

Simple two spectrophotometric methods for estimation of Cephalexin in pure and pharmaceutical dosage form

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Abstract

This study describes two extraction-free and direct spectrophotometric methods for the determination of Cephalexin in a pure and pharmaceutical dosage form. In this study, a zero-order method has exhibited maximum absorption max at 263 nm, while area under curve method has calculated in the range 260-266nm. The Linearity of both methods in the range (10- 60 nm), the limit of detection (LOD) is 0.808 for Zero-order method and 0.781 for the area under curve method. These methods are successfully applied to determination of Cephalexin in a pharmaceutical dosage form.

Introduction:

Cephalexin is a kind of antibiotics has been used to infections of skin and organs by kill bacteria in both human and animals [1, 2]. Many analytical methods in the literature have been suggested for estimation of cephalexin in serum, meat and pharmaceutical dosage form. Most of these methods are depended on temperature control, lead to increased environmental pollution, took a long time for operation and expensive, these methods include HPLC-mass in plasma of swine [3], RP-UFLC in pharmaceutical forms [4], electrochemical immunosensor in meat samples [5], fluorescence probes [6], used three methods spectrophotometric, voltammetric and titration by means of potassium carbonate as analytical reagent[7], flame atomic emission[8], First and second derivative spectrophotometric [9], electrochemical sensors in serum [10] and RP-HPLC [11].

In this study, developed a fast, simple, inexpensive and free of environmental effects methods included spectrophotometric methods namely: zero-order and area under curve used for determination of Cephalexin in pure and marketed capsule formulations.

Experimental

1. Instruments

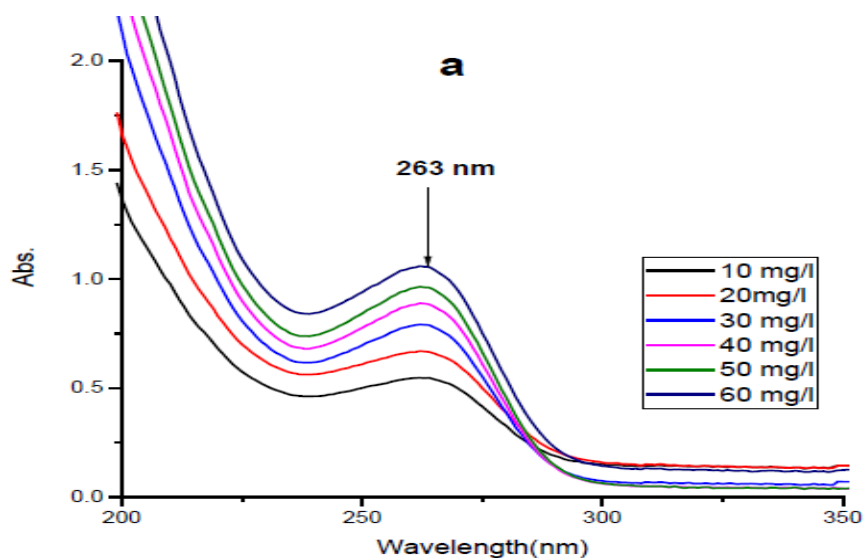
Shimadzu spectrophotometer model (1800) was used for the spectrum and absorbance measurements using quartz cells.

- Solutions:** 0.1 gm of pure cephalexin ($C_{16}H_{17}N_3O_4S$, M.wt= 347.4 g/mol, SDI -iraq) was dissolved in ethanol and completed the volume to 100 ml with ethanol to obtain 1000 mg/L.
- Preparation of calibration the curves:** six different volumes were taken from stock standard solution 1000mg/L are: (0.5, 1, 1.5, 2, 2.5 and 3 ml), these volumes transferred to set of 50 ml volumetric flasks and diluted with ethanol to an obtained concentration of pure cephalexin equal to (10, 20, 30, 40, 50 and 60 mg/L).
- Assay of market Cephalexin capsules:** 10 capsules of Cephalexin were weighed. Taken 0.1 gm of this powder and dissolved with ethanol, this solution is transferred into a 100 ml volumetric flask to give the concentration of 1000 mg/L. A set of appropriate dilution was made in 50 ml volumetric flask with ethanol and the concentrations of Cephalexin were determined.

