



Simultaneous spectrophotometric determination of binary mixtures of surfactants using continuous wavelet transformation

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ABSTRACT

This work presents a simple, rapid, and novel method for simultaneous determination of binary mixtures of some surfactants using continuous wavelet transformation. The method is based on the difference in the effect of surfactants Cetyltrimethylammoniumbromide (CTAB), dodecyl trimethylammonium bromide (DTAB), cetylpyridinium bromide (CPB) and TritonX-100 (TX-100) on the absorption spectra of complex of Beryllium with Chrome Azurol S (CAS) at pH 5.4. Binary mixtures of CTAB–DTAB, DTAB–CPB and CTAB–TX-100 were analyzed without prior separation steps. Different mother wavelets from the family of continuous wavelet transforms were selected and applied under the optimal conditions for simultaneous determinations. The proposed methods, under the working conditions, were successfully applied to simultaneous determination of surfactants in hair conditioner and mouthwash samples.

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1. Introduction

Cationic surfactants (CS) composed of quaternary ammonium groups. They are extensively used as preservatives or antiseptic agents in industrial and commercial products. Due to their ability to stabilize emulsions and their antibacterial properties, surfactants used widely in cosmetics, hygiene products and some drugs are ingested by the human body. CS is toxic even at low concentrations [1] and also is environmental pollutants [2]. Therefore, detection of trace CS in various environmental samples is of great interest and increasing importance in environment protection and many other related fields.

Mixtures of surfactants are found in numerous applications, ranging from industrial or technological systems to domestic products. Different surfactants are intentionally mixed to exploit synergistic behavior in mixed systems or to improve performance quality of a single formulation. The properties of the systems can be tuned simply by varying the composition, which is an attractive alternative to the synthesis of new materials [3–6].

There is an interest to develop methods for simultaneous determination of surfactants in different samples. Several methods have been reported for the simultaneous determination of surfactants. Fourier transform infrared–attenuated total reflectance (FTIR–ATR) was proposed for the simultaneous determination of sodium lauryl ether sulfate (SLES), cocoamidopropyl betaine

(CAPB) and alkylpolyglucoside (APG) [7]. Reversed phase HPLC [8], capillary electrophoresis with indirect UV detection [9], HPLC with evaporative light scattering detection [10], liquid chromatography with mass spectrometry and tandem mass spectrometry [11], liquid chromatography with fluorescence detection [12] and mixed aggregate-based methodology [13] were used for the determination of mixtures of surfactants. These methods are either time-consuming and/or require complicated and expensive instruments.

A wavelet transform (WT) is the representation of a function by wavelets. The wavelets are scaled and translated copies of a finite-length or fast-decaying oscillating waveform. WT to overcome the problem of noisy and incomplete data has been successfully put on a sound statistical basis by Donoho and Johnstone [14,15]. WT was applied to denoising of data [16,17], compressing of signals [18,19], simultaneous determination of chemical species in binary [20–32], and ternary [33–35,] mixtures and determination of enantiomeric ratio [19].

In this study, our aim was to develop a spectrophotometric method for the analysis of binary mixtures of different surfactants in synthetic and formulated products, with no separation steps. The method is based on the difference in the effects of surfactants on the spectrum of the complexes of Chrome Azurol S with Be^{2+} cation. CWT was applied for simultaneous determination of different binary mixtures of CTAB, DTAB, CPB and TX-100. The applicability of different continuous mother wavelet was tested on different synthetic data. The method was validated by simultaneous determination of mixtures of surfactants in some synthetic samples and hair conditioner and mouthwash samples.

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Table 1
Statistical results of calibration graphs obtained in zero cross points by proposed method.

