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## Simultaneous Determination Of Linear Alkyl Benzene Sulfonate And Coconut Fatty Acid Diethanol Amide In Liquid Detergent By UV-Vis Spectroscopy And Partial Least Squares

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### ABSTRACT

The method aims, simultaneous determination of linear alkyl benzene sulfonate(LABS) and coconut fatty acid diethanol amid(Coconut) in liquid detergent by UV-vis spectroscopy. The absorbance spectra of these compounds are strongly overlapped which doesn't permit their direct determination. The proposed method is based on using partial least squares (PLS) model for resolving highly overlapping signals obtained from mixture of them. 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 provided upper precise and accuracy. The experimental calibration was designed with 16 standard samples the concentrations were varied between 0.0010-0.0025 g/ml for LABS and 0.0025-0.0040 g/ml for coconut. The statistical parameters such as square correlation of coefficient(R<sup>2</sup>), root mean square error cross-validation (RMSECV) have been evaluated and number of factors and spectral region have been optimized. The optimized model applied to the determination of these components liquid detergent. The results for LABS in zero and first order derivative spectra and coconut in zero, first and second order derivative spectra were satisfied. The error obtained for LABS and coconut in this method 2.86% and 2.50% respectively.

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### KEYWORDS

Partial least squares;  
Linear alkyl benzene sulfonate;  
Coconut fatty acid diethanol amide;  
Simultaneous;  
Spectroscopy.

## INTRODUCTION

The use of surfactants in industrial and domestic detergents has become wide spread. LABS and coconut are an anionic and nonionic surfactants, respectively, that they using in liquid detergents.

Techniques such as gas-liquid chromatography (GLC)<sup>[1]</sup>, thin layer chromatography(TLC) and high performance liquid chromatography(HPLC)<sup>[2]</sup> have been used in surfactants analysis.

For several years, titrimetric method has been used for determination of LABS and coconut in liquid detergents.

However, these techniques are neither sufficiently selective nor are they easily adapted to routine analysis. On the other hand, spectroscopy method due to it's simplicity, is by far the most wide spread method of analysis and is also used in surfactants analysis.

The simultaneous determination of LABS and coconut in one single stage using UV-vis spectroscopy is difficult when both species are present in medium, because of highly over lapping signals which prevent their calibration. Many processes propose solutions to the common problem of over lapping signals, of which the majority employ mathematical approaches<sup>[3-8]</sup>. A soft calibration method such as Partial Least Squares(PLS)<sup>[9-11]</sup> have made useful contributions to the resolution of over lapping signals. PLS is a proven method in the resolution of spectrophotometric<sup>[12]</sup> electrochemical signals<sup>[13-14]</sup>. Also it is particularly useful when we need to predict a set of dependent variables from a very large set of independent variables.

The goal of PLS regression is to predict Y from X and to describe their common structure. When Y is a vector and X is full rank, this goal could be accomplished using ordinary multiple regression. More data could be found in literature<sup>[15]</sup>.

The aim of our research is to simultaneously determine LABS and coconut in liquid detergent by UV-vis spectroscopy and by applying multivariate calibration methodology(PLS).

## EXPERIMENTAL

## Apparatus and software

A double beam UV-vis spectrometer BIO-TEK-KONTRON(UV-KON922) equipped with quartz cell. The pH of solution was measured with a SUNTEX 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.

## Reagent and procedure

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

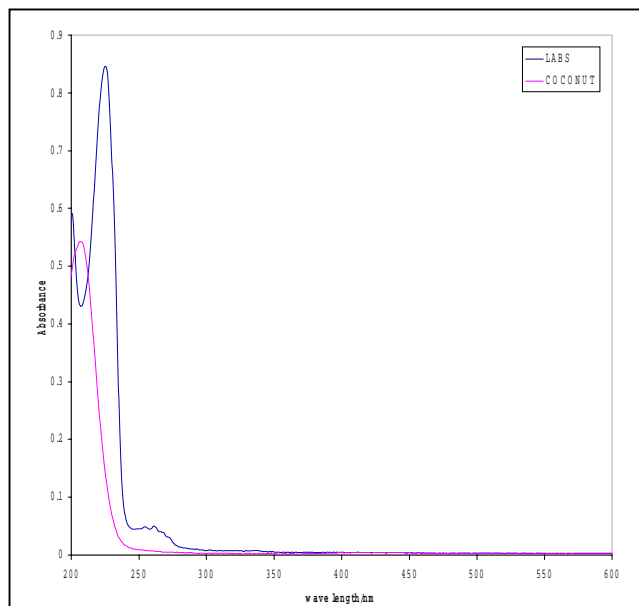
To optimize and evaluate the prediction capability of PLS-UV-vis model, standard solutions of LABS and coconut were made up in aqueous solution in various concentrations between 0.0010-0.0025 g/ml and 0.0025-0.0040 g/ml respectively, as shown in TABLE 1.

Also compounds such as 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 two compounds, formalin and essence in all 16 standard solutions were kept constant and equal to 0.1%(w/w).

