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Determination of sodium alginate in algae by near-infrared spectroscopy

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ABSTRACT

In this study, the components of natural seaweed fiber were determined by near-infrared spectroscopy, and the qualitative analysis model of natural seaweed was established by using support vector machine algorithm based on principal component analysis. For the natural algae containing sodium alginate, a near-infrared spectrum analysis model for the content of sodium alginate was established by using multi-model method. The model has very good analytical and predictive ability. The model can be used to distinguish the composition and active component contents of natural algae quickly, and it also has the advantages of fast, nondestructive, convenient and so on.

Keywords: Near-infrared spectrum; Natural algae; Support vector machine algorithm; Multi-model method

1. Introduction

Sodium alginate is a by-product of iodine and mannitol extracted from kelp or sargassum of brown algae. Its molecule was β-D-mannitonic acid (β-D-mannuronic M) and α-L-guluronic acid (α-L-guluronic acid G) linked by (1 → 4) bond [1]. The nature of sodium alginate is a kind of natural polysaccharide, and its aqueous solution has high viscosity. Sodium alginate is widely used in food, medicine, textile, printing and dyeing, paper making, daily chemical and other products. And it is used as thickener, emulsifier, stabilizer, adhesive, sizing agent and so on [2]. The aqueous solution of sodium alginate has high viscosity, and has been widely used in food industry as thickener, stabilizer, emulsifier and so on. In the textile industry, sodium alginate is a widely used reactive dye color pulp, and its performance is superior to that of grain starch and other sizing materials [3,4]. The fabrics printed by dye color pulp with sodium alginate have bright patterns, clear lines, high color supply, uniform color, good permeability and plasticity [5]. It is the best dye color pulp in modern printing and dyeing industry, and has been widely used in cotton, wool, silk, nylon and other fabrics printing, and is especially suitable for the preparation of dyeing printing pulp. Mixing or replacing starch with seaweed gum can not only save a large amount of grain but also make the warp fiber not hairy, friction resistant, broken rate less, thus improve the weaving efficiency. Sodium alginate has pretty good dyeing effect on both cotton fiber and synthetic fiber.

The technological process for the preparation of sodium alginate is as follows: Dried or wet natural seaweed is crushed, washed and impurity removed, extracted with strong alkali water to clear crude alginate solution, precipitated by calcium chloride to obtain colored calcium alginate, decolorized, deflavored, treated with acid, and soluble impurities removed to obtain alginate precipitation. Sodium alginate was obtained by the reaction with sodium carbonate, and then dried, crushed and sifted to obtain sodium alginate powder [6,7]. The algae with high sodium alginate are kelp or sargassum from brown algae, but the alginate content of all kinds of algae purchased from the market differ greatly. In 2015, the yield of sodium alginate from a large-scale sodium alginate manufacturer in Qingdao was 11.8% [8]. The reason for the low output is that a large number of raw materials

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with low content or even no sodium alginate enter the production line, which results in a great waste of equipment and auxiliary materials. If the initial content of sodium alginate in seaweed raw materials can be detected and screened, the output rate of sodium alginate can be improved, the cost can be saved while the production efficiency can be increased. In 2016, the manufacturer began to sort the natural seaweed artificially, which increased the sodium alginate productivity by 16.2%. However, this method has strong subjectivity, poor sorting effect, low working efficiency and high labor cost, so it is difficult to meet the needs of large-scale fine sorting and classification of textile printing and dyeing enterprises. So that it is urgent to improve the sorting methods of the raw materials [9].