Mother wavelet	Surfactant	λ (nm)	Linear range	Regression equation	r	LOD (mol L ⁻¹)	LOQ (mol L ⁻¹)
mexh	TX-100	548	8×10^{-5} – 4×10^{-4}	$Y = -27734 + 2.8668x$	0.9993	2.90×10^{-7}	9.67×10^{-7}
mexh	CTAB	560	2×10^{-7} – 2×10^{-5}	$Y = 70271 + 0.0975x$	0.9996	6.29×10^{-8}	2.09×10^{-7}
morl	TX-100	550	8×10^{-5} – 4×10^{-4}	$Y = -22553x + 2.464$	0.9989	3.46×10^{-7}	1.15×10^{-6}
morl	CTAB	560	2×10^{-7} – 2×10^{-5}	$Y = 55945x + 0.6969$	0.9995	7.06×10^{-8}	2.35×10^{-7}
meyr	TX-100	627	8×10^{-5} – 4×10^{-4}	$Y = -21961 + 2.3353x$	0.9991	4.10×10^{-7}	1.36×10^{-6}
meyr	CTAB	637	2×10^{-7} – 2×10^{-5}	$Y = 66584x + 0.2566$	0.9992	7.78×10^{-8}	2.59×10^{-7}
sym4	TX-100	540	8×10^{-5} – 4×10^{-4}	$Y = -30901x + 2.725$	0.9992	1.74×10^{-7}	5.80×10^{-7}
sym4	CTAB	553	2×10^{-7} – 2×10^{-5}	$Y = 81668x + 0.6166$	0.9989	7.96×10^{-8}	2.65×10^{-7}
coif1	TX-100	544	8×10^{-5} – 4×10^{-4}	$Y = -27281x + 2.484$	0.9993	3.29×10^{-7}	1.08×10^{-6}
coif1	CTAB	556	2×10^{-7} – 2×10^{-5}	$Y = 61451x - 0.4032$	0.9996	6.20×10^{-8}	2.06×10^{-7}
db6	DTAB	594	4×10^{-6} – 6×10^{-5}	$Y = -56625x - 0.6377$	0.9992	1.20×10^{-7}	4.01×10^{-7}
db6	CPB	584	2×10^{-7} – 3×10^{-5}	$Y = 76443x + 0.18$	0.9993	6.17×10^{-8}	2.05×10^{-7}
sym8	DTAB	551	6×10^{-6} – 6×10^{-5}	$Y = 61043x + 0.0278$	0.9990	1.51×10^{-7}	5.04×10^{-7}
sym8	CPB	541	6×10^{-7} – 3×10^{-5}	$Y = -75759 + 0.241x$	0.9993	1.12×10^{-7}	3.74×10^{-7}
morl	DTAB	554	4×10^{-6} – 6×10^{-5}	$Y = 46530x + 0.3712$	0.9990	2.21×10^{-7}	7.36×10^{-7}
morl	CPB	545	6×10^{-7} – 2×10^{-5}	$Y = -65323 + 0.3616x$	0.9996	1.45×10^{-7}	4.85×10^{-7}
meyr	DTAB	627	6×10^{-6} – 6×10^{-5}	$Y = 55892x + 0.485$	0.9992	1.53×10^{-7}	5.10×10^{-7}
meyr	CPB	618	6×10^{-7} – 3×10^{-5}	$Y = 72569x + 0.2856$	0.9993	1.35×10^{-7}	4.51×10^{-7}
mexh	DTAB	548	6×10^{-6} – 6×10^{-5}	$Y = 48360x + 0.1929$	0.9996	7.29×10^{-8}	2.43×10^{-7}
mexh	CTAB	540	2×10^{-7} – 2×10^{-5}	$Y = -39009x + 0.703$	0.9996	1.52×10^{-7}	5.09×10^{-7}
db4	DTAB	545	4×10^{-6} – 6×10^{-5}	$Y = 61133x - 0.2815$	0.9993	1.40×10^{-8}	4.67×10^{-8}
db4	CTAB	532	2×10^{-7} – 2×10^{-5}	$Y = -48662x - 1.8191$	0.9995	3.45×10^{-8}	1.15×10^{-7}
db6	DTAB	510	4×10^{-6} – 6×10^{-5}	$Y = 44762x - 0.4565$	0.9998	2.43×10^{-7}	8.11×10^{-7}
db6	CTAB	498	2×10^{-7} – 2×10^{-5}	$Y = -49410x - 2.5073$	0.9999	3.24×10^{-8}	1.08×10^{-7}
sym4	DTAB	541	4×10^{-6} – 6×10^{-5}	$Y = 52615x - 0.156$	0.9995	1.10×10^{-7}	3.67×10^{-7}
sym4	CTAB	532	2×10^{-7} – 2×10^{-5}	$Y = -37453x - 0.4482$	0.9997	5.80×10^{-8}	1.93×10^{-7}

