Quantitative Detection of Components in Polymer-Bonded Explosives through Near-Infrared Spectroscopy with Partial Least Square Regression

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ABSTRACT: The components in polymer-bonded explosive X, including cyclotetramethylene-tetranitramine, paraaffin, and polytetrafluoroethylene, were determined using near-infrared (NIR) spectroscopy. Using partial least squares as the multivariate calibration method, quantitative calibration models for components in X were verified internally and externally. The possible combinations of eight general spectral pretreatment methods and different bands of the scanning spectral region (12,500–4000 cm\(^{-1}\)) were established. The models were analyzed, evaluated, and optimized via the fitting effect. The data were combined with the mathematical meaning of the model spectral pretreatment methods and the chemical significance of the modeling spectral bands. Prediction performance offered optimal quantitative calibration models. Paired bilateral Student’s t tests show that there is no significant difference between the values obtained by NIR and chemical analysis methods, and the NIR method has good accuracy. Moreover, the precision of the NIR method is better than that of the chemical method, and the analysis time is reduced from 2 days to a few minutes.

1. INTRODUCTION
Near-infrared (NIR) light is an electromagnetic wave between the visible and mid-infrared spectra with wavelengths from 0.8 to 2.5 μm (wavenumbers from 12,500 to 4000 cm\(^{-1}\)). This is the earliest known nonvisible spectrum. NIR spectroscopy is an indirect analytical method for quickly identifying the composition of unknown samples according to their spectra; calibration models were established via chemometrics using information on groups, composition, and properties given by NIR spectra and the standard or approved reference methods. NIR spectroscopy has two advantages: ease of obtaining the spectra over the visible region and rich information in the infrared region. It has been one of the fastest growing and most striking spectral analysis tools since the 1990s. Classical chemical methods (capacity and gravimetric method), separation technology, preseparation analysis technologies, and chemometrics can determine the content of components in explosives. However, conventional chemical analysis methods generally use solvent extraction to extract analytes according to their solubility. The component contents are then determined via gravimetric methods. Solvent elution methods are gradually being replaced by new separation methods because of their time-consuming operation and serious environmental pollution. Chromatography\(^2\)–\(^{13}\) is a powerful separation method for analysis of explosives. It is a convenient, rapid, and reliable tool to measure the compositions of explosives. Analysis without preseparation uses polargraphy,\(^14\)–\(^{17}\) colorimetric methods,\(^18\)–\(^{19}\) and infrared spectroscopy.\(^20\)–\(^{21}\) Preseparation-free analysis might meet the requirement of rapid analysis in the detection of explosives and is more reliable than chemical methods for trace analysis. Although these methods are relatively convenient and rapid, they are still time-consuming, laborious, and toxic. They must be done offline and are destructive. However, NIR spectroscopy offers a new approach to address these problems.

In recent years, NIR spectroscopy has been successfully applied to the composition analysis of explosives (such as cyclotrimethylene-trinitramine (RDX),\(^22\)–\(^{23}\) cyclotetramethylene-tetranitramine (HMX),\(^23\) and HMX-based polymer-bonded explosives (PBX))\(^24\) because it is simple, rapid, nondestructive, and environmentally friendly and offers simultaneous measurement of multiple components. Based on the diffusion reflection model, Hardmeyer et al.\(^25\) determined the concentration of a...
stabilizer in single- and double-base propellants using a scanning NIR spectrometer. The sample was not pretreated. Standard chemometrics was used to establish the standard scale model whose prediction results were within the range of HPLC analysis values. Modiano26 used NIR harmonic absorption spectroscopy to detect the concentration of CO and N₂O during the combustion of nitramine propellant XM39 and homogeneous gunpowder JA2. An IR-sensitive photodiode detector matrix was used in experiments combined with the least squares fitting technology. Xu et al.27 rapidly analyzed the explosives real-time detection.