To solve the above problems, it is necessary to design an automatic preliminary screening method for natural algae. Traditional methods for determining the contents of sodium alginate are dissolution method and microscope method, but these methods have long detection cycle and high detection cost, and the industrial use of chemical reagents will also cause pollution to the environment. Compared with the traditional methods, near-infrared spectroscopy (NIR) is an advanced information measurement and information processing technology, which has the advantages of fast analysis speed and high efficiency [10]. However, in the process of industrial production, the test sample quantity of natural seaweed is very large, so it is impossible to select infrared spectrum manually. Therefore, it is necessary to design an intelligent learning algorithm to automatically sort infrared spectra. Nowadays, there are two popular artificial intelligence learning algorithms: neural network algorithm (BP) and support vector machine (SVM) algorithm [11]. Among them, BP is more suitable for solving more complex classification problems. It is characterized by high complexity, long learning time and large data demand. And it has the advantages of good effect on the classification of multi-component or complex systems, and is often used to simulate the complex activities of human thinking; while SVM is a kind of generalized linear classifier for binary classification of data by supervising learning. The decision boundary is the maximum margin hyperplane for solving learning samples. This algorithm has the characteristics of simple and fast processing speed, and has a good effect on binary classification. In this paper, the composition and content of natural algae were determined by NIR. Because infrared spectra are composed of simple two-dimensional tables, and industrial production requires rapid processing of a large number of sample data. Therefore, SVM method based on principal component analysis (PCA) and the multi-model method are used in modeling to establish a qualitative analysis model of natural algae by NIR, which was applied in determination of the presence of sodium alginate in natural algae [12]. This method gives full play to the unique advantages of SVM in the case of small sample and multi-dimensional, so is suitable for the establishment of near-infrared spectral discriminant model. The SVM method based on PCA can improve the accuracy of model discrimination. After the samples containing natural sodium alginate were separated by qualitative analysis model, the near-infrared spectrum analysis model of sodium alginate content was established by multi-model method. The multi-model method can make full use of spectral data, and the model has good stability. The prediction accuracy of the model can be improved by using only cotton samples.

2. Materials and methods

2.1. Experimental methods and materials

In order to establish the near-infrared spectrum analysis model of alginate content in seaweed raw material, the samples of seaweed raw material must be representative and the basic data of sodium alginate content in the sample should be accurate. In practice, there are two methods for obtaining data on the content of sodium alginate. The first method is to collect representative algal samples containing sodium alginate and measure the content of sodium alginate directly by chemical method. The data obtained by this method are representative, but the method has the disadvantages of heavy workload, complex chemical measurement process and serious pollution of chemical reagents [13]. Because of the long steps of the experiment, the error of each link may result in the inaccuracy of the final result. The second method is to mix pure sodium alginate with other components. By adjusting the weight ratio of sodium alginate to other components, a series of samples with different percentage of sodium alginate content are obtained. The operation of this method is relatively simple, and the accurate sample alginate content can be obtained by using this method. But to use this method, we should pay attention to whether the sample is representative or not.

The second method was used in this study. In order to make the samples representative, the project group collected the samples of seaweed from coastal areas of Bohai Sea, Shandong Province Huang Hai, the Yangtze River Delta, Fujian coastal areas and Pearl River Delta in 2017 or 2018 [14]. Samples of sodium alginate were obtained from different manufacturers in Liaoning, Shandong, Shanghai and Guangzhou in 2017 or 2018 [15]. The collected samples of seaweed raw materials were beaten by a plant pulverizer into a powder that could pass 80 mesh sieve, and a certain amount of powder was weighed with 1/10,000 scales at a time, and sodium alginate was mixed with the residue after production. Fifty samples containing sodium alginate and 15 samples without sodium alginate were obtained. The actual content of sodium alginate in the sample was determined by the actual value of weighing.

2.2. Main instruments and equipments

Fourier transform near-infrared spectrometer, Instrument Model: Antaris II, Manufacturer and address: Thermo Fisher Scientific, 168 Third Avenue, Waltham, MA USA 02451.

Samples of seaweed raw materials with different mass ratios were put into the sample pool and scanned at the speed of 1,200 nm/min in the region of 800–2,700 nm. The resolution was selected at 5 nm, and the near-infrared spectral data of the samples were collected. After each scan, the instrument subtracts the background spectrum from the sample spectrum, thus obtaining the final near-infrared spectral data of the sample. Each spectral data consist of 381 reflectivity values. Fig. 1 is a near-infrared spectrogram