2. Experimental

2.1. Standard solutions

All the solutions were prepared using reagent grade substances. A 1.0000 g L⁻¹ stock solution of Be²⁺ was prepared in 0.10 mol L⁻¹ sulfuric acid using BeSO₄·4H₂O (Merck). Working solutions were prepared by appropriate dilution of the stock solution every day. Stock CTAB, (1.0 × 10⁻³ mol L⁻¹, Merck) DTAB, (1.0 × 10⁻³ mol L⁻¹, Merck), CPB (1.0 × 10⁻³ mol L⁻¹, Fluka) and TX-100 (1.0 × 10⁻¹ mol L⁻¹, Merck) solutions were prepared in distilled water. Acetate buffer solution of pH 5.4 was prepared by mixing 85.5 mL of 0.2 mol L⁻¹ sodium acetate and 14.5 mL of 0.2 mol L⁻¹ acetic acid [36].

2.2. Apparatus

Spectra were recorded and stored using a single beam UV-mini-WPA spectrophotometer equipped with a PC using a 1-cm path length glass cell. A model 713 Metrohm pH meter was used for measurement of the pH of the solutions. Calculations were performed using Toolbox for MATLAB 7.1 and EXCEL (XP windows 2003 operating system).

2.3. General procedure

An aliquot of the solution containing appropriate amounts of surfactants (see Table 1), 2.0 mL of 1.0 × 10⁻³ mol L⁻¹ CAS solution, 1.0 mL of 4.0 mg L⁻¹ Be²⁺ solution and 2.0 mL of pH 5.4 acetate buffer solution was transferred into a 10.0-mL volumetric flask. The solution was then diluted to the mark with water. A portion of the solution was transferred into a 1.0 cm glass cell to record the absorbance spectra in the wavelength range 450–700 nm against a blank solution that was also prepared in the same way except that distilled water was added instead of surfactants.

2.4. Preparation of real samples

Sample preparation was performed as described previously [37]. Accurately weighed amounts of shampoo were dissolved in 100 mL

doubly distilled water. All sample solutions were filtered with Whatman filter paper no. 42. The amount of surfactants was then determined after proper dilution by the proposed method.

3. Results and discussion

Triphenylmethane dye Chrome Azurol S (3''-Sulfo-2'',6''-dichloro-3,3'-dimethyl-4-hydroxyfuchsonone-5,5'-dicarboxylic acid, trisodium salt; (CAS)) reacts with beryllium to form the anionic complex Be(CAS)₂ [39,40]. CAS has been widely exploited in the presence of various cationic or non-ionic surfactants or surfactant mixtures. Species formed in such systems have high molar absorptivities and considerable color contrast [41]. It was observed that there is a small difference between the absorption spectra of the ternary complexes of Be²⁺ with CAS and different surfactants. The absorption spectra of complexes of different surfactants with CAS and Be²⁺ are shown in Fig. 1. As Fig. 1 shows the spectra of the complexes overlap with each other, and therefore each surfactant interfere in the spectrophotometric determination of the others based on their ternary complexes with Be–CAS complex. Classical methods cannot resolve this system. But these systems can be suitable for simultaneous determination of surfactants using continuous wavelet transforms.

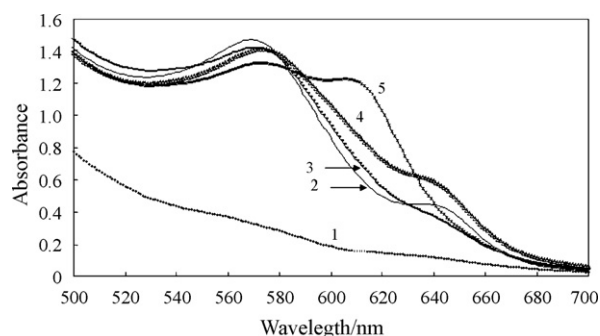


Fig. 1. Absorption spectra of ternary complexes of (1) Be–CAS with (2) DTAB, (3) CTAB, (4) CPB and (5) TX-100.

