	0.56
16	1.55	70.00	55.00	0.50	0.52	0.53
17	1.55	80.00	55.00	0.43	0.44	0.48
18	1.55	90.00	55.00	0.34	0.37	0.35

1.2. Method

The content of acid-insoluble (Klason) lignin was measured as per the TAPPI T222 om-02 (2002) standard. SPSS 20 Statistics software (IBM Corp., New York, NY, USA) was used to analyze the data and simulate the correlations between Alkaline extraction conditions and lignin content of pulp. The goodness of fit (R^2), F-value, and P-value were used to analysis the model correlations. 15 groups of data were randomly selected for model fitting, and the remaining 3 groups of data were used for model verification.

2. Results and discussion

2.1. Correlation analysis of sodium hydroxide dosage and lignin content

As shown in Figure 1, the relative content of lignin in pulp decreased with the increase of sodium hydroxide dosage. Sodium hydroxide dissolves the lignin that attaches to the surface of the pulp fiber (Pang et al., 2011). Since lignin in pulp was extracted by the strengthened alkaline extraction method of hydrogen peroxide. H_2O_2 can ionize hydroperoxide ion which has bleaching effect under alkaline conditions, react with lignin chromophores (such as carbonyl, conjugated carbonyl group, quinone-type or methylene quinone groups), and achieve the purpose of bleaching (Xu et al., 2014). Some studies found that the degradation of 2, 5-dihydroxyl -[1,4] -benzoquinone (DHBQ) by hydrogen peroxide on pulp fiber is affected by alkali metal (Hosoya et al., 2015). Alkaline extraction reaction increases as the increase of sodium hydroxide, also the alkalinity of pulp delignification increased, which promoted the reaction between hydrogen peroxide and lignin, the lignin dissolved by sodium hydroxide increased. Therefore, as the increase of sodium hydroxide dosage, the lignin content of pulp decreased. Some most suitable models were selected to regress sodium hydroxide dosage versus lignin content (Figure 2). It can be seen that a cubic curve can fit the relationship between the relative content of lignin and content of sodium hydroxide better than quadratic curve. The fitting relation evaluation is shown in Table 2.

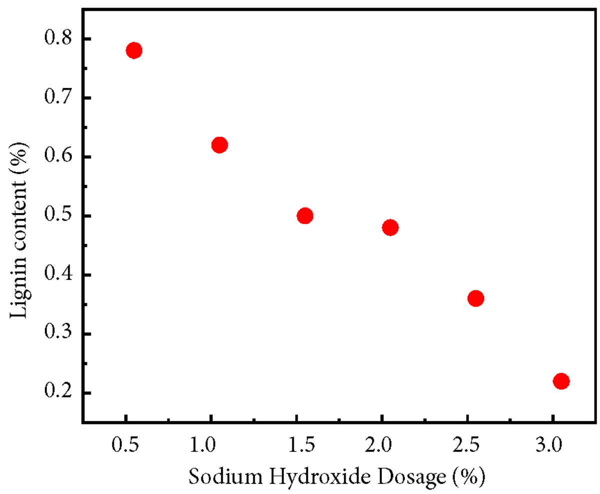

Figure 1. The correlation between sodium hydroxide dosage and lignin content

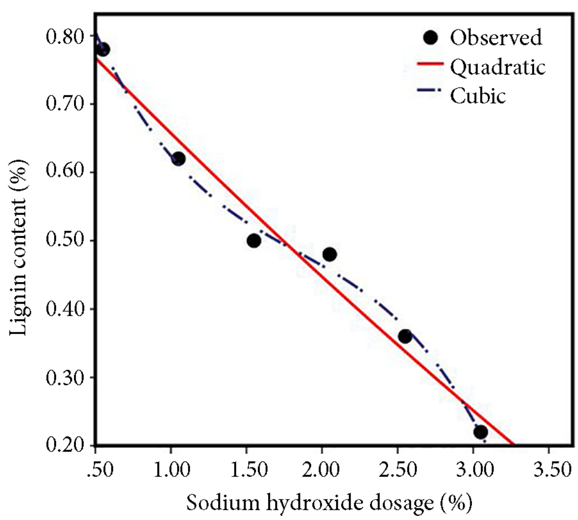

Figure 2. Model equation correlations between sodium hydroxide dosage and lignin content

Table 2. Fitting results of sodium hydroxide dosage and lignin content

Equation	R^2	F-value	Significant P-value
Cubic	0.994	107.995	0.005
Quadratic	0.970	48.999	0.009

As shown in Table 2, R^2 = 0.994 and F = 107.995 for the cubic function, which was the largest in the fitting relationship. Therefore, the best model relationship between the removal amount of lignin in pulp and content of sodium hydroxide was a cubic equation which can be expressed as Eq. (1).

$$y = y_0 - y_i = a_1 x_1 + b_1 x_1^2 + c_1 x_1^3 + d_1 , \quad (1)$$

where, y_0 and y_i represent the initial and residual lignin content of pulp respectively, a_1, b_1, c_1 are the coefficients of the linear and quadratic, cubic terms, respectively; the variables x_1 and y are content of sodium hydroxide and the removed lignin, respectively; d_1 is a constant.