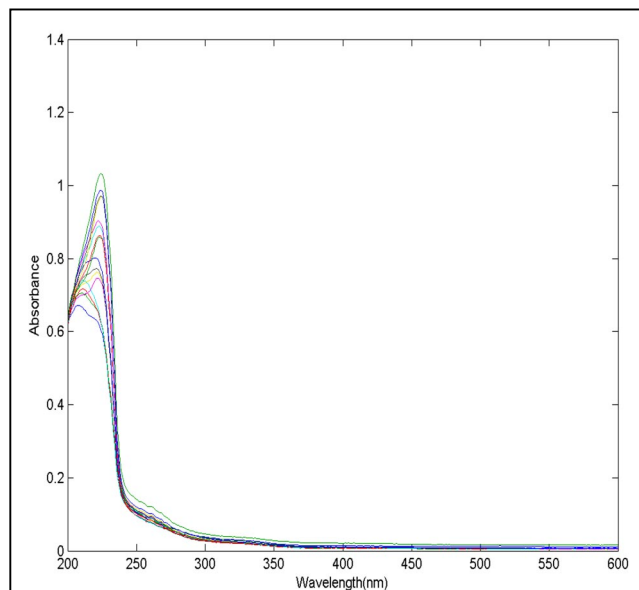
**TABLE 1: Concentrations of standard solutions of LABS and coconut**

Sample	LABS(g/ml)	Coconut(g/ml)
1	0.0010	0.0025
2	0.0010	0.0030
3	0.0010	0.0035
4	0.0010	0.0040
5	0.0015	0.0025
6	0.0015	0.0030
7	0.0015	0.0035
8	0.0015	0.0040
9	0.0020	0.0025
10	0.0020	0.0030
11	0.0020	0.0035
12	0.0020	0.0040
13	0.0025	0.0025
14	0.0025	0.0030
15	0.0025	0.0035
16	0.0025	0.0040

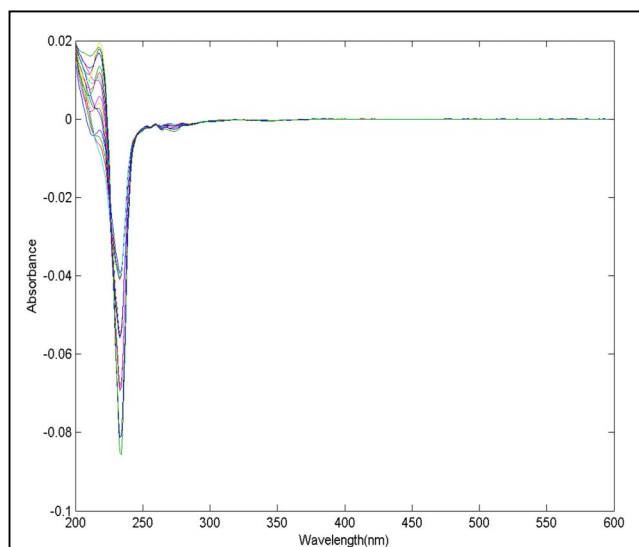
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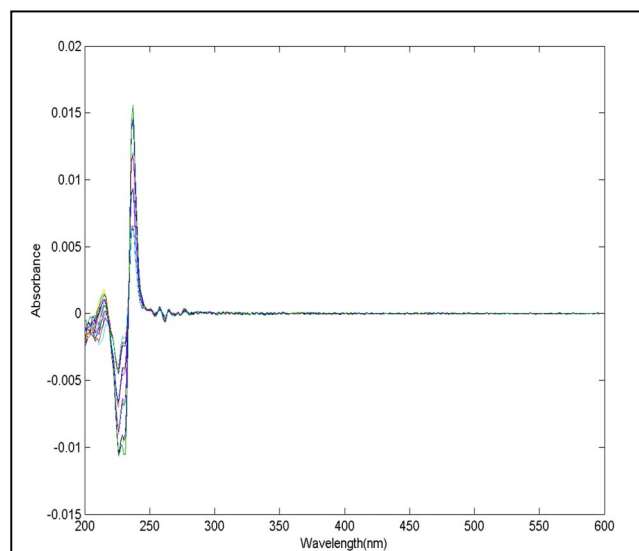
**Figure 1: The absorbance spectra of LABS and Coconut**



**Figure 2: UV-vis zero order derivative spectra of mixed solution liquid detergent**



**Figure 3: UV-vis first order derivative spectra of mixed solution liquid detergent**



**Figure 4: UV-vis second order derivative spectra of mixed solution liquid detergent**

these standards were used to construct PLS models. The UV-vis spectra were obtained in wavelength region 200-600 nm. Then first and second order derivative has also recorded and used for determination of LABS and coconut. In figure 1 the absorbance spectra of each LABS and Coconut are shown. Also zero, first and second order derivative absorbance spectra of mixed components are illustrated in figure(2-4) respectively.