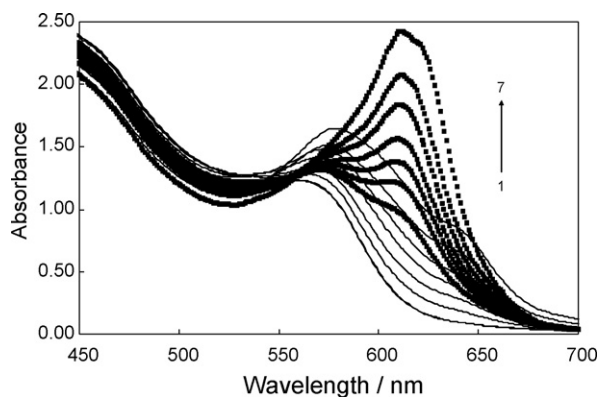


Fig. 2. Absorption spectra of Be–CAS in the presence of (1) 4.0×10^{-7} , (2) 5.0×10^{-6} , (3) 1.0×10^{-5} , (4) 1.5×10^{-5} , (5) 3.0×10^{-5} , (6) 5.0×10^{-5} mol L⁻¹ CTAB (—) and in the concentration of (1) 2.0×10^{-5} , (2) 1.0×10^{-4} (3) 1.2×10^{-4} (4) 1.5×10^{-4} and (5) 1.6×10^{-4} (6) 1.8×10^{-4} and 3.0×10^{-4} mol L⁻¹ TX-100 (■).

3.1. Optimization of the experimental conditions

The overall processes are influenced by pH of the solutions, concentration of beryllium and CAS which affects the completeness of complex formation reactions. In order to find the optimum pH, the effect of pH in the range 3.6–5.6 and different buffer solutions on the complex formation reactions were investigated. The results showed that for Be–CAS–CTAB system the difference between the absorbance of sample and blank solutions (ΔA) increased by increasing pH in the range 3.6–5.4 and decrease at higher pHs. Therefore, pH 5.4 was used for further analysis. For simultaneous determination of surfactants, the effect of other different parameters were investigated and the optimum working conditions affecting the development of the colored complexes were obtained as follows: CAS concentration is 2.0×10^{-4} mol L⁻¹ and beryllium concentration is 0.40 mg L⁻¹ at pH is 5.4.

All the experiments were performed at room temperature.

3.2. Spectral characteristics and selection of appropriate mother wavelets

Fig. 1 shows the spectra of standard solutions of complexes of Be–CAS and different surfactants (CTAB, DTAB, CPB and TX-100) at optimum conditions. Strong overlap presented in the spectral region 450–700 nm make mutual interferences of considered surfactants in the simultaneous determination of them. Hence, simultaneous determination of surfactants is not possible by using the classical analytical methodologies. Application of continuous wavelet transform can provide successful determination of investigated surfactants without prior separation steps. Absorption spectra for Be–CAS complex in the linear concentration range of 2.0×10^{-7} – 2×10^{-5} mol L⁻¹, 6.0×10^{-7} – 6.0×10^{-5} mol L⁻¹, 2.0×10^{-7} – 5.0×10^{-5} mol L⁻¹ and 8.0×10^{-5} – 3×10^{-4} mol L⁻¹ for CTAB, DTAB, CPB and TX-100, respectively, in the spectral region 450–700 nm, were recorded against blank solution. Various wavelet families were tested to find the optimal signal processing for obtaining desirable calibration graphs and reliable determination of the investigated elements. Mexican hat (mexh), meyer, coiflet, morlet (morl), symlets (sym) and daubechies (db) were selected for further analysis as mother wavelets for transformation of absorption spectra. The value of scaling factor for determination of CTAB and TX-100 in their binary mixture was optimized for different mother wavelets. Fig. 2 shows the absorption spectra for the calibration sets of CTAB and TX-100. Fig. 3a shows the graphs of CWT for these calibration sets of CTAB and TX-100 that were obtained by mexh ($a=50$) on the absorption spectra at the same

Table 2

Mean recoveries and relative standard deviations for the simultaneous determination of CTAB and TX-100 in various synthetic mixtures by the proposed method.

