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FT-IR Spectroscopic Assay Method for Amlodipine Besylate in Bulk and Tablet Formulations

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ABSTRACT

A simple, precise and rapid method has been developed for the quantification of Amlodipine Besylate (AMB) using FT-IR technique in bulk and tablet formulations. The method involves the measurement of absorbance of carbonyl group (C=O) peak at 1676 cm^{-1}. The limit of LOD and limit of LOQ were 0.001mg and 0.032mg. The proposed method was validated for pharmaceuticals in tablet form and %RSD was found to be less than 2 with recovery levels >98%.

Keywords: FT-IR, Amlodipine Besylate, Carbonyl peak, Tablet formulation.

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INTRODUCTION

Chemically, Amlodipine Besylate (AMB) (Fig 1) is 3-Ethyl-5-methyl (±)-2-[(2-aminoethoxy) methyl]-4-(2-chlorophenyl)-1, 4-dihydro-6-methyl-3, 5-pyridinedicarboxylate, monobenzenesulphonate and is is an anti-hypertensive and an antianginal agent in the form of besylate salt. Many methods were available in literature for its determination using Spectrophotometric [1-6], HPLC [7-8], Colorimetric [9], Voltammetry [10], HPTLC [11] techniques, but methods for its determination using Infrared Spectroscopy technique were not available. Hence an attempt was made to develop a new analytical method which is simple, rapid and non-destructive using FT-IR for the assay of Amlodipine Besylate in pure and tablet forms.

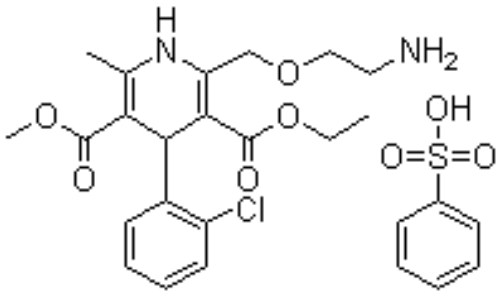

Figure 1. Chemical structure of AMB

EXPERIMENTAL

Materials and Methods

Materials

FT-IR spectra were recorded on Thermo Nicolet, Model Nexus 670, USA, Spectrometer. KBr used for recording the spectra was spectroscopic grade obtained from Sigma-Aldrich, Germany.

The reference standard of Amlodipine Besylate was obtained as a gift sample from M/s Dr. Reddy's Laboratories, Hyderabad. Single dosage form tablets Amlong (2.5mg) and Amlong (5.0mg) were procured from local market, Hyderabad.

Methods

Standards preparation and calibration:

Fused KBr pellet spectra were recorded between 4000cm^{-1} and 400cm^{-1} by averaging 32 scans with a resolution of 4cm^{-1} with a DTGS detector. For calibration, the spectra were recorded by compressing the standard substance AMB in the concentration range 0.2mg to 0.49mg in spectral grade KBr.

Sample preparation and formulation analysis:

Ten tablets of Amlong were weighed and ground to a fine powder. A known quantity of it equivalent to the concentration of the individual drug in the calibration range was compressed with spectral grade KBr. Four KBr discs of different concentrations were prepared and spectra were recorded under similar experimental conditions as standard.

Recovery studies

To validate the developed method for its accuracy, reproducibility and interferences from formulation excipients, recovery studies were carried out. A known amount of pure drug was added to the preanalysed tablet powder at two concentration levels 50% &100% and analysed by the proposed method.

Linearity:

Standard sample of AMB was prepared in different concentration range from 0.20mg – 0.49mg. The linearity of the investigation was done by measuring the peak absorbance of the drug at different concentrations as mentioned above. Calibration plot was constructed by plotting peak absorbance against the concentration of the drug.

The limit of detection (LOD) is the lowest concentration of an analyte in a sample that can be detected and the limit of quantification (LOQ) is the lowest concentration of an analyte in a sample that can be quantitated. Both LOD and LOQ were experimentally verified and calculated using the following equation.

$$LOD = 3.3 (SD/Slope)$$
$$LOQ = 10 (SD/Slope)$$

Precision and Accuracy:

Precision study was performed by taking four readings for each concentration and %RSD was calculated. Accuracy was determined from the recovery studies. The percent mean recovery and percent RSD were calculated.

RESULTS AND DISCUSSION

The FT-IR spectrum of pure AMB by KBr pellet method is given in Figure 2. The compound exhibited strong sharp signal at 1676cm^{-1} (AMB) which is due to the absorption of carbonyl group (C=O) and this feature is taken for the quantitative analysis. A calibration has been carried out with known quantities of standard as mentioned in the experimental section. The overlain spectra and calibration plot of different concentrations were given in Figure 3 and Figure 4 respectively**.** The calibration was followed Beer-Lamberts law in the concentration range studied (0.2mg - 0.49mg) .The optical parameters were evaluated and included in Table

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1. The Limit Of Detection and Limit of Quantification (LOD&LOQ) were found to be 0.001mg and 0.032mg respectively with a correlation coefficient(R^2) 0.9986.