Results and discussion

1-Zero order method

Zero order spectra were recorded a single peak at the wavelength of 263 nm for pure Cephalexin (Fig.1); Beer-Lambert's law was applying in the range 10-60 mg/l and the linearity data for this method is presented in Table 1.



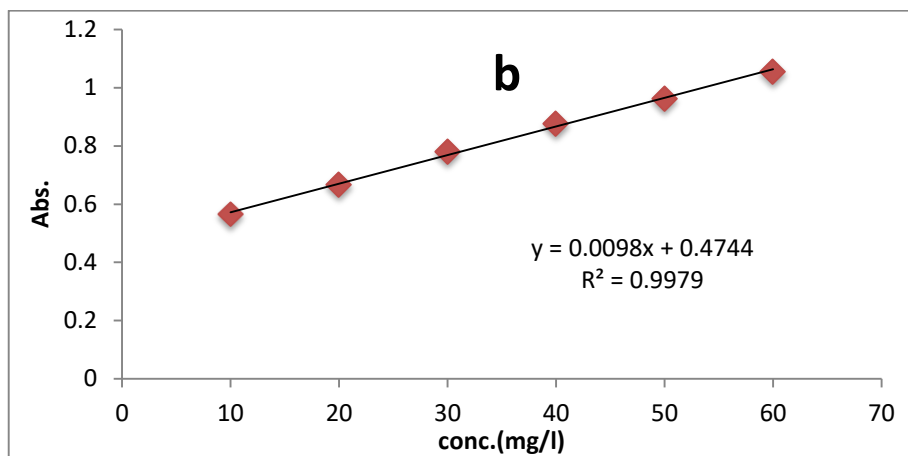


Fig. 1: (a) Zero-order spectra of Cephalexin 10-60 mg/l at peak= 263 nm and (b) the calibration curve taken of Cephalexin for this method.

2- Area under curve method

Area under curve for Cephalexin standard solutions was calculated in the range of 260-266nm (Figure 2), the principle of work Area under curve is depended on the following equation [12, 13] :

$$AUC = \frac{\lambda_1}{\lambda_2} Ad\lambda = 0.061x + 2.7418 \quad (R^2 \ 0.9976)$$

Where, λ_1 is a starting wavelength of 260 nm, λ_2 is the endpoint wavelength of 266 nm; A is absorbance of Cephalexin standard solutions, $d\lambda$ is the area under curve between two selected wavelengths, x is the concentration of Cephalexin standard solutions and R^2 is a determination coefficient.

The linearity between 10- 60 mg/L of Cephalexin standard solutions and other validation parameters are shown in table1 and figure3. Table1 has shown the values of statistical parameters of two methods.

