

## Determination of Coconut Fatty Acid Diethanol Amid in Liquid Detergent by Spectrophotometry and Multivariate Calibration

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This paper describes a procedure for the determination of coconut fatty acid diethanol amid in liquid detergent by UV-vis spectrophotometry. A partial least squares (PLS) regression was performed to resolve highly overlapping spectrophotometric signals obtained from coconut and other components in solution. Inputs of model were taken from absorbance spectral data which recorded in wavelength region 200-600 nm. In other steps, first and second order derivative spectra were considered as inputs of model. The proposed method has found to be precise and accuracy. The experimental calibration was designed with 16 standard samples. The concentrations for coconut were varied between 0.0025-0.0040 g/mL. The statistical parameters such as square correlation coefficient ( $R^2$ ), root mean squares error cross-validation (RMSECV) has been evaluated and number of factors and spectral have been optimized. The optimized model, obtained using PLS-1 was applied to the determination of compound in liquid detergent. The results for coconut in zero, first and second order derivative spectra were found to be satisfied. The error obtained in this method was  $\pm 2.50\%$ .

**Key Words:** Spectrophotometric, Coconut fatty acid diethanol amid, Detergent, Multivariate calibration, PLS.

### INTRODUCTION

The use of surfactants in industrial and domestic detergents has become wide spread. Coconut fatty acid is non-ionic surfactants that using in liquid detergents.

Techniques such as gas-liquid chromatography and liquid chromatography (HPLC)<sup>1,2</sup> have been used in surfactants analysis. For several years, titrimetric method has been used for determination coconut fatty acid in liquid detergent. However, these techniques are neither sufficiently selective nor are easily adapted to routine analyses. On the other hand,

spectrophotometric method do to its simplicity, is by far the most wide spread method of analysis and is also used in surfactants analysis.

Calibration is now days one of the most important fields of chemometrics, and spectral data are perhaps the most common type of data to which chemometrics techniques are applied. Owing to the development of new instrumentation, data sets in which object is described by several hundreds of variables can be easily obtained.

Partial least squares (PLS)<sup>3</sup> can easily treat these very large data matrices, extracting the relevant part of the information and producing reliable but very complex models. PLS was considered to be almost insensitive to noise and therefore, it was commonly selected that no feature selection at all was required<sup>4</sup>. In the last few years, it has been recognized that an efficient feature selection can be highly beneficial both to improve the predictive ability of the model and to greatly reduce its complexity<sup>5</sup>. In the last few years, several techniques devoted to feature selection in PLS models applied to spectral data have been presented. Three of these methods are iterative variable selection (IVS)<sup>6</sup>, uninformative variable elimination (UVE)<sup>7</sup> and iterative predictor weighting (IPW)<sup>8</sup>. The theory and application of PLS in spectrometry has been discussed by several workers<sup>9-15</sup>. In addition, several multicomponent determination of analytical substances based on the application of these methods to spectrophotometric data have been reported<sup>16-19</sup>. A particularly detailed study of multivariate calibration by PLS was carried out for the spectrophotometry determination of surfactants.

This paper deals with the direct determination of coconut fatty acid at the presence of the related matrix of liquid detergent, *e.g.*, linear alkyl benzene sulfonate (LABS), formalin and essence by using UV-Vis spectrometry and by applying partial least squares (PLS) method.

## EXPERIMENTAL

A double beam UV-Vis spectrometer BIO-TEK-Kontron (UV-KON922) equipped with quartz cell. The pH of solution was measured with a Suntext model (SP-701) pH meter. Data analysis was performed using software in MATLAB (release 6.5) for the statistical processing of data and the application of PLS method.

All reagents were of analytical grade and distilled water was used as solvent through the procedure.

To optimize and evaluate the prediction capability of PLS UV-Vis model, standard solutions of coconut fatty acid were made up in aqueous solution in various concentrations between 0.0025-0.0040 g/mL.

The compounds such as linear alkyl benzene sulfonate (LABS), formalin and essence were added to each standard solution to make the matrix

of these solutions similar to the matrix of liquid detergent. The concentration of these compounds, LABS, formalin and essence in all 16 standard solutions were kept constant and equal to 16, 0.1, and 0.1% (w/w), respectively. These standards were used to construct PLS models. The UV-vis spectra were obtained in the 200-600 nm spectra range, Then first and second order derivative has also recorded and used for determination of coconut. In Fig. 1 the absorbance spectra of each coconut and matrix are shown. Also zero, first and second order derivative absorbance spectra of mixed components are illustrated in Figs. 2-4, respectively. 16 standard solutions were prepared by dissolving appropriate a month of standard coconut fatty acid and the related matrixes in distilled water, so that the concentration of the coconut varies between 0.0025-0.0040 g/mL, but the concentrations of the related matrixes in all these standard solutions were kept constant.