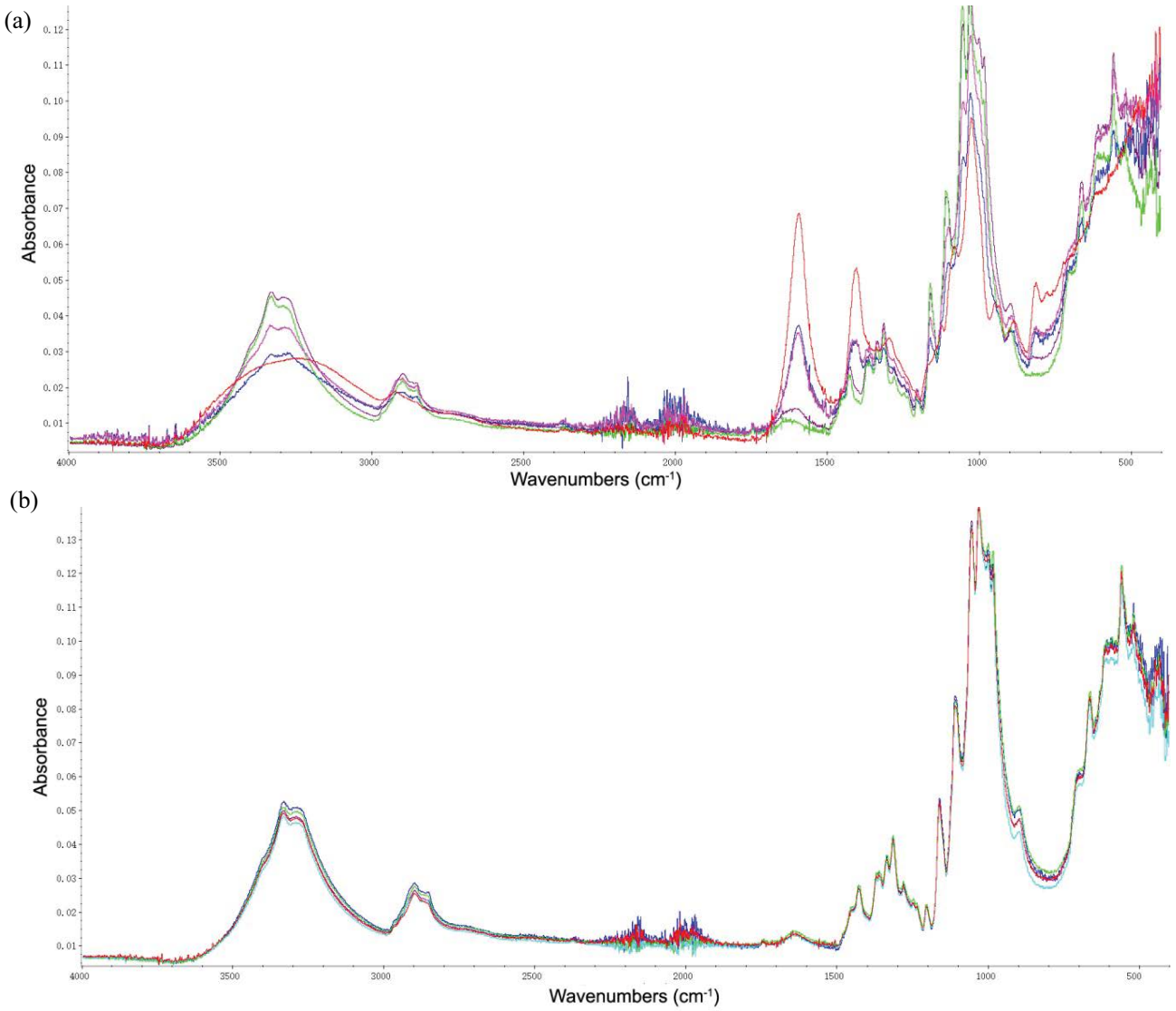

Fig. 1. Near-infrared spectra of samples of two seaweed raw materials: (a) sample spectra containing sodium alginate, and (b) sample spectra without sodium alginate.

of two samples of algae raw material, and the number of samples is 50 and 15.

It can be seen from Fig. 1 that there are some differences in the near-infrared spectra of the two seaweed raw material samples. The samples containing sodium alginate can be separated by NIR, so it is reasonable to use the modeling method described in the brief introduction. So it makes sense to use the modeling approach described earlier.

2.3. Modeling principles and methods

2.3.1. Qualitative analysis modeling principles and methods

In order to determine whether the samples contain sodium alginate or not, SVM method based on PCA is used to establish a qualitative analysis model of sodium alginate by NIR. SVM is a classification method proposed by Vapnik et al [16]. It maximizes the distance between two kinds of samples by finding the optimal classification hyperplane to separate the two kinds of samples. PCA is an effective method for dimensionality reduction. The model can be simplified by reducing the dimension of spectral data as input variables of SVM method.

