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Comparative study on the selectivity of various spectrophotometric techniques for the determination of binary mixture of fenbendazole and rafoxanide

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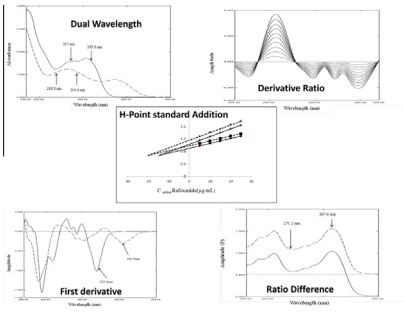
HIGHLIGHTS

- Application of five different spectrophotometric methods.
- Comparing selectivity of five spectrophotometric methods.
- Simultaneous determination of fenbendazole and rafoxanide.
- One way ANOVA statistical analysis for comparison of the proposed methods.

GRAPHICAL ABSTRACT



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ABSTRACT

Five different spectrophotometric methods were applied for simultaneous determination of fenbendazole and rafoxanide in their binary mixture; namely first derivative, derivative ratio, ratio difference, dual wavelength and H-point standard addition spectrophotometric methods. Different factors affecting each of the applied spectrophotometric methods were studied and the selectivity of the applied methods was compared. The applied methods were validated as per the ICH guidelines and good accuracy; specificity and precision were proven within the concentration range of 5–50 μg/mL for both drugs. Statistical analysis using one-way ANOVA proved no significant differences among the proposed methods for the determination of the two drugs. The proposed methods successfully determined both drugs in laboratory prepared and commercially available binary mixtures, and were found applicable for the routine analysis in quality control laboratories.

Introduction

Fenbendazole (Fen) is methyl [5-(phenylsulfanyl)-1H-benzoim idazol-2-yl] carbamate, a white or almost white powder [1], used as an anthelmintic drug for the treatment of benzimidazole susceptible mature and immature stages of nematodes and cistodes of the gastrointestinal and respiratory tracts of cattle and sheep [2].

Rafoxanide (Raf) is N-[3-chloro-4-(4-chlorophenoxy) phe nyl]-2-hydroxy-3,5-diiodo-benzamide [3]. It has an activity against fasciola hepatica in the mature and immature stages [2].

The mixture of Fen and Raf is formulated as oral suspension which is widely used as a veterinary broad spectrum anthelmintic.

Literature survey revealed that several methods have been reported for determination of Fen in single component preparations and in different mixtures. These include High performance and ultra-high performance liquid chromatography [1,4–6], densitometry [7], electrophoresis [8–10], voltammetry [11,12],

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potentiometry [1], colorimetry via ion pair complex formation with bromothymol blue [13] and derivative spectrophotometry [14].

Raf was determined via HPLC [15,16], GC [17], colorimetry via formation of charge transfer complex with iron III [18].

Spectrophotometry has been widely accepted and extensively used in pharmaceutical analysis as an alternative to the costly high performance liquid chromatography. Different spectrophotometric methods have been introduced for the resolution of overlapped spectral data to allow simultaneous determination of compounds in mixtures.

Derivative spectrophotometry is a useful technique for extracting qualitative and quantitative information from spectra composed of unresolved bands and offers background correction and better selectivity than normal spectrophotometry for resolving binary mixtures and some ternary mixtures [19,20].

Derivative ratio spectrophotometry is one of the popular methods used in simultaneous determination of compounds in the presence of interfering substances or mixtures of drugs [21,22].

Ratio difference (ΔP) spectrophotometry developed by Elzanfaly et al. represented a simple solution with minimal data manipulation for the resolution of overlapped spectra by simply calculating the difference in amplitudes between two wavelengths in the ratio spectrum [23].

Dual wavelength spectrophotometry was used to resolve binary mixtures with overlapping spectral data, where the interference of one component is nullified by careful selection of a pair of wavelengths at which the difference in absorptivity for one component remains zero while the other component exhibits a linear quantitative relationship [24].