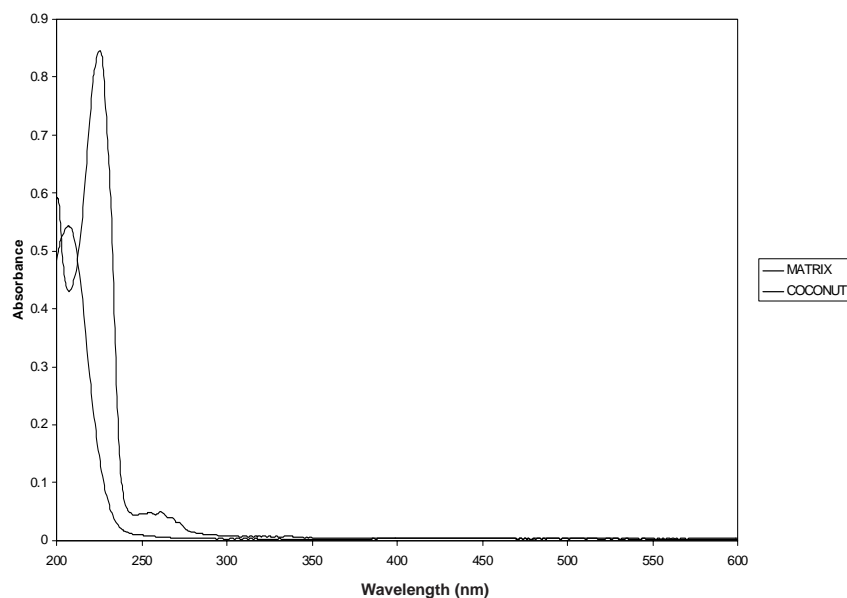


Fig. 1. Absorbance spectra of coconut fatty acid and matrix

## RESULTS AND DISCUSSION

Coconut fatty acid and LABS are the main components of the liquid detergents. They are used in the detergents as hand softening and cleaning agents<sup>20</sup>.

The spectra of coconut fatty acid in Fig. 1 shows one main absorbance band at 200-242 nm. In this figure, LABS as matrix show appreciable absorbance at this spectral region of 200-242 nm and overlap with coconut

band. On the other hand, Fig. 1 shows a direct relationship between the absorbance at 200-242 nm and the concentration of the standard solution. However if one can overcome the problem of overlapping the coconut fatty acid band with the band of matrix, the quantitative analysis of coconut fatty acid in liquid detergent can be carried out using these absorbance spectra.