2.2. Correlation analysis between alkaline extraction time and pulp lignin content

As shown in Figure 3, the lignin relative content decreased as the increased of reaction time. With the increased of reaction time, the lignin dissolved by sodium hydroxide increased, and the lignin content of pulp decreased. In alkaline medium, fiber can be inflated to facilitate diffusion of bleaching chemicals and soluble substances, the higher the degree of fiber swelling, the more lignin dissolved as the extension of alkali extraction time (Bajpai, 2012); the degradation of lignin by hydrogen peroxide is more complete (Sun et al., 2018; Olsson, 2002). 5, 8-dihydroxyl -[1,4] -naphthoquinone

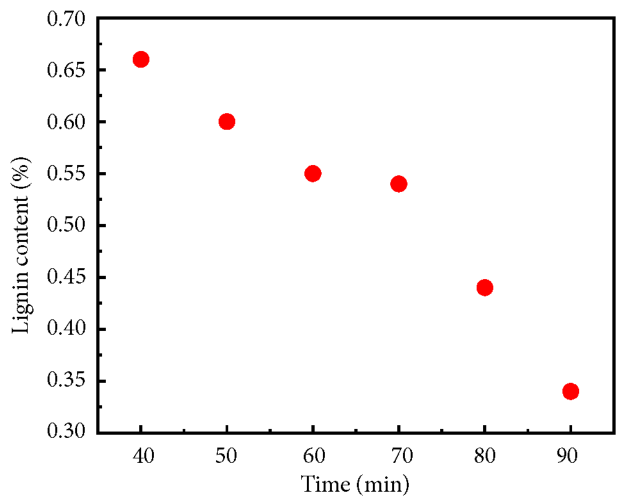

Figure 3. Correlation between reaction time and lignin content

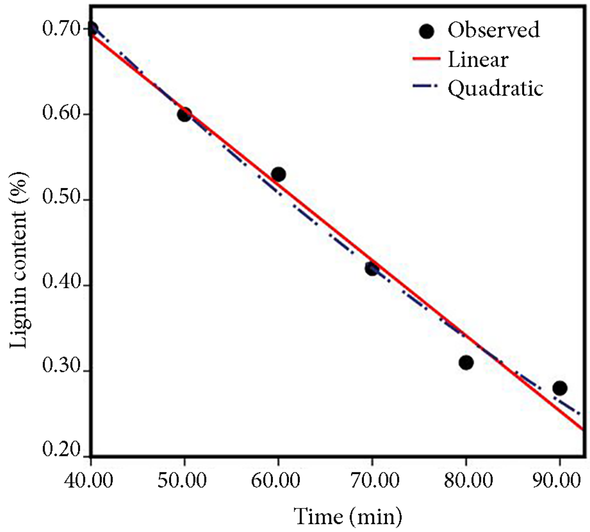

Figure 4. Model equation correlations between reaction time and lignin content

(DHNQ) in pulp fiber was degradated by hydrogen peroxide more completely as the extension of reaction time (Zwirchmayr et al., 2017). Therefore, the relative content of lignin in pulp decreased with the increased of reaction time. The most suitable model equations were selected to fit the correlation between alkaline extraction time and lignin content according to the curve presented in Figure 4. The results of the regression analyses are shown in Table 3.

Table 3. Fitting results of reaction time and relative lignin content

Equation	R^2	F-value	Significant P-value
Quadratic	0.988	128.775	0.001
Linear	0.985	269.245	0.000

As shown in Table 3, R^2 = 0.988, which are the largest values in all fitting relationships in Table 3. Therefore, the best model relationship between reaction time and lignin content shows a quadratic curve that is expressed as Eq. (2):

$$y = a_2 x_2 + b_2 x_2^2 + d_2, \qquad (2)$$

where a_2, b_2 are the coefficients of the linear and quadratic terms, respectively. The variables x_2 is reaction time; d_2 is a constant.