Mother wavelet	Surfactant ^a	
	TX-100	CTAB
mexh-50	103.56 ± 2.21	101.19 ± 2.96
morl-160	103.46 ± 2.06	97.96 ± 2.73
meyr-160	103.47 ± 2.06	103.10 ± 5.33
sym ₄ -140	102.55 ± 1.21	101.84 ± 4.41
coif1-170	103.08 ± 2.64	100.14 ± 5.60

^a Average of five determinations ± standard deviation.

wavelength range. In this study we used the zero cross points for simultaneous determination of different surfactants. So a calibration graph of standard solutions of each surfactant was obtained with CWT signals versus standards concentrations. The created calibration curves were used for prediction of unknown binary mixtures of surfactants. As Table 1 shows, though as predicted the obtained calibration equations for different mother wavelet are different, but all of them are suitable and there is not significant preferable each other over another. Therefore, in analyzing the real samples each mother wavelet can be used. Calibration graphs for CTAB and TX-100 with mexh ($a=50$) were obtained by measuring the CWT signals at the 560 nm and 548 nm correspond to zero crosses of TX-100 and CTAB, respectively. The calibration graphs and the statistical results are shown in Table 1. For the prediction of concentration of CTAB and TX-100 in synthetic binary mixtures the same procedure was used and concentrations of CTAB and TX-100 in binary mixtures were estimated by CWT-signal calibration equations at zero crosses. The estimated recoveries of CTAB and TX-100 which obtained by proposed mother wavelets are given in Table 2. Also transformation of calibration spectra of CTAB and TX-100 were performed by morl ($a=160$), meyer ($a=160$), sym₄ ($a=140$) and coif1 ($a=170$) (Fig. 3b, c, d and e, respectively). Transformation of their binary mixtures were also performed by morl ($a=160$), meyer ($a=160$), sym₄ ($a=140$) and coif1 ($a=170$). The optimum scaling value was found as 160, 160, 140 and 170 for morl, meyer, sym₄ and coif1, respectively. The amplitude of CWT signals at wavelengths corresponds to zero crosses of TX-100, were created the calibration graphs for CTAB at 560 nm, 637 nm, 553 nm and 556 nm for morl, meyer, sym₄ and coif, respectively. Also the calibration graphs for TX-100 at 550 nm, 627 nm, 540 nm and 544 nm for morl, meyer, sym₄ and coif1, respectively, were built by measuring the amplitude of CWT signals at zero crosses of CTAB. Linear regression analysis and the statistical results are shown in Table 1. By using the calibration equations, CTAB and TX-100 were determined in binary synthetic mixtures with proposed mother wavelets. Mean recovery results are presented in Table 2.

In the similar way, simultaneous determination of DTAB and CTAB in binary mixtures by transformation of spectra of standard solutions was performed. Transformation of their spectra were performed by mexh ($a=50$), db4 ($a=110$), db6 ($a=120$) and sym₄ ($a=140$). Amplitude of CWT-mexh graphs yields straight lines at 540 nm for CTAB (correspond to zero cross of DTAB) and 548 nm for DTAB (corresponds to zero cross of CTAB). Also straight lines from amplitudes of CWT-db4 graphs at 532 and 545 nm, of CWT-db6 graphs at 498 and 510 nm and of CWT-sym₄ at 532 and 541 nm were obtained for determination of CTAB and DTAB in their binary mixtures, respectively. Its regression coefficients and statistical results at zero crosses are shown in Table 1. Mean recoveries and the relative standard deviations were calculated and the results are given in Table 3.

Quantitative determination of CPB and DTAB in binary mixtures was also performed in the same way. Transformation of their spec-