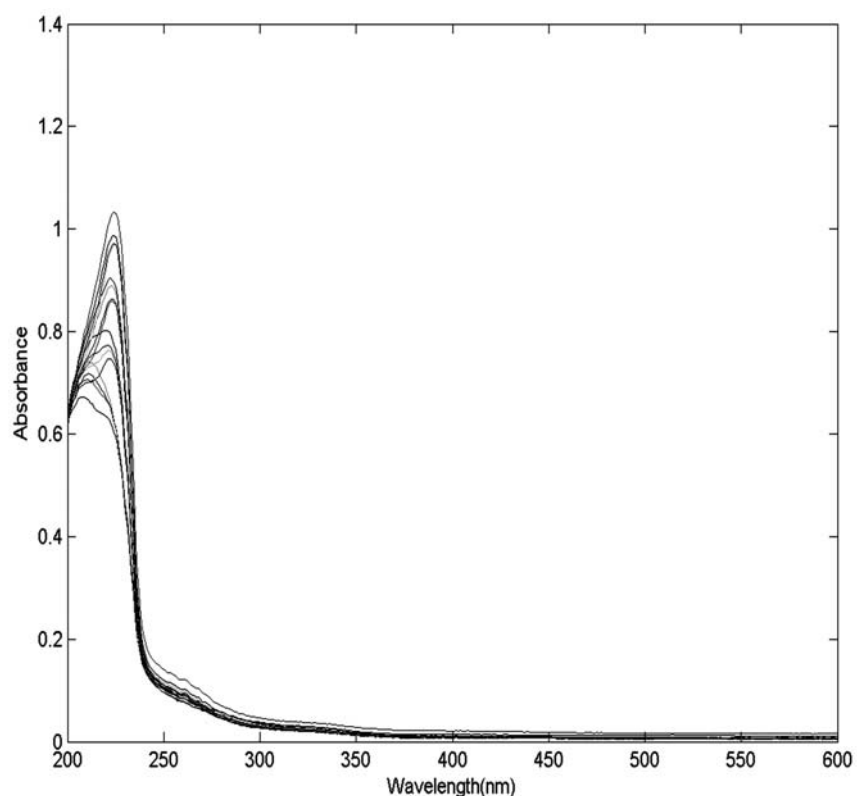


Fig. 2. UV-Vis zero order derivative spectra of mixed solution liquid detergent

Multivariate calibrations are useful tools to be used in spectral analysis in order to overcome the spectral overlapping and to improve the precision and the predictive ability of the UV-Vis spectrophotometry. With the aim of quantitative analysis of coconut fatty acid in liquid detergent, the PLS multivariate model was applied with the absorption spectra data. Spectral information was mean centered prior to PLS data treatment.

The unique way to verify that one calibration model works well is to take new samples and predict them by the model. In this work, spectral region between 200-600 nm containing 401 wavelength was selected for recording the absorbance spectra.

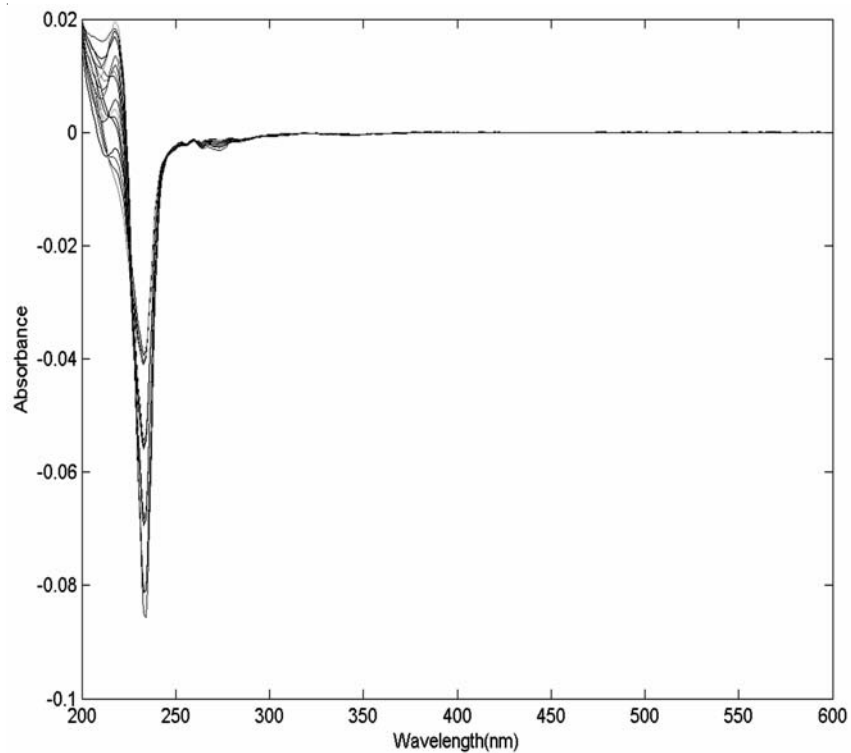


Fig. 3. UV-Vis first order derivative spectra of mixed solution liquid detergent

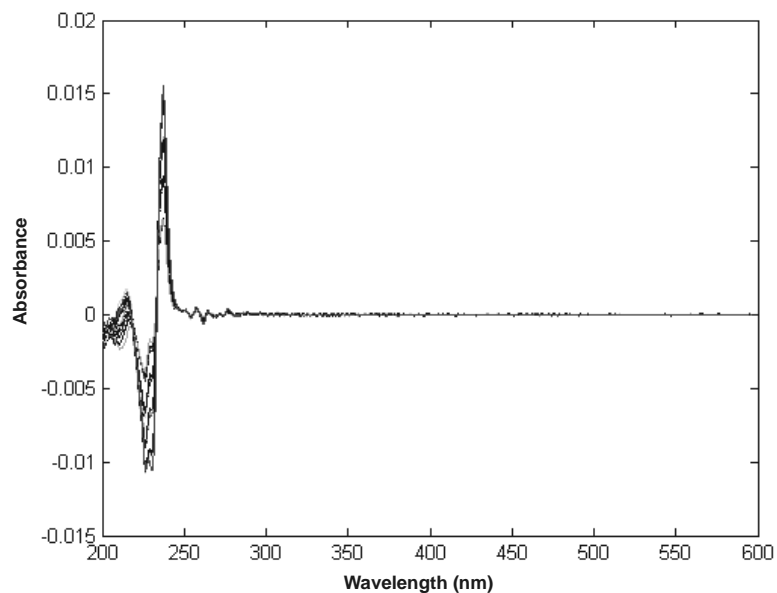


Fig. 4. UV-Vis second order derivative spectra of mixed solution liquid detergent