61 samples were randomly selected from 161 seaweed samples to form the test group, and the remaining 100 samples were selected to form the training group. There were 38 samples containing sodium alginate and 13 samples without sodium alginate in the test group. And there were 61 samples containing sodium alginate and 18 samples without sodium alginate in the training group. The PCA of the spectral data of the training group shows that the interpretation rate of the first four principal component differences is greater than 90, which can fully reflect most of the information of the original 381-dimensional spectral data. The first four principal components of the spectral data are recorded as $x(1)$, $x(2)$, $x(3)$, $x(4)$. Then the equation of the optimal classification hyperplane can be set to, Eq. (1).

$$\omega^T x + b = 0 \tag{1}$$

In which $\omega = (\omega_1, \omega_2, \omega_3, \omega_4)^T, x\big| = [x(1), x(2), x(3), x(4)]^T$. The samples in the training group were numbered as 1,2,...,100. Suppose that the first four major components of sample i composition is vector, x_i if the sample i contains sodium alginate, it is labeled as $y_i = 1$, if not, it is labeled as $y_i = -1$.

In order to find the optimal classification hyperplane, the following optimization problems need to be solved, see Eq. (2):

$$\min \frac{1}{2}\omega^T\omega \qquad s,t, y_i(\omega^T x_i + b) \geq 1, i = 1,2,...,100 \tag{2}$$

The duality of the problem is given as follows:

$$\min \frac{1}{2}\sum_{i=1}^{100}\sum_{j=1}^{100}\alpha_i\alpha_j y_i y_j x_i^T x_j - \sum_{i=1}^{100}\alpha_i$$

$$\text{s.t.}\sum_{i=1}^{100}\alpha_i y_i = 0 \tag{3}$$

$$\alpha_i \geq 0, i = 1,2,...,100$$

quadprog function of MATLAB was used to getSolution of quadratic programming problem (3) α_1, α_2,…, α_{100}, then the solution of Eq. (2) is given in Eq. (4) as follows:

$$\omega = \sum_{i=1}^{100}\alpha_i y_i x_i \tag{4}$$

$$b = -\frac{1}{2}(\max_{y_i=-1}\omega^T x_j + \min_{y_i=1}\omega^T x_j)$$

Thus, the decision function for classification can be obtained in Eq. (5).

$$g(x) = \text{sgn}(\omega^T x + b) \tag{5}$$

The sample of the test group was numbered as 101, 102,..., 161, bringing the spectral data of the sample i into Eq. (5), the resulting value is recorded as $g(x_i)(101 \leq i \leq 161)$. If $g(x_i) = 1$, then the sample i is judged to be containing sodium alginate.

2.3.2. Quantitative analysis of modeling principles and methods

In this method, spectral data are grouped according to wavelength, and a submodel is established from each set of data, and then the prediction results of each submodel are weighted to get the final prediction results. After the weighted average, the prediction error of each submodel will be desalinated by other submodels, so the error of the prediction result after the weighted average is generally smaller than that of the single submodel.

In order to divide the training group and test group more reasonably, 99 algae samples containing sodium alginate were arranged according to the order of sodium alginate contents. The sodium alginate content in the sample m is recorded as z_m. Filter out 25 samples with a sequence numbers of $1 + 4N$ ($N = 0,1,\ldots,24$) to form a test group. The rest 74 samples were set into training group. And the sodium alginate of the test group could be represented by vectors $Z^V = (z_1, z_5, z_9, z_{13}, \ldots, z_{97})$. The cotton content of the training group can be similarly represented by vector Z^C.

The reflectivity of each wavelength in the spectral data of the test group forms a 25-dimensional vector.

These vectors were arranged in order of wavelength from large to small, and recorded as $X_1^V, X_2^V, X_3^V, \ldots, X_{381}^V$. The reflectivity of the corresponding training group was recorded as $X_1^C, X_2^C, X_3^C, \ldots, X_{381}^C$, in which each $X_i^C (i = 1,2,3,\ldots,381)$ is 74 dimensional vector. In order to reduce the multiplex collinearity between reflectivity, the method of selecting one reflectivity per 100 nm was adopted to obtain the data needed to establish the submodel. That is, the reflectivity of the training group was divided into 20 groups. The group i was consisted by $X_i^C, X_{i+20}^C, X_{i+40}^C, \ldots, X_{i+360}^C (1 \leq i \leq 20)$, ($X_{381}^C$ was abandoned). In this way, the prediction error of the submodel can be reduced.