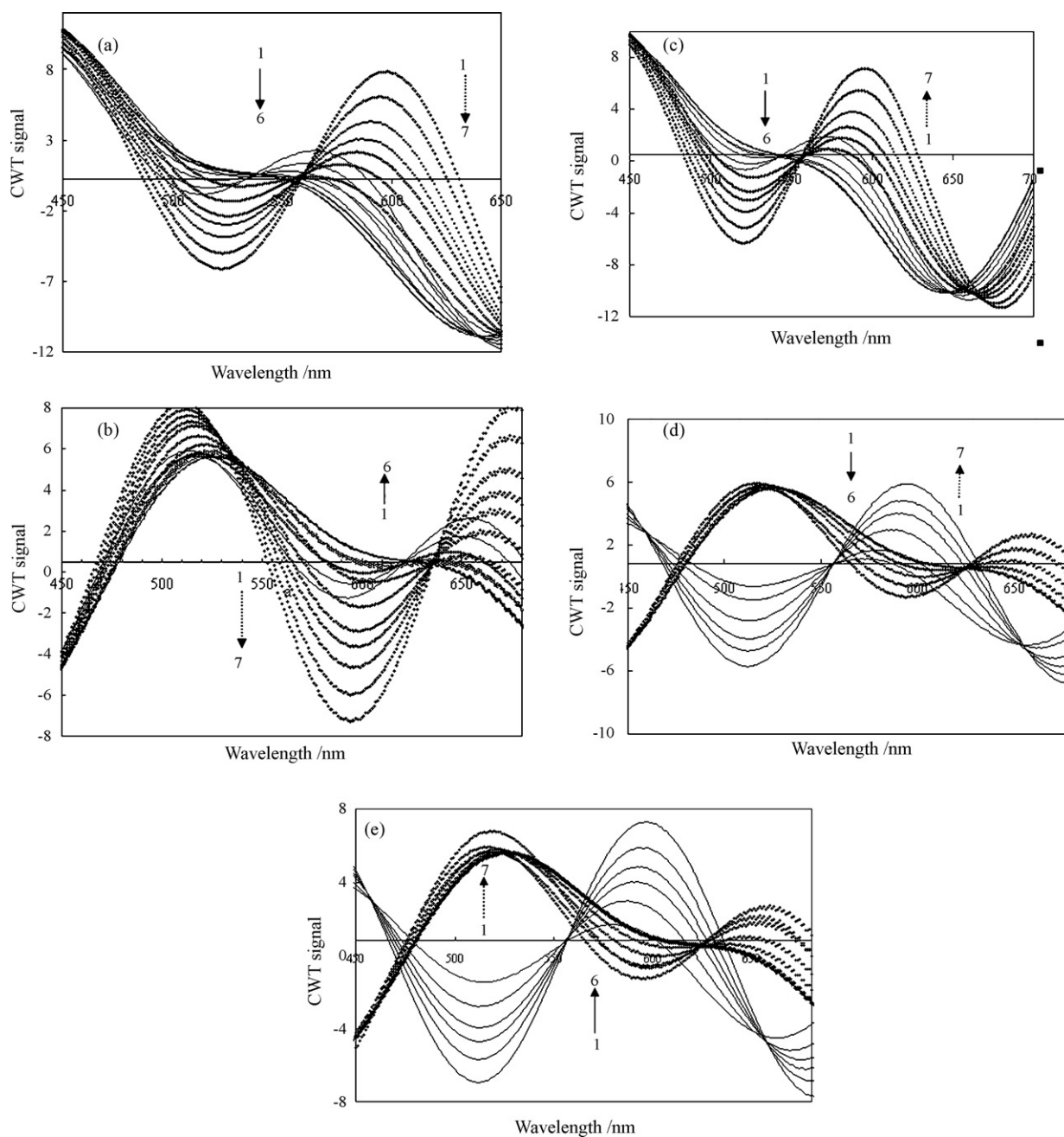


Fig. 3. CWT signals of Be-CAS in the presence of (1) 4.0×10^{-7} , (2) 5.0×10^{-6} , (3) 1.0×10^{-5} , (4) 1.5×10^{-5} , (5) 3.0×10^{-5} ; (6) 5.0×10^{-5} mol L⁻¹ CTAB (—) and in the concentration of (1) 2.0×10^{-5} , (2) 1.0×10^{-4} (3) 1.2×10^{-4} (4) 1.5×10^{-4} and (5) 1.6×10^{-4} (6) 1.8×10^{-4} and 3.0×10^{-4} mol L⁻¹ TX-100 (■) by (a) mexh, (b) meyr, (c) coif1, (d) morl and (e) sym4.

tra were performed by Db6 ($a = 120$), Sym8 ($a = 160$), morl ($a = 160$) and meyr ($a = 150$). Amplitude of CWT-mexh graphs yields straight lines at 540 nm for CTAB (correspond to zero cross of DTAB) and 548 nm for DTAB (corresponds to zero cross of CTAB).

Table 3

Mean recoveries and relative standard deviation for the simultaneous determination of DTAB and CTAB in various synthetic mixtures by the proposed method.

Mother wavelet	Surfactant ^a	
	DTAB	CTAB
mexh-50	102.20 ± 1.62	100.03 ± 3.35
sym4-140	102.45 ± 2.12	100.37 ± 2.42
db4-110	101.61 ± 1.91	99.59 ± 2.86
db6-120	101.28 ± 2.66	101.18 ± 1.93

^a Average of five determinations ± standard deviation.

