Application Of Partial Least Squares And Spectrophotometry For Determination Of Louramide

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ABSTRACT

The aim of this study was determination of lauramide as a nonionic surfactant in liquid detergent multivariate calibration. A partial least squares (PLS) algorithm is used to perform the calibration. The concentration of lauramide for experimental calibration matrix was varied between 0.005- 0.02(g/ml). Absorbance data of the calibration standards were taken between 200–600 nm with UV-vis spectrometer. Then absorbance data and concentration were fed to model as input and output, respectively. For achieving the best model parameters of model were evaluated. In other step, first order derivative spectra were considered as input of model and parameters of model were tested.The relative errors in each step were calculated. Relative error less than 2.7% for zero and first derivative spectral data set respectively, were obtained.
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INTRODUCTION

Lauramide is a nonionic surfactant which used in liquid detergent. It is cheap, non-toxic, improve leaf surface spreading and sticking and improve herbicide absorption. The determination of lauramide in finished products presents interference problems that are not well-solve and demand HPLC methods, frequently preceded by laborious solvent extraction[1-5].

Multivariate calibration relates easily obtainable signals,e.g. spectra, to properties of interest that are more difficult to directly measure typically the concentration of the components in the system. The main goal of multivariate calibration is focused on developing a robust regression model that will give reliable prediction of a certain response from a set

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of considered predictors in order to ensure the accurate determination of future samples. There are many multivariate calibration methods, including partial least squares (PLS) and principle component regression (PCR).

Partial least squares (PLS) has become a usual tool for multivariate calibration models, because of the quality of the obtained calibration models and the ease of its implementation. It allows a rapid determination of components, usually with no need of a prior separation for analysis. The theoretical relationship among PLS has been treated extensively in the literature[6].

In this paper, a UV-vis spectrophotometric method is proposed for determination of lauramide at the presence of the related matrix of liquid detergent e.g. Sodium Lauryl Ether Sulfate, Betaine, Glycerin, Color and Essence using partial least squares (PLS) multivariate calibration.

EXPERIMENTAL

Apparatus and software

UV-vis spectrophotometer (UVIKON 922) was used for absorbance measurements in the wavelength region of 200 – 600 nm and 1 cm pathlength quartz spectrophotometric cells. MATLAB 6.5.0 (Math Works) and the related toolbox was applied for calculation.

Reagent and procedure

All solutions were prepared with analytical grade reagents. Stock solution was prepared by dissolving 10 g of lauramide in distilled water and diluting to 1000 ml. Matrix solution was prepared by dissolving 14.28 g of sodium lauryl ether sulfate, 21.85 g of betaine, 21.87 g of glycerin, 0.05 g of color and 2.15 g of essence respectively, in distilled water and diluting to 1000 ml. Absorption spectra of aqueous solution of lauramide and each compounds of the matrix between 200 and 600 nm are shown in figure 1.

Sixteen standard solution were prepared by dissolving suitable volumes of stock solution of lauramide and the related matrixes in 100 ml volu-
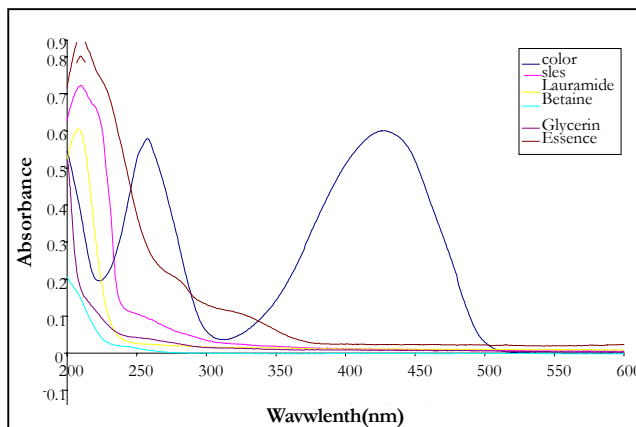

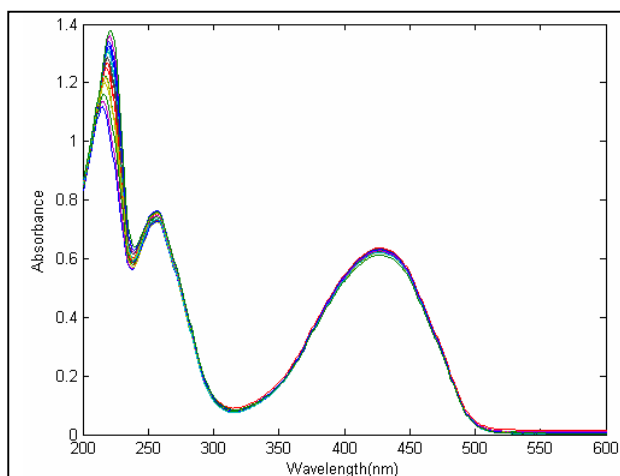**Figure 1: Absorption spectra of lauramide and components of matrix**