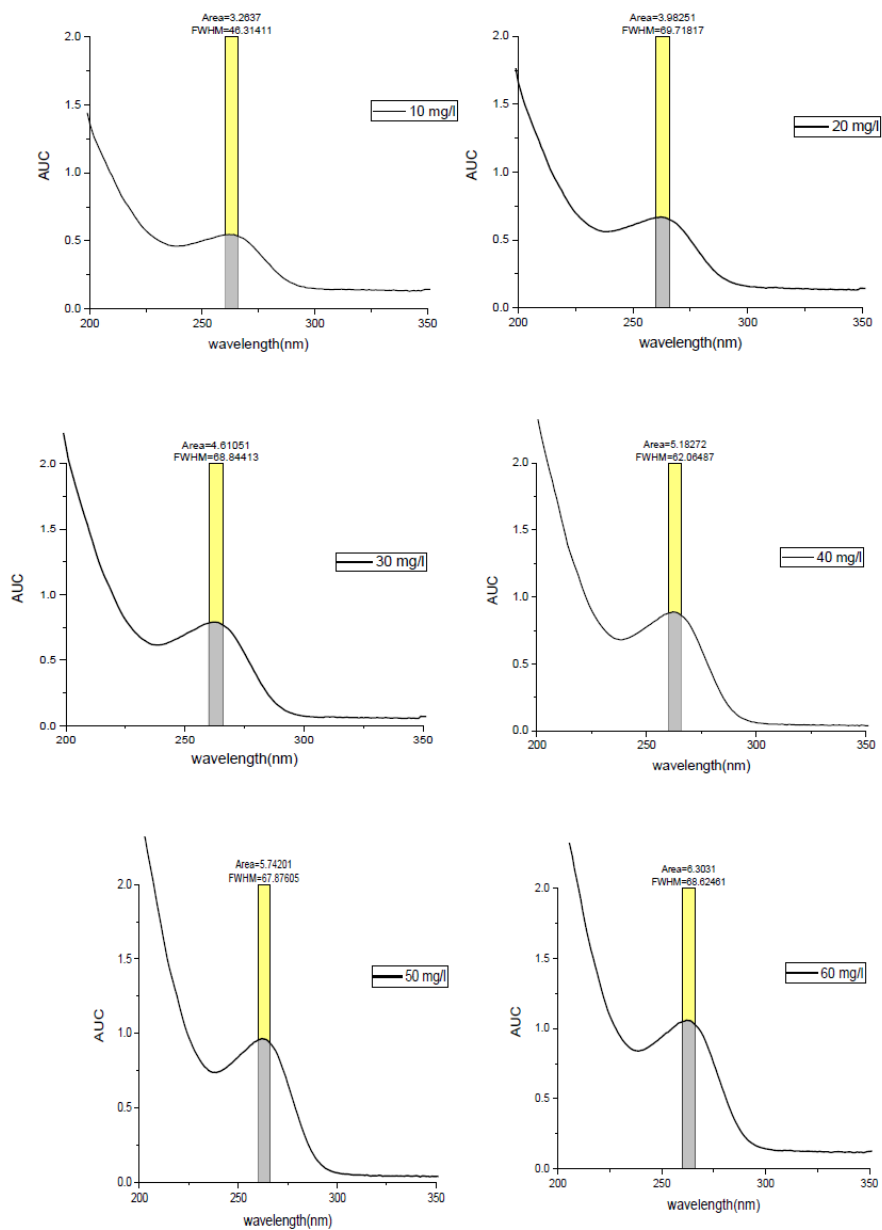


Fig. 2: Area under curves of Cephalexin with the concentrations of 10-60 mg/L and at the wavelength range (260-266nm)

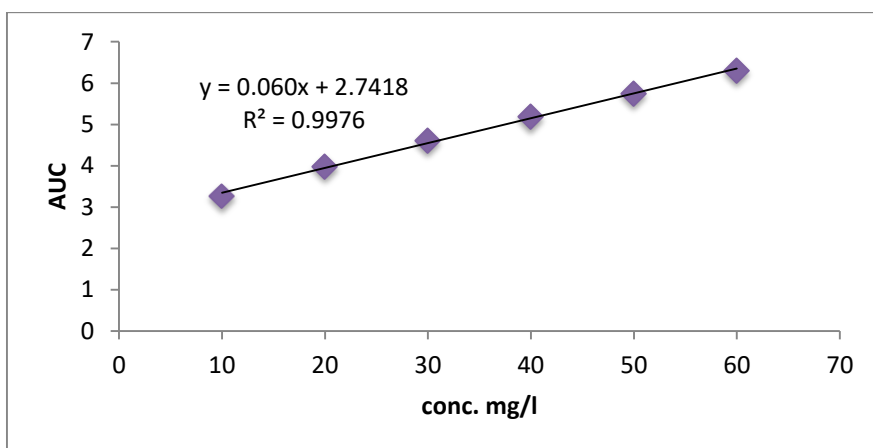


Fig. 3: Area under curve method-calibration curve for Cephalexin 10-60 mg/L at area (260-266nm).