Table 1. Comparison Table of HMX Calibration Modeling Parameters

| No. | M | spectral pretreatment methods | wavelength range/cm⁻¹ | RMSECV | K | R | RMSEP% |
|-----|---|--------------------------------|------------------------|--------|---|---|--------|
| 1   | 67| minimum–maximum normalization | 7502.2–6098.2; 5450.2–4597.7 | 0.458  | 15 | 0.9808 | 0.511  |
| 2   | 68| first derivative + SNV     | 5450.2–4597.7          | 0.507  | 10 | 0.9775 | 0.455  |
| 3   | 68| first derivative + MSC     | 7502.2–6098.2; 5450.2–4597.7 | 0.526  | 7  | 0.9753 | 0.411  |
| 4   | 70| first derivative + SNV     | 7502.2–4597.7          | 0.551  | 10 | 0.9709 | 0.294  |
| 5   | 70| minimum–maximum normalization | 5450.2–4597.7          | 0.558  | 9  | 0.9717 | 0.439  |
| 6   | 70| vector normalization       | 7502.2–6098.2; 5450.2–4597.7 | 0.564  | 14 | 0.9715 | 0.429  |
| 7   | 68| first derivative + MSC     | 7502.2–4597.7          | 0.564  | 10 | 0.9714 | 0.338  |
| 8   | 70| first derivative + SNV     | 7502.2–6098.2; 5450.2–4597.7 | 0.594  | 12 | 0.9688 | 0.509  |
| 9   | 68| second derivative          | 7502.2–4597.7          | 0.608  | 10 | 0.9666 | 0.497  |

(No.: model number; M: model sample number after eliminating outliers; K: dimension of the principal component; R: cross validation correlation coefficient).

Table 2. Comparison Table of Paraffin Calibration Modeling Parameters

| No. | M | spectral pretreatment methods | wavelength range/cm⁻¹ | RMSECV | K | R | RMSEP% |
|-----|---|--------------------------------|------------------------|--------|---|---|--------|
| 1   | 70| MSC                            | 12493.3–7498.3; 5450.2–4597.7 | 0.115  | 4  | 0.9816 | 0.081  |
| 2   | 69| elimination constant offset    | 12493.3–6098.2; 5450.2–4597.7 | 0.117  | 4  | 0.9808 | 0.093  |
| 3   | 70| MSC                            | 12493.3–6098.2; 5450.2–4597.7 | 0.117  | 4  | 0.9810 | 0.066  |
| 4   | 68| SNV                            | 6102–4597.7            | 0.117  | 8  | 0.9769 | 0.123  |
| 5   | 70| MSC                            | 12493.3–7498.3; 6102–4246.7 | 0.125  | 6  | 0.9770 | 0.101  |
| 6   | 70| minimum–maximum normalization | 12493.3–6098.2; 5450.2–4597.7 | 0.126  | 3  | 0.9751 | 0.133  |
| 7   | 72| first derivative + SNV         | 12493.3–5446.3; 4601.6–4246.7 | 0.128  | 6  | 0.9770 | 0.078  |
| 8   | 70| SNV                            | 12493.3–6098.2; 5450.2–4597.7 | 0.129  | 2  | 0.9737 | 0.057  |
| 9   | 71| minimum–maximum normalization | 12493.3–6098.2         | 0.130  | 5  | 0.9762 | 0.072  |

2. RESULTS AND DISCUSSION

2.1. Establishment and Optimization of the Quantitative Calibration Models. The quantitative calibration models for each component in PBX X were established with various possible combinations of general spectrum pretreatment methods and the different bands in the scanning spectral region. The first few models with a smaller root mean square error of cross validation (RMSECV) for each component were selected and evaluated by the root mean squared error of prediction (RMSEP) of the validation set samples to get the optimal models. The modeling parameters of models for HMX, paraffin, and polytetrafluoroethylene (PTFE) are listed in Tables 1–3, respectively.

2.1.1. HMX. The molecular structure of octogen (HMX, 1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane) is shown in Figure 1a. HMX has both C=O, n, and polytetrafluoroethylene (PTFE) are listed in Tables 1–3, respectively.
strong absorption in the mid-infrared region of 1560 cm\(^{-1}\). The second overtones (three times the frequency of the fundamental vibration) appear in the NIR region (Figure 2a).