Z^C and $X_i^C, X_{i+20}^C, X_{i+40}^C, \ldots, X_{i+360}^C$ were used to set up the submodel i ($i = 1,2,\ldots,20$). Suppose that Z^C could be represented by Eq. (6) as follows:

$$Z_i^C = a_{i0} I^C + \sum_{k=1}^{19}\alpha_{ik} X_{i+20(k-1)}^C \tag{6}$$

Approximate representation, we got Eq. (7):

$$Z^C = Z_i^C + \varepsilon_i \tag{7}$$

The vector I^C in Eq. (6) represents the 74 dimensional vectors whose total component was 1, $\alpha_{ik} (k = 0,1,2,\ldots,19)$ is an undetermined constant. The vector ε_i in Eq. (7) represents error vector. Using the regression function regress of MATLAB is to get the value of parameter $\alpha_{ik} (k = 0,1,2,\ldots,19)$ and Z_i^C corresponding goodness of fit $R^2(Z_i^C)$.

The better the fitting degree is, the better the prediction effect of the corresponding submodel is.

So in this paper, $\sum_{i=1}^{20} q_i Z_i^C$ was taken as Z^C Approximate vector, and q_i was taken as weight coefficient, see in Eq. (8).

$$q_i = \frac{\left(1 - R^2\left(Z_i^C\right)\right)^{-2}}{\sum_{j=1}^{20}\left(1 - R^2\left(Z_j^C\right)\right)^{-2}} \tag{8}$$

For the test group, Eq. (9) is obtained by imitating Eq. (6).

$$Z_i^V = a_{i0} I^V + \sum_{k=1}^{19} a_{ik} X_{i+20(k-1)}^V \tag{9}$$

In which I^V is the 25-dimensional vector with total component as 1, and then $\sum_{i=1}^{20} q_i Z_i^V$ is the predictive value vector of Z^V.

The number of parameters in the submodel was closely related to the final prediction effect of the model. Proper reduction of the number of parameter α_{ik} can further reduce

the multiple linearity between the reflectivity and improve the prediction accuracy of the model. In general, when the parameters in each submodel are reduced to about 9, the final prediction effect of the model is better. If the parameters are too few, the prediction error of the submodel will be large because of the under-fitting, which leads to increasing of the error of the prediction results after the weighted average. In this paper, the parameters in each submodel were reduced to 8. For specific methods of reducing parameters, refer the study by Jing and Hou [12].

3. Results and discussions

For the qualitative analysis model in section 2.3.1, the solution of Eq. (2) can be obtained by the method of this summary, see Eq. (10).

$$\omega = (0.0056, 0.0333, -0.0365, -0.0384)^T, b = 6.4404 \quad (10)$$

So see Eq. (11) for the decision function

$$g(x) = \mathrm{sgn}(0.0056x(1) + 0.0333x(2) - 0.0365x(3) - 0.0384x(4) + 6.4404 \quad (11)$$

Using 61 samples of the test group to test the model, the result showed that the accuracy of model discrimination is 100. It was shown that the NIR qualitative analysis model established on the SVM method based on PCA can well separate the samples of waste textiles containing and without cotton. If we do not use the method in section 2.2.1, but simply use the SVM method to model, and if the PCA of the spectral data of the sample were not done, the resolution of the model would become worse. In the case where the test group and the training group remain unchanged, the accuracy of the model determination was about 98%, and one sample was misjudged. It can be seen that the SVM method based on PCA is a better method to establish the qualitative analysis model of near infrared spectrum. For the quantitative analysis model in Section 2.3.2, the weight coefficient of q_i (i = 1,2,…,20) in each sub-model can be obtained by the method in this section, they are 0.0593, 0.0661, 0.0444, 0.0498, 0.0404, 0.0414, 0.0329, 0.0651, 0.0330, 0.0321, 0.0579, 0.0538, 0.0332, 0.0366, 0.0766, 0.0661, 0.0598, 0.0465, 0.0494, 0.0557. The predicted and actual values of the sample alginate content are shown in Table 1.

Sample pairing t test was taken and the predicted and actual values of sodium alginate were gotten in Table 1. Set the significant level α = 0.05, we got the corresponding p-value of the data 0.201 by using the SPSS statistical software, and which was far bigger than 0.05. So there is no significant difference between the predicted value and the actual value. According to the data in Table 1, the fitting optimization degree of the model is 0.8952, and the correlation coefficient between the predicted value and the actual value of the sodium alginate content in the test group is 0.9504, so the prediction effect of the model is good.