H-point standard addition method (HPSAM) shares the same principle with the dual wavelength spectrophotometry and the standard addition method. However HPSAM permits the correction of proportional and constant errors resulting from interfering compound and sample matrix [25].

To the best of our knowledge, no method has been reported for the simultaneous determination of Fen and Raf in their pharmaceutical preparations.

The aim of the present work was to develop five different simple, sensitive and precise spectrophotometric methods; namely first derivative, derivative ratio, ratio difference, dual wavelength and H-point standard addition spectrophotometric methods capable of simultaneous determination of the studied drugs in the absence of a reported method for their simultaneous determination in their available dosage form, and to compare their selectivities. Different factors affecting each of the applied spectrophotometric methods were studied and experimental conditions were optimized.

Experimental

Apparatus

Spectrophotometer: Shimadzu UV-2450 PC Series Spectrophotometer (Tokyo – Japan) with two matched 1 cm quartz cells using the following spectral parameters; a single fast scan mode and a slit width (2 nm), connected to a computer loaded with shimadzu UVPC software and used for all the absorbance measurements and data manipulation.

SPSS program (PASW Statistics 18) version 18.0.0.

Materials

Fen and Raf working standards were kindly supplied by Pharma-Swede, Egypt. Their purities were found to be 99.00 ± 0.586% [1] and 99.40 ± 0.521% [26], respectively.

Parafluke® suspension, labeled to contain 50 mg/mL of Fen and Raf, batch number S110640, manufactured by Pharma-Swede, Egypt.

All chemicals and solvents used were of analytical grade; Methanol; ADWIC [Cairo-Egypt], Hydrochloric acid; ADWIC [Cairo-Egypt]; (0.1 N methanolic solution).

Solutions

Standard solutions

Fen and Raf stock standard solutions 100 μg/mL in 0.1 N methanolic HCl.

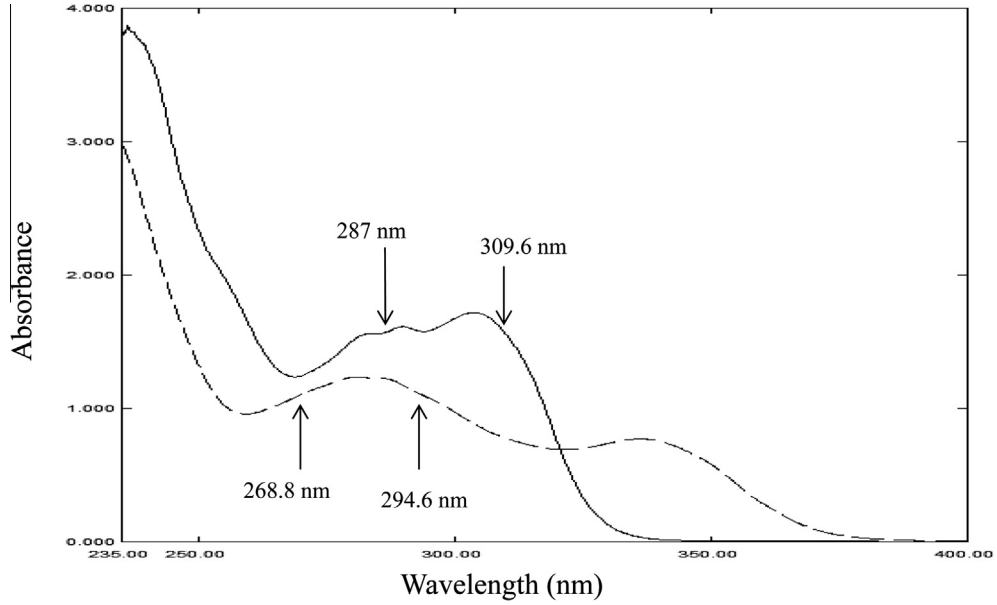

Fig. 1. Absorption spectra of 50 μg/mL Fen (—) and 50 μg/mL Raf (----) using 0.1 N methanolic HCl as a blank.