The cross validation correlation coefficient (R) values of all models in Table 1 are above 0.96, which means that the models have a good fit. There are two possible reasons why the absorption region of 5450–4597 cm\(^{-1}\) was included in the modeling range of all models. One is that only HMX molecules in the X explosive contain \(\text{NO}_2\) functional groups. The second overtones of \(\text{NO}_2\) stretching vibration appear around 4610 cm\(^{-1}\) in the NIR region with strong characteristics and high discrimination; these can effectively improve the reliability of the model. The other reason is that the other components acted as background disturbances in the process of establishing the HMX quantitative calibration model. The 5450–4597 cm\(^{-1}\) absorption bands contain the disturbance information such as the second overtones of \(\text{C}^\equiv\text{O}\) stretching vibration in other component molecules and the third overtones of \(\text{C}^\equiv\text{F}\) stretching vibration in the PTFE molecule. Therefore, modeling with these functional group absorption bands can best reflect the content and property changes of components and can increase model robustness.

The first three models with a smaller RMSEP are model 4, model 7, and model 3. Compared with model 3, the modeling bands of model 4 and model 7 changed from 7502–6098 cm\(^{-1}\) + 5450–4597 cm\(^{-1}\) to 7502–4597 cm\(^{-1}\), which adds the first overtones of \(\text{C}^\equiv\text{H}\) stretching vibration (6098–5450 cm\(^{-1}\)). The RMSEP of model 4 and model 7 decreased from 0.411% to 0.338% and 0.294%. This means that using the first overtones of \(\text{C}^\equiv\text{H}\) stretching vibration to model can effectively improve the model prediction performance. The first derivative is used in a combined spectral pretreatment method for all three models. The NIR absorptions of the X explosive are the sum of the NIR absorptions of each component, and the content of HMX is the highest. The spectral S/N ratio is also high, and the spectral resolution can be effectively improved just using the method of first derivative. Eliminating the baseline drift could reduce the interference from noise amplification caused by differential treatment on spectral signals, thus improving model quality. The fitting effect and prediction performance of model 4 are better than those of model 7, indicating that standard normal variable transformation (SNV) is better than multiplicative scatter correction (MSC) in eliminating the scatter correction effect.

Model 5 was established with the spectral band of 5450.2–4597.2 cm\(^{-1}\) as the only modeling band. The small RMSECV and RMSEP of model 5 indicate that it is critical to improve the model quality to use this band as the modeling band. The RMSECV of model 1 is the smallest, and its RMSEP is the largest in the nine models, indicating that model 1 is over-fitted. Its principal factor number is the largest, which also reflects this point.

In summary, model 4 is an optimal HMX calibration model due to its reasonable spectral pretreatment method and choice of principal factor number. Model 4 has good fitting effects and the best prediction performance.

2.1.2. Paraffin. The molecular structure of paraffin, \(\text{CH}_3(\text{CH}_2)_n\text{CH}_3\) (Figure 1b), has \(\text{C}^\equiv\text{H}\) groups that have abundant absorption in the NIR region (Figure 2b). As shown in Table 2, R for all models is greater than 0.97, indicating the goodness of fitting, and K is smaller than the other components because there were many \(\text{C}^\equiv\text{H}\) groups in paraffin. The three models with the best prediction performance are model 8, model 3, and model 9. The RMSEP of model 8 and model 3 decreased from 0.072% to 0.057% and 0.066%, respectively, versus model 9 because of the additional functional group band 5450.2–4597.7 cm\(^{-1}\) in models. The K decreased from 5 to 2 and 4, respectively, both of which indicated that this functional group band could increase the quality of models. Model 8 and model 3 had the same sample numbers and spectral bands for modeling but different spectral pretreatment methods; thus, the prediction performance of model 8 was better than that of model 3 because SNV was more suitable for eliminating scattering effects than MSC. The lower K of model 8 than model 3 also provided evidence for the better suitability of SNV. After comprehensive consideration, the optimum quantitative calibration model of paraffin was finally determined to be model 8.

The bands used to establish the optimum model for paraffin did not contain the first overtones of \(\text{C}^\equiv\text{H}\) stretching vibrations. The second and third overtones of \(\text{C}^\equiv\text{H}\) stretching vibrations and the combination bands of \(\text{C}^\equiv\text{H}\) stretching and...
bending vibrations indicated that these bands could clearly reflect the information of C–H in paraffin.