Straight lines from amplitudes of CWT-db6 graphs at 584 nm and 594 nm, of CWT-sym6 graphs at 541 nm and 551 nm, of CWT-morl at 545 nm and 554 nm and of CWT-meyr graphs at 618 nm and 627 nm were obtained for determination of CPB and DTAB in

Table 4

Mean recoveries and relative standard deviation for the simultaneous determination of DTAB and CPB in various synthetic mixtures by the proposed method.

Mother wavelet	Surfactant ^a	
	DTAB	CPB
db6-120	101.14 ± 2.27	99.84 ± 2.48
sym8-160	101.63 ± 2.60	100.23 ± 2.36
morl-160	101.35 ± 2.04	99.94 ± 1.76
meyr-150	100.83 ± 1.85	99.43 ± 1.76

^a Average of five determinations ± standard deviation.

Table 5
Simultaneous determination of binary mixtures of surfactants in hair conditioner and mouthwash samples by proposed and reference methods.

Sample	Surfactant		Mother wavelet	Recovery for added surfactant ^a	Amount of present surfactant ^a ($\mu\text{g g}^{-1}$)		$t_{\text{calculated}}$	$t_{\text{theoretical}}$
	Added	Present			Proposed method	Reference method		
Hair conditioner	CTAB	DTAB	mexh	101.85 ± 1.53	653.87 ± 2.67	650.72 ± 1.71 ^a	2.22	2.78
			db6	101.44 ± 2.99	649.33 ± 1.49		1.40	
			db4	101.45 ± 2.18	652.98 ± 2.85		2.23	
			sym8	101.85 ± 1.53	652.98 ± 2.85		2.62	
Mouthwash	CPB	DTAB	db6	103.63 ± 2.0	728.05 ± 2.82	738.32 ± 3.78 ^b	3.77	4.30
			sym8	100.65 ± 1.44	740.70 ± 2.14		0.94	
			morl	100.44 ± 1.73	733.71 ± 2.07		1.85	
			meyr	101.53 ± 1.76	734.44 ± 4.32		0.28	

^a Average of five determinations ± standard deviation.

^b Average of four determinations ± standard deviation.

their binary mixtures, respectively. Its regression coefficients and statistical results at zero crosses are shown in Table 1. Mean recoveries and the relative standard deviations were calculated and their results were given in Table 4.

Mean recoveries for the simultaneous determination of CTAB–TX-100, DTAB–CTAB and DTAB–CPB show that there were no significant differences for prediction of concentrations with different mother wavelets. But mexh, sym4 and sym8 are the best mother wavelets for simultaneous determination of CTAB–TX-100, DTAB–CTAB and DTAB–CPB in binary mixtures, respectively.

The limit of detection (LOD) and limit of quantitation (LOQ) [42] of the method for the determination of surfactants were calculated and are given in Table 1. The obtained results show that proposed method based on using different mother wavelets is suitable for the simultaneous determination of binary mixtures of different surfactant types.

3.3. Applications

Results for the analysis of synthetic mixtures by the proposed methods (Table 2) were satisfactory for the simultaneous determination of binary mixtures of CTAB–TX-100, CTAB–DTAB and CPB–DTAB. To demonstrate the applicability of the proposed method to real samples, it was applied to the simultaneous determination of surfactants in hair conditioner (Latife, Iran) and mouthwash samples (Oral B, UK). As the hair conditioner and mouthwash samples contain only one of the surfactants, a known amount of another proper surfactant (Table 5) was added. The proposed method with different mother wavelets was used. The results are summarized in Table 5. The amount of surfactants was determined by reference methods [38] too. The calculated t -values are less than the tabulated values with 4 degrees of freedom at 95% confidence interval. This indicates the proposed methods to be as precise and accurate as the standard methods.

4. Conclusion

A CWT-zero crossing technique was developed to analyze the signal of different surfactants in their binary mixtures. In spite of strong spectral overlapping of the absorption spectra of the ternary complexes of Be–CAS with CTAB, CPB, DTAB and TX-100; a simple, very sensitive, precise, and easy to understand and method for the simultaneous determination of CTAB–TX-100, CTAB–DTAB and DTAB–CPB binary mixtures of surfactants in the synthetic and commercial samples has been proposed. The method does not require a pre-treatment process. Good results were obtained by utilizing CWT show that proposed method is appropriate for the quality control and the routine analysis in the mixtures and commercial products.

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