2.3. Correlation between alkaline extraction temperature and pulp lignin content

As shown in Figure 5, as the increase of the reaction temperature, the efficiency of sodium hydroxide in dissolving lignin increased, then the lignin content decreased. The raise of temperature made the lignin solubility increased (Eriksson & Grén, 1997). We strengthened alkaline extraction by adding hydrogen peroxide to dissolve lignin. Since H_2O_2 may decompose and generate HOO- ion under alkaline condition which is an active group, and the number of active groups increases with increasing of reaction temperature, chromophoric groups in lignin reduced or part of lignin dissolved during the bleaching process (Li et al., 2012). Some studies found that the reaction rate of hydrogen peroxide delignification increases with the increase of reaction temperature (Cui et al., 2000; Moldenius & Sjögren, 1982). So, as the temperature increases, the amount of active groups produced by hydrogen peroxide increases, the lignin content attached to the pulp surface decreases. Some models were selected to regress alkaline extraction temperature versus lignin content (Figure 6). The results from the model regression analyses are shown in Table 4.

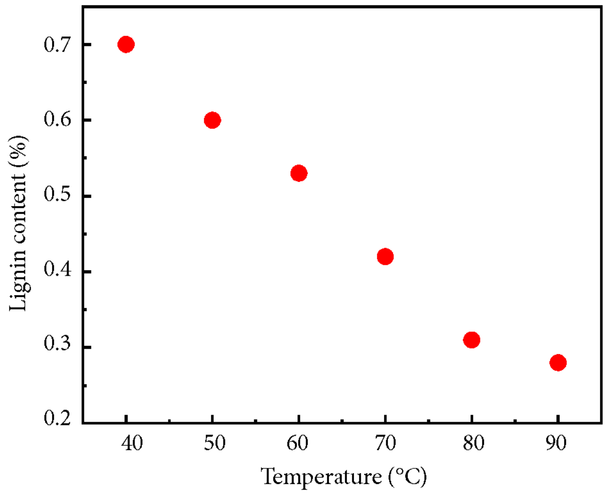

Figure 5. Correlation between reaction temperature and lignin content

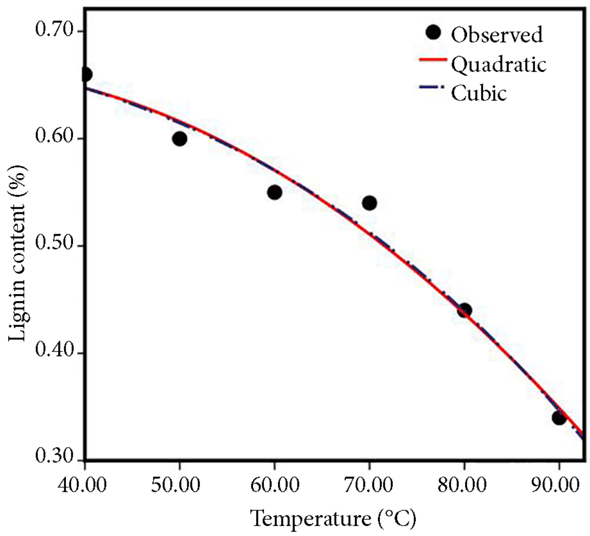

Figure 6. Model equation correlations between reaction temperature and lignin content

Table 4. Fitting results of reaction temperature and lignin content

Equation	R^2	F-value	Significant P-value
Cubic	0.976	60.906	0.004
Quadratic	0.973	54.278	0.004

As shown in Table 4, R^2 = 0.976 and F = 60.906 for the cubic function, which was the largest in the fitting relationship. Therefore, the best fitting relationship between alkaline extraction temperature and pulp lignin content was a cubic equation that is expressed as Eq. (3):

$$y = a_3x_3 + b_3x_3^2 + c_3x_3^3 + d_3\,, \tag{3}$$

where, a_3, b_3, c_3 are the coefficients of the linear and quadratic, cubic terms, respectively; the variables x_3 is reaction temperature; d_3 is a constant;

The relationship between alkaline extraction conditions and lignin content of pulp can be obtained from the above three equations, as shown in the following equation.

$$y = a_1x_1 + a_2x_2 + a_3x_3 + b_1x_1^2 + b_2x_2^2 + b_3x_3^2 + c_1x_1^3 + c_2x_3^3 + d. \tag{4}$$

Based on Eq. (4), 15 groups of experimental data were randomly selected for model regression. The results from regression yielded the values for the equation parameters (a_1 to a_3, b_1 to b_3, c_1 to c_2): as shown in Table 5.

Table 5. Values for the equation parameters

Items	Values
a_1	0.636808507
a_2	0.00855296
a_3	0.106725986
b_1	−0.267131657
b_2	$-6.73831*10^{-7}$
b_3	−0.00165168
c_1	0.048628145
c_2	$8.61188*10^{-6}$
d	−1.624316714

3. Model verification

As shown in Figure 7, that goodness of fit R^2 = 0.9942, indicating a strong correlation between the calculated value of lignin content and the experimental value. Three experiments were selected randomly, were used to verify the accuracy of the model. As shown in Table 6 that the maximum relative error between the calculated value of lignin content and the experimental value is less than 6.25% which indicates that the lignin content soft sensor model based on alkaline extraction condition of pulp is acceptable and feasible.