When using the method in Section 2.3.2 to establish the near-infrared spectrum analysis model of sodium alginate content, if the two seaweed samples are mixed to model the model, the prediction ability of the model becomes worse. In order to compare the two models, the test group in Section 2.3.2 was still used as the present test group, and the multi-model method was used to establish the Jinyong external spectrum analysis model of sodium alginate content. The modeling results show that the goodness of fit of the model was 0.8532, and the correlation coefficient between the predicted value and the actual value of sodium alginate content in the test group was 0.9342, both of which were not as high as those in this section. Therefore, it is necessary to establish a qualitative analysis model of seaweed raw material by NIR, and to select the samples containing sodium alginate with the model. In practical application, the qualitative analysis model can be used to determine whether the seaweed raw material contains sodium alginate or not at first, and then the quantitative analysis model can be used for the seaweed raw material containing sodium alginate. It can also be seen from Table 1 that the two sample points with the largest and smallest actual content of sodium

Table 1
Comparison of predicted and actual values for sodium alginate content in test group samples

Number	Prediction values of sodium alginate content	Actual values of sodium alginate content	Number	Prediction values of sodium alginate content	Actual values of sodium alginate content
1	18.55%	1.00%	40	44.25%	57.40%
4	25.28%	12.68%	43	57.08%	59.80%
7	21.99%	20.80%	46	54.60%	62.75%
10	19.91%	25.60%	49	57.43%	58.43%%
13	30.28%	30.28%	52	63.45%	62.84%
16	31.84%	33.64%	55	65.21%	64.82%
19	45.01%	37.00%	58	68.78%	64.65%
22	29.68%	40.75%	61	72.34%	70.45%
25	48.49%	44.00%	64	74.65%	73.73%
28	42.41%	47.00%	67	78.85%	75.22%
31	61.85%	50.00%	70	80.54%	81.63%
34	53.41%	52.40%	73	84.03%	83.51%
37	53.42%	54.80%			

alginate deviate a little more, which indicates that the prediction ability of the model for samples with almost pure sodium alginate or almost no sodium alginate is slightly poor. The reason for this problem may be related to the wide range of changes in the content of sodium alginate in the sample. In order to improve the prediction ability of the quantitative analysis model for the samples with almost pure alginate and almost no sodium alginate, the prediction model should be established by using the samples with higher sodium alginate content and lower sodium alginate content, respectively.

Recalculating the parameters $a_{ik}(k = 0,1,2,\ldots,19)$ for each submodel in Section 2.3.2 with samples containing more than 70% sodium alginate in the training group, and then recalculating the weight coefficients of each submodel. A prediction model for high sodium alginate content samples was established. Samples with sodium alginate contents of less than 30% in the training group in this section were used to establish a prediction model suitable for a low sodium alginate content sample. The new model has improved the prediction results of samples with almost pure alginate and almost no sodium alginate. For example, the new prediction values of the two samples with the largest and smallest actual cotton contents in the test group were 87.62% and 6.40%, respectively, which was a significant improvement over the predicted results in Section 2.3.2. The method of using the new model is as follows: First, the prediction model of sodium alginate content in Section 2.3.2 is used to obtain the preliminary prediction value of sodium alginate content in the sample. If the preliminary prediction value is higher than 70% or less than 30%, then the new model could be used to predict the content of sodium alginate in the sample, and the result could be used as the final prediction value of the content of sodium alginate in the sample. If the preliminary prediction value is not in the range of 30% to 70%, the preliminary prediction value could be used as the final prediction value for the sample sodium alginate content. Using the above method, the final predicted values of sodium alginate content in each sample in Section 2.3.2 test group were obtained. The corresponding goodness of fit and correlation coefficient of these final predicted values were 0.9194 and 0.9616, respectively. This is an improvement over the values in Section 2.3.2. Therefore, a better prediction result can be obtained by using the new model and the prediction model in Section 2 intelligently.

4. Conclusion

In this paper, SVM method based on PCA was used to establish a qualitative analysis model for natural seaweed samples by NIR. The model can separate the samples of natural algae with and without sodium alginate, and the accuracy of the test reached to 100%. For the samples containing sodium alginate, the near-infrared spectrum analysis model of alginate content of seaweed sample was established by using multi-model method. By using the prediction model of sodium alginate content reasonably, the goodness of fit of the model can reach 0.9194. The correlation coefficient between the predicted value and the actual value of sodium alginate content in the test group was 0.9616. The necessity of using the above two modeling methods was also discussed. By using the above-mentioned qualitative analysis model and the quantitative analysis model, the content of the sodium alginate of the natural seaweed sample can be well determined. This new method is expected to be used for rapid determination of natural seaweed samples or some other natural algae components and content. The results of this paper lay a foundation for fine sorting and classification of natural algae samples.

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