2.1.3. Polytetrafluoroethylene (PTFE). The molecular structure of PTFE is shown in Figure 1c. There was a C–F stretching vibration absorption of PTFE at 1240 cm$^{-1}$, and its third overtones were in NIR (Figure 2c). The model fitting effect of PTFE was worse than that of other components (largest R was 0.9589) because there were no hydrogen-containing groups but only C–F bonds in PTFE. Compared with the other components, the modeling principal component dimension of PTFE was larger due to the weak intensity and the easy interference of the third overtones of C–F stretching vibration. The larger principal component dimension of PTFE made the relationship between the content of PTFE and the NIR spectrum more accurate, thus establishing the benefits of the model. The third overtones of C–F stretching vibration in PTFE had peaks at 5450.2–4597.7 cm$^{-1}$ and showed the molecular information; therefore, all PTFE models in Table 3 contained this band.

Table 3 shows that the best prediction performance models are model 7 and model 6—both of these have almost the same prediction performance. The RMSEP of model 7 and model 6 is 0.222% and 0.225%, respectively, although they have different spectral pretreatment methods (SNV for model 6 and no treatment for model 7). It is better for model pretreatment to use the SNV method considering the scattering effect, sample particle size, and size distribution. Therefore, model 6 was determined to be the optimum quantitative calibration model for PTFE.

2.1.4. The Optimum Quantitative Calibration Model of Each Component in PBX X. The optimum quantitative calibration model for each component in PBX X is shown in Table 4. These models have good fitting effects and prediction performance; the different modeling parameters show the

![Figure 3. Cross validation scatter plot of the optimum model for (a) HMX, (b) paraffin, and (c) PTFE.](image_url)

![Figure 4. Relationship of RMSECV and principal factor dimension of the optimum model for (a) HMX, (b) paraffin, and (c) PTFE.](image_url)

![Figure 5. Pretreated NIR pattern of the optimum model for (a) HMX, (b) paraffin, and (c) PTFE.](image_url)
differences in chemical structure and content between the components in PBX X. The cross validation scatter plot, the relationship of RMSECV and principal factor dimension, and the pretreated NIR pattern of the optimum models are shown in Figures 3–5, respectively.

2.2. The Comparison of NIR Prediction and Chemical Analysis. 2.2.1. The Evaluation of Accuracy. The optimum models were used to predict the 14 samples in the prediction set to further investigate the quality and application effect of models. The results were compared with the chemical analysis results. Paired bilateral Student’s t tests were used for the prediction and chemical analysis values of the 14 samples. Meanwhile, δ (defined in eq 1) and δ̅ (the average value of δ for the 14 samples) were used to evaluate the accuracy of NIR. The results were compared with the chemical analysis values. The residuals between NIR prediction values and chemical analysis values of the components in 14 samples are shown in Table 6.

\[ \delta = \frac{\text{Prediction value of NIR}}{\text{Chemical analysis value}} \times 100\% \]  

(1)

Table 5. δ̅ and |δ|calculate of the Components

| component | HMX | paraffin | PTFE |
|-----------|-----|---------|------|
| δ̅/ (%)   | 100.09 | 101.35  | 111.03 |
| | 0.874  | 1.100  | 1.340  |

δ̅ of each component is close to 100%, which shows that NIR prediction values are basically consistent with the chemical values. δ̅ of PTFE deviates the most from 100% relative to that of the others because there is no C–H group in PTFE, and the content of PTFE is close to the detection limit of the NIR method. Given a significance level of 0.05, t(0.05, 13) is equal to 2.160; this value is greater than |δ|calculate of all components, which indicates that there is no significant difference between the NIR prediction and the chemical analysis results. The residuals between NIR prediction values and chemical analysis values of the components in 14 samples are shown in Table 6.

2.2.2. Evaluation of Precision. The optimal quantitative calibration models were used to predict 10 samples in the same batch. The standard deviation of NIR and chemical methods are shown in Table 7. The results show that the precision of NIR is better than that of chemical methods for HMX and paraffin. The PTFE model has relatively worse prediction performance due to no C–H bands and low content; thus, the precision of NIR is slightly lower.