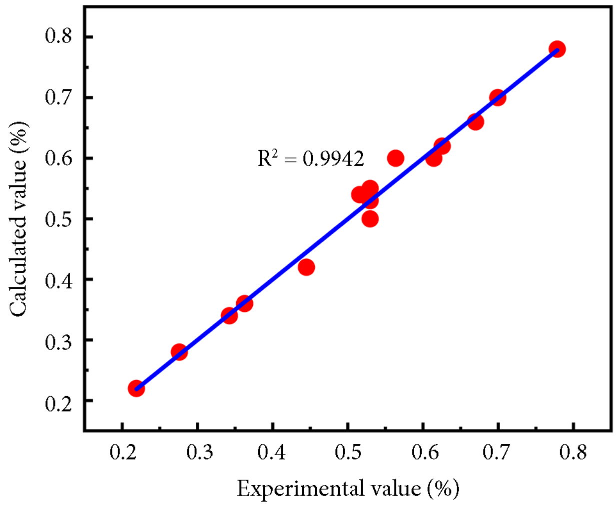

Figure 7. Correlation between the experimental value of lignin content and the calculated value

Table 6. Relative errors between experimental and calculated values of lignin content

Serial number	Experimental value (%)	Calculated value (%)	Relative error (%)
4	0.48	0.45	6.25
11	0.34	0.36	5.88
17	0.44	0.45	2.27

4. Possible application and future perspectives

All the three factors selected (sodium hydroxide dosage, alkali extraction temperature and time) can be collected online in this paper. From the results above, it can be seen that the correlation between the lignin content and the amount of sodium hydroxide, the alkali extraction temperature, reaction time is obvious. The amount of sodium hydroxide, temperature, and time are variables that can be directly controlled in the actual process, and the data that can be directly accessed online during the control process and the prediction process. If the initial lignin content is known, the lignin content in pulp after alkaline extraction can be measured quickly and accurately based on the online parameters such as sodium hydroxide dosage, time and temperature, to provide the basis of fixing operation condition for the subsequent D_1 bleaching process. Especially in the case of adjusting or optimizing the bleaching process, the lignin content can be obtained quickly based on this method, to avoid more AOX caused by excessive use of ClO_2. As known to us, lignin content of pulp (Kappa value) is a strictly controlled parameter in the cooking process, and the increase of lignin content will increase the cost of subsequent bleaching and increase the generation of AOX. Considering that the parameters controlled by the cooking process are similar to the alkaline extraction process, therefore, the model may also be applied to the cooking process by appropriate modifying which can directly get the content of lignin.

The model is only a model of a certain period or a certain variable in the whole bleaching system, and is necessary to obtain the quality indexes (whiteness, kappa number, viscosity, etc.) of the bleached pulp because it is the intermediate function of the whole system model. In order to predict the total AOX production of the bleaching system, this model is also needed to predict the AOX produced by the subsequent bleaching (D_1) section. Based on the same method, the prediction models of other stages can be established, then the system model of pulp bleaching could be established to simulate the process of pulp bleaching. Systematic and comprehensive optimization of pulp bleaching processes to reduce bleaching pollutant emissions and bleaching costs can be achieved, which is also the main research direction in the future.

Conclusions

In this paper, the variation of sodium hydroxide dosage, reaction time and temperature with the different relative lignin content in pulp, was analyzed respectively, a measurement model for lignin content was established. The results showed the models can realize the function of prediction well. As the model established, a direct and simple method to obtain the lignin content of pulp emerged, and a reliable base is provided for determining the dosage of bleaching agent in the following bleaching section. The reasonable dosage of bleaching agent is helpful to reduce the pollutants generation.

When the prediction models of other stages are established, then the system model of pulp bleaching could be built, bleaching operation can be simulated and predicted. It has a practical significance to provide the possibility for achieving precise control and intelligent control of the bleaching process. Systematic and comprehensive optimization of pulp bleaching processes is the main research direction in the future.

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Conflicts of interest

We declare that all the authors have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Authors contribution

Yongjun Yin provided the guidance of thinking for this research and revised the paper as supervisor. Zhichao Ma completed paper and conducted a number of experiments. Shaoxu Chen conducted the experiments related to this paper. Yanying Zhou, Xiaodan Lu, Tingting Lin assisted conducting the experiments of this study.

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