For constructing model, standard solutions randomly were separated in two sets with calibration set and testing set. The calibration model was also evaluated in each absorbance spectra data (zero, first and second order derivative) by varying the number of PLS factors and wavelength region in order to optimize the model.

To select the optimum number of factors in PLS algorithm, the cross-validation, leaving out one sample at a time, was used. The prediction error sum of squares (PRESS) was calculated each time a new factor was added giving rise to different PLS models one reasonable choice for the optimum number of factors would be that that number ( $h^*$ ) which yielded the minimum PRESS. However, the best calibration models in zero, first and second order derivative were obtained with (4, 3, 2) PLS factors, respectively.

With this optimum number of factors, Root mean squares error of cross-validation (RMSECV) was calculated for these standard sample using equation:

$$\text{RMSECV} = \sqrt{\frac{\sum_{i=1}^n (\hat{y}_i - y_i)^2}{n}}$$

in which,  $n$  is number of samples,  $\hat{y}_i$  the known concentration,  $y_i$  the predicted concentration. The values of (RMSECV) which is an estimate of the absolute error of prediction by cross-validation for each component in the calibration sample an square correlation of coefficient ( $R^2$ ), obtained when plots of actual versus predicted concentration were constructed for zero, first and second order derivative are summarized in Table-1.

TABLE-1  
STATISTICAL PARAMETERS OBTAINED FOR THE PLS-1 MODEL  
IN ZERO, FIRST AND SECOND ORDER DERIVATIVE SPECTRA (nm)

Component	RMSECV	$R^2$	Wavelength	Order derivative
Coconut	0.000122	0.9776	200-216	Zero
	8.69e-0.05	0.9897	200-214	First
	0.000141	0.9691	200-238	Second

Different spectral regions were evaluated by performing PLS calibration method and the spectral wavelength of 200-216 nm for coconut at zero order derivative was selected as most convenient. In order to ensure that this calibration model would provide accurate prediction, samples were proposed as validation set and their concentrations were predicted by using the calibration curve. Table-2 shows the prediction results for coconut fatty acid provided by PLS-1.

The same procedure also used for constructing PLS models based upon using first and second order derivative spectra for determination coconut

fatty acid in liquid detergent. In the first order derivative, several factors and spectral region for achieving the best model were evaluated. The spectral wavelength of region 200-214 nm for coconut were selected as the best wavelength region. Results are given Table-3.

TABLE-2  
RESULTS OBTAINED (g/mL) IN THE DETERMINATION OF  
COCONUT FATTY ACID BY APPLYING PLS-1 AT ZERO ORDER  
DERIVATIVE SPECTRA

Sample	Prediction	Actual
1	0.00410	0.00400
2	0.00280	0.00275
3	0.00350	0.00350

TABLE-3  
RESULTS OBTAINED (g/mL) IN THE DETERMINATION OF  
COCONUT FATTY ACID BY APPLYING PLS-1 AT FIRST ORDER  
DERIVATIVE SPECTRA

Sample	Prediction	Actual
1	0.00410	0.00400
2	0.00270	0.00275
3	0.00350	0.00350
4	0.00300	0.00300

For second order derivative the optimum error were achieved in region 200-238 nm wavelength for coconut fatty acid. Results are given in Table-4.

TABLE-4  
RESULTS OBTAINED (g/mL) IN THE DETERMINATION OF  
COCONUT FATTY ACID BY APPLYING PLS-1 AT SECOND ORDER  
DERIVATIVE SPECTRA

Sample	Prediction	Actual
1	0.00410	0.00400
2	0.00270	0.00275

### Conclusions

The application of multivariate calibration techniques has resulted improved applicability precision and accuracy in multicomponents spectral analysis. The results obtained show that determine coconut fatty acid in liquid detergent by UV-vis spectrophotometry and with a multivariate calibration technique can be an alternative and fast and inexpensive method. The results for coconut in zero, first and second order derivative spectra were satisfied. Also the error obtained for coconut fatty acid in this method was  $\pm 2.50\%$ .

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