3. CONCLUSIONS

In this study, the components in mixed explosives were quantitatively analyzed with NIR spectroscopy, and a rapid measurement method was developed for the components in the X explosive. This method offers a new way to develop online explosive analysis. Internal interaction validation and external validation of the NIR quantitative calibration models of components in the X explosive were conducted. The results show that the models have good fitting effects and reliable prediction performance. Among the NIR diffuse reflection spectral pretreatment methods, SNV is better than MSC. The first derivative + SNV is the best when the S/N ratio is high. Compared with chemical analysis methods, the results show that there are no significant differences between the NIR prediction values and the chemical analysis values; however, the analysis time is significantly reduced. It was concluded that NIR analysis can replace chemical methods for the rapid detection of components in a mixed explosive. NIR spectroscopy has an increasingly important role in quality control and online analysis of explosives.

4. EXPERIMENTAL SECTION

4.1. Preparation and Division of Samples. The reagents used were received and were provided by Xi’an Modern Chemistry Research Institute. According to the production process of PBX X, 65 samples with different ratios of components were prepared with a water suspension method. In addition to the 65 self-made samples, 31 X explosives of different batches were collected to set up models. All 96 samples were divided into three groups: calibration set, validation set, and prediction set.

The calibration set had a total of 72 samples including 7 X explosives samples and 65 self-made samples. The validation set was used to estimate and optimize models and was composed of 10 samples randomly taken from the remaining 24 samples. The prediction set was mainly used to compare the values obtained by the optimal models and chemical analysis and contained the last 14 samples.

4.2. Sample Certification. The reagents used in the sample certification process are of analytical grade. The chemical analysis for X explosive composition is described as follows: the saturated petroleum ether solution of HMX is used to separate paraffin, and then, HMX is decomposed using hot concentrated sulfuric acid. PTFE is obtained by washing the sample using the mixture of acetone and ethyl acetate. The contents of all components are obtained by gravimetric methods, while each sample is measured twice and averaged.
4.3. Spectral Acquisition. The sample was placed in a 5-cm-diameter sample cup and shaken gently to smooth the surface, and the spectrum was collected by diffuse reflection on a BRUKER MPA FT-NIR preheated for 2 h with a PbS detector. The sample cup was rotated at a constant speed during spectral collection to reduce errors due to sample loading and nonuniformity of sample particles. The samples were scanned 64 times with a resolution of 8 cm⁻¹ in the spectrum region of 4000–12,500 cm⁻¹. Each sample was measured three times, while the background was scanned every time; the average spectrum was taken for modeling. All of the abovementioned conditions for spectrum acquisition are optimal conditions. The optimal resolution (or scanning times) was determined by the RMSEP and R of the models (or signal to noise) and scanning time.

4.4. Model Establishment, Optimization, and Evaluation. The quantitative calibration models for components in the X explosive were established using BRUKER OPUS 6.0 spectrum analysis software using PLS as the multivariate partial least square; RDX, cyclotrimethylene-trinitramine; HMX, cyclotetramethylene-tetranitramine; PTFE, polytetrafluoroethylene; MSC, multiplicative scatter correction; SNV, standard normal variable transformation; RMSECV, root mean square error of cross validation; RMSEP, root mean squared error of prediction.

**Notes**

The authors declare no competing financial interest.

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**ABBREVIATIONS AND SYMBOLS**

**Abbreviations**

PBX, polymer-bonded explosive; NIR, near-infrared; PLS, partial least square; RDX, cyclotrimethylene-trinitramine; HMX, cyclotetramethylene-tetranitramine; PTFE, polytetrafluoroethylene; MSC, multiplicative scatter correction; SNV, standard normal variable transformation; RMSECV, root mean square error of cross validation; RMSEP, root mean squared error of prediction.

**Symbols**

R, cross validation correlation coefficient; NO, model number; M, model sample number after eliminating outliers; K, the dimension of the principal component; δ, percentage of NIR prediction value to chemical analysis value; ̄δ, the average value of δ for the 14 samples in the prediction set; |t|_calculated, the calculated value in the paired bilateral Student’s t tests; S_{NIR}, standard deviation of NIR prediction; S_{Chemical}, standard deviation of the chemical method.

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