Figure 2(a): Zero order derivative spectra of standard solutions containing lauramide and the related matrixes
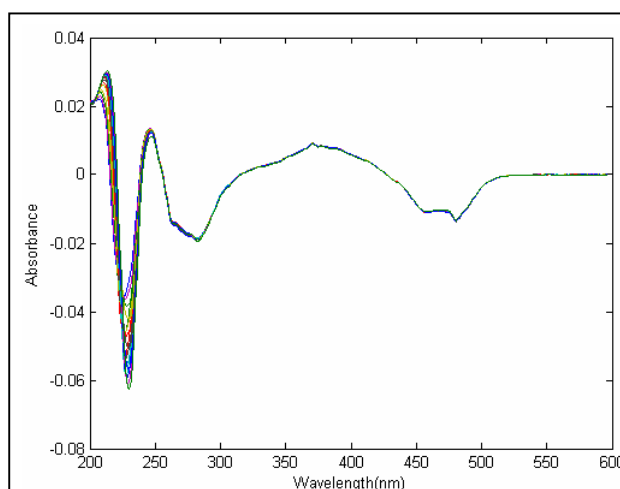

Figure 2(b): First order derivative spectra of standard solutions containing lauramide and the related matrixes

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metric flask and completed to the final volume with distilled water. The final concentration ranges of these solutions varied between 0.005–0.020 (g/ml) for lauramide. The concentration of the related matrixes in all these standard solutions were kept constant. In other step first order derivative spectra of these solutions were calculated. Figure 2 shows the absorbance spectra of standard solutions between 200 and 600nm in (a) zero-derivative spectra (b) first-derivative spectra.

RESULTS AND DISCUSSION

As can be seen from figure 1. lauramide, sodium lauryl ether sulfate and essence exhibited overlapped bands in spectral region 200-240 nm. Quantitative by the traditional univariate calibration method was unsuitable here, so multivariate PLS regression calibration was used instead. PLS methodology entails choosing a wavelength range and a number of factors leading to acceptably accurate and precise results. For this purpose must be found best model. For constructing model standard solutions randomly were separated in two sets which called calibration set and testing set.

Calibration set used for design the model. Several parameters and factors for achieving the best model were evaluated. In the best model different spectral regions were evaluated and the wavelength region of 221-241nm was selected as most convenient spectral region. The predicted results for lauramide in this region are show in TABLE 1.

The selection of the number of factors used in the calibration with PLS is very important for achieving the best prediction. As a first approach, the number of factors were estimated by cross-validation method. The cross-validation method employed was to eliminate only one sample at a time and then PLS calibrate the remaining standard spectra [7].By using this calibration the concentration of the sample, left out was predicted. This process was repeated until each standard had been left out once. One reasonable choice for the optimum number of factors would be that number (h*) which yielded the minimum PRESS.

TABLE 1: Relative error (RE) and results obtained in the determination of lauramide using PLS in the optimum wavelength range and zero-derivative spectrophotmetry for testing set

Sample	Actual(ml/g)	Prediction(ml/g)	RE(%)
1	0.0200	0.0195	2.5
2	0.0075	0.0073	2.6

TABLE 2: Relative error (RE) and results obtained in the determination of lauramide using PLS in the optimum wavelength range and first-derivative spectrophotometry for testing set

Sample	Actual(g/ml)	Prediction(g/ml)	RE(%)
1	0.0200	0.0200	0.0
2	0.0075	0.0073	2.6

$$\text{PRESS} = \sum_{i=1}^{m} (\hat{C}i - Ci)$$

represent the estimated $\hat{C}i$. In this equation, m is the total number of calibration samples concentration and Ci the reference concentration for the ith sample left out of the calibration during cross- validation. The optimal number of factor for lauramide at zero-derivative spectra was obtained 7.

The prediction error of lauramide was calculated as the relative error (RE). The results of RE (%)are shown in TABLE 1.

In other step, first order derivative spectra of standard solutions were studied. For obtaining the best PLS model the same procedure which discussed above were used. The wavelength region of 226-251 nm was selected as most appropriate spectral region. The predicted results and RE (%) for lauramide in this region are shown in TABLE 2. The optimal number of factor was obtained 4.

CONCLUSION

A UV-vis spectrophotometric method of analysis was successfully research and developed for determination of lauramide at the presence of related matrix in liquid detergent. Multivariate calibration model were built from the zero and first derivative spectral data matrices on the basis of PLS method. This technique consists in a simple, fast and inexpensive procedure which does not require separation step.

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