## RESULTS AND DISCUSSION

LABS and coconut are the main components commonly used in liquid detergents they are used in detergents as cleaning(LABS) and hand softening (Coconut) agents.

The spectra of LABS in figure 1 shows one main absorbance band at 200-242 nm, also in this figure shows one main absorbance band at 200-242 nm for

coconut. However multivariate calibration are useful tools to be used in spectral analysis in order to overcome the spectral overlapping and to improve the precision and predictive ability. PLS multivariate model was applied to the absorption spectral data. The spectral region between 200-600 nm containing 401 wavelength values was selected for recording the absorbance spectra. For constructing model, standard solution randomly were separated in two sets with called calibration set and testing set. Also the calibration models was 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 method, leaving out one sample at a time, was used. Given the set of 16 calibration spectra, the PLS-1 calibration on 15 calibration spectra was performed and using this calibration, the concentration of the compounds in the sample which left out during calibration was predicted. This process was repeated until each calibration sample had been left out once. Found concentration compared with the known concentration of the reference sample and prediction error sum of squares(PRESS) was calculated. The PRESS was calculated as follows;

$$\text{PRESS} = \sum_{i=1}^m (\hat{c}_i - c_i)^2$$

In this equation, m is total

number of calibration samples,  $\hat{c}_i$  represents the estimated concentration and  $c_i$  the reference concentration for  $i$ th sample left out of the calibration during cross-validation.

The prediction error some of squares(PRESS) was calculated each time a new factor was added giving rise to different PLS models. Optimum number of factors is the case which yielded the minimum PRESS. The best calibration models for LABS and Coconut in zero, first and second order derivative were obtained with(3,3,1) and (4,3,2) PLS factors, respectively.

The values of root mean squares error of cross-validation(RMSECV) which is an estimate of the absolute error of prediction by cross-validation for each component in the calibration sample an square

**TABLE 2: Statistical parameters obtained for the PLS-1 model in zero, first and second order derivative spectra**

Component	RMSECV	R <sup>2</sup>	Wavelength (nm)	Order derivative
LABS	5.81e-0.05	0.9954	223-245	Zero
	4.72e-0.05	0.9969	252-264	First
	4.20e-0.05	0.9972	219-237	Second
Coconut	0.000122	0.9776	200-216	Zero
	8.69e-0.05	0.9897	200-214	First
	0.000141	0.9691	200-238	Second

**TABLE 3: Results obtained in the determination of LABS and Coconut by applying PLS-1 at zero order derivative spectra**

Sample	Prediction		Actual	
	LABS (g/ml)	Coconut (g/ml)	LABS (g/ml)	Coconut (g/ml)
1	0.00180	0.00410	0.00175	0.00400
2	0.00200	0.00280	0.00200	0.00275
3	0.00150	0.00350	0.00150	0.00350

correlation of coefficient(R<sup>2</sup>), obtained when plots of actual versus predicted concentration were constructed, for zero, first and second order derivative are summarized in TABLE 2.

Different spectral regions were evaluated by performing PLS calibration method and the spectral wavelength of 223-245 nm for LABS and 200-216 nm for Coconut at zero order derivative were selected as most convenient. Also 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 a calibration curve. TABLE 3 shows the prediction results for LABS and Coconut provided by PLS-1.

Also the same procedure used for constructing PLS models based upon using first and second order derivative spectra for determination LABS and Coconut 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 252-264 nm for LABS and region 200-214 nm for Coconut were selected as the best wavelength region. Results are given TABLE 4.

For second order derivative the optimum error were achieved in region 219-237 nm wavelength for

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**TABLE 4: Results obtained in the determination of LABS and coconut by applying PLS-1 at first order derivative spectra**

Sample	Prediction		Actual	
	LABS (g/ml)	Coconut (g/ml)	LABS (g/ml)	Coconut (g/ml)
1	0.00180	0.00410	0.00175	0.00400
2	0.00200	0.00270	0.00200	0.00275
3	0.00150	0.00350	0.00150	0.00350
4	0.00200	0.00300	0.00200	0.00300

**TABLE 5: Results obtained in the determination of LABS and coconut by applying PLS-1 at second order derivative spectra**

Sample	Prediction		Actual	
	LABS (g/ml)	Coconut (g/ml)	LABS (g/ml)	Coconut (g/ml)
1	0.00190	0.00410	0.00175	0.00400
2	0.00200	0.00270	0.00200	0.00275

LABS and 200-238 nm for Coconut. Results are given in TABLE 5.

### CONCLUSIONS

The application of multivariate calibration techniques has resulted improved applicability precision and accuracy in multicomponents spectral analysis. The results obtained show that simultaneously determine LABS and coconut in liquid detergent by UV-vis spectroscopy and with a multivariate calibration technique can be an alternative and fast and inexpensive method. The results for LABS in zero and first order derivative spectra and coconut in zero, first and second order derivative spectra were satisfied. Also the error obtained for LABS and Coconut in this method were 2.86% and 2.50% respectively.

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