Table 1: Statistical parameters obtained from the calibration curves of both methods.

method	Zero-order	Area under curve
Wavelength nm	263 nm	260-266 nm
R ²	0.9979	0.9976
Linearity range(mg/L)	10-60	10-60
Equation for linearity	$y = 0.0098x + 0.4744$	$Y=0.060x+2.7418$
Slope	0.0098	0.060
Intercept	0.4744	2.7418
SD of intercept	0.022	0.142
Mean*±SD	108.327± 4.140	101.265± 5.075
LOD (mg/L)	0.808	0.781

* mean of recovery, LOD = limit of detection = $3.3 \times \text{SD}/S$ and SD is the standard deviation of the intercept

Accuracy and Precision

Percentage of relative error (E%) and Recovery (Rec%) were used to check the accuracy and precision of this study. two concentrations were taken 20 and 40 mg/L of Cephalexin standard solutions. Each concentration was scanned three times. The accuracy and precision data were summarized in table2.

Table 2: Relative error and Recovery of two methods.

Methods	Concentration of Cephalexin (mg/ L)		Relative error (Er* %)	Recovery (Rec* %)	RSD*%
	Taken	found			
Zero-order	20	19.77	-1.15	98.85	0.189
	40	39	-2.50	97.50	0.578
Area under curve	20	20.68	-3.4	103.40	0.907
	40	38.83	-2.925	97.075	1.069

*Average of three time, $Er\% = \frac{\text{found}-\text{taken}}{\text{taken}} \times 100$, $Rec\% = E\% + 100$, $RSD\% = \text{relative standard deviation}$

Application

Cephalexin 500 Capsules (Each capsule contains Cephalexin monohydrate 500 mg) were determined by using two suggested methods. Two concentrations 25 and 55 mg/L of market Cephalexin capsule were measured three times by using UV-Visible spectrophotometer. Figures 4 and 5 of the market Cephalexin capsule shows there were no differences between them and the Cephalexin standard curves (Figures 1 and 2), it means that two proposed methods are suitable for the determination of Cephalexin in pure and capsule formulation. The percentage of relative error and recoveries are listed in table3.

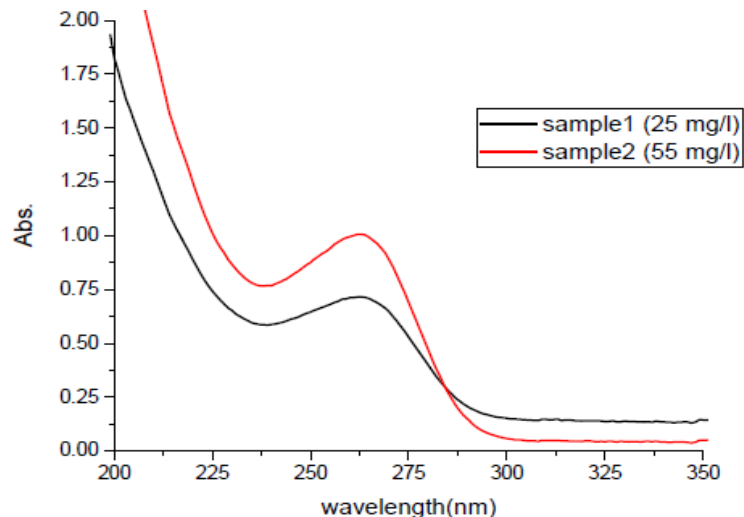


Fig. 4: Zero-order method-spectrum of market Cephalexin capsule at 25 and 55 mg/L

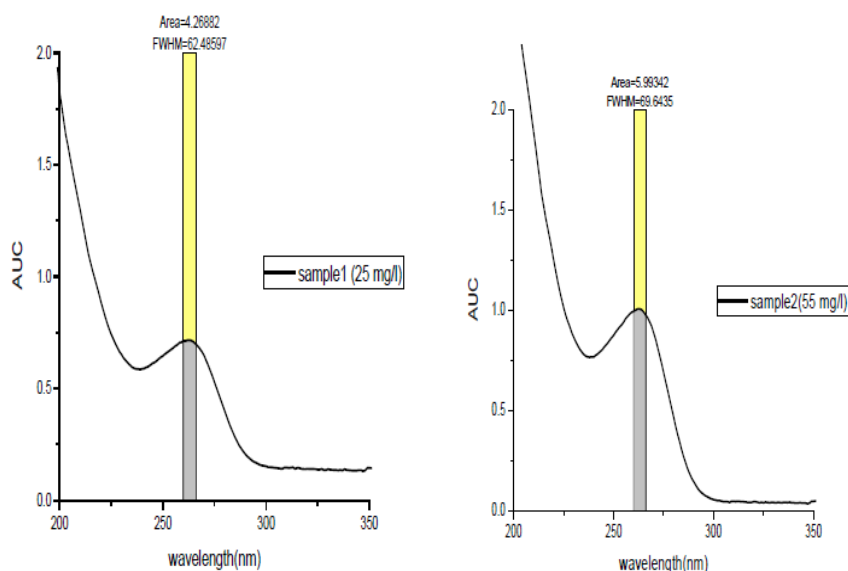


Fig. 5: Area under curve method-spectrum of market Cephalexin capsule at 25 and 55 mg/L

Table 3: The relative error and recovery of the market Cephalexin capsule at 25 and 55 mg/L

Pharmaceutical market tablet	method	Conc. of Cephalexin capsule mg/L		E* %	Rec.*%	RSD*%
		taken	found			
Cephalexin 500 Capsules-SDI	Zero-order	25	24.66	-1.36	106.640	0.793
		55	56.11	+2.01	102.01	0.665
	Area under curve	25	25.28	+1.12	101.12	0.873
		55	56.86	+3.381	103.381	0.702

*Average of three time, SDI= the state Company for Drugs Industry and Medical Appliances Samarra Iraq

Comparison with previous studies

A comparison of two analytical parameters of present methods (linearity range and LOD) with previous studies for determination of Cephalexin in different samples by using different methods are presented in table 4.

Table 4: Comparison with previous studies.

Method	Range	LOD	Samples	Ref.
RP-UFLC	1-120 µg/ml	0.24 µg/ml	Pharmaceutical dosage form	4
Electrochemical immunosensor	1-800 ng/ml	45.7 ng/ml	Meat samples	5
Fluorescence probes	0.1-50 µg /L	0.06 µg /L	Milk samples	6
Kinetic- spectrophotometric	1-16 µg/ mL	1.0 µg /mL	Drug samples	7
Colorimetric spectrophotometric	5-40 µg/ mL	2.814 µg/ mL	Pharmaceutical preparations	8
Zero-order spectrophotometric	10-60 mg/L	0.808 mg/L	Capsules	Present study
AUC spectrophotometric	10-60 mg/L	0.781 mg/L	Capsules	Present study

Conclusion

A fast, simple and cheap two spectrophotometric methods were used for estimation of cephalexin. Two methods had a high level of sensitivity for the detection of cephalexin in the range 10–60 mg/L. This study showed it can be determination of cephalexin at the absence of extraction, pH control and color development steps, which mean these methods, were suitable for estimation of cephalexin in pure and market capsule.

Acknowledgement

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طريقتين طيفيتين بسيطتين لتقدير السيفالكسين بصورتيه النقية والصيدلانية

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معلومات البحث:	الخلاصة:
تاريخ الاستلام: 2020/09/17 تاريخ القبول: 2021/01/04	في هذه الدراسة، تم تقدير السيفالكسين بطريقتين طيفيتين بسيطتين دون الحاجة إلى الاستخلاص لتقدير السيفالكسين بصورتيه النقية والمستحضرات الدوائية. كلا الطريقتان تعتمدان على طيف الأشعة فوق البنفسجية، حيث أظهرت طريقة المرتبة الصفيرية أعلى امتصاص عند الطول الموجي 263 نانومتر، بينما تم في الطريقة الثانية الاعتماد على المساحة تحت المنحني ضمن المدى (260-266 نانومتر). كان مدى تقدير التراكيز للطريقتين (10-60 ملغم/ لتر)، حد الكشف بلغ 0.808 ملغم/لتر لطريقة المرتبة الصفيرية وبلغ 0.781 ملغم/ لتر لطريقة المساحة تحت المنحني. وتم تطبيق الطريقتان بنجاح لتقدير السيفالكسين في المستحضرات الصيدلانية.
الكلمات المفتاحية:	
سيفالكسين، المساحة تحت المنحني، التقدير الطيفي	