Table 1: Optical characteristics

Parameters	Amlodipine Besylate
Linearity	0.2 - 0.49mg
Correlation coefficient (R^2)	0.99863
Regression equation (Y*)	Y= -0.00727 + 1.035 x
Slope (B)	1.03599
Intercept (A)	-0.00727
LOD (mg)	0.001
LOQ (mg)	0.032

Y=A + Bx, where x is the concentration of analyte and y is the absorbance value

Table 2: Recovery studies of Amlodipine Besylate

S.No.	Tablet	%Level	Preanalysed conc. (mg)	Standard conc. added (mg)	Total amount (mg)	*Amount of drug recovered (mg)	Mean %recovery	%RSD
1.	**Amlong (2.5mg)**	50%	2.5	1.27	3.77	3.69	98.02	0.58
		100%	2.5	2.53	5.03	4.94	98.33	0.60
2.	**Amlong (5.0mg)**	50%	5.0	2.55	7.55	7.41	98.20	1.40
		100%	5.0	5.0	10.0	9.80	98.07	0.40

*Average of four determinations

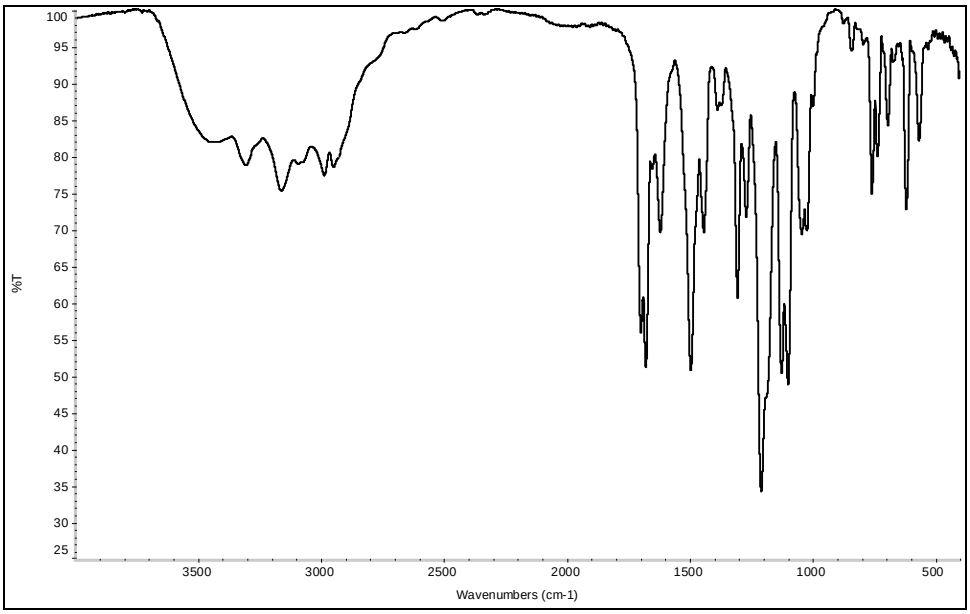

Figure 2 FT-IR spectrum of Amlodipine Besylate Standard

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Table 3: Analysis of commercial formulations

S. No.	Tablet	Label claim	% assay*	±SD	% RSD
1.	**Amlong**	2.5mg	100.69	0.68	0.67
2.	**Amlong**	5.0mg	98.49	0.54	0.55

*Average of four determinations

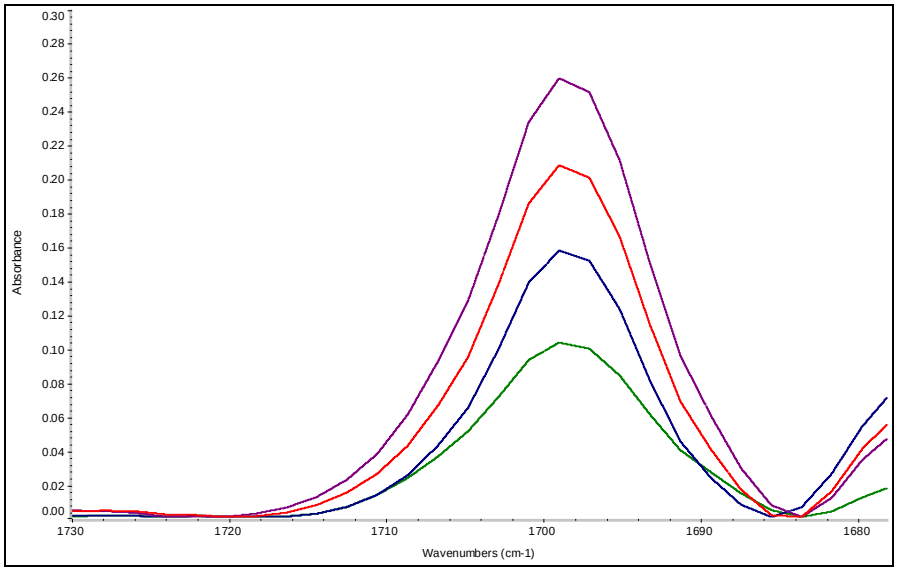

Figure 3: Amlodipine Besylate standard – overlain spectra of four concentrations at 1676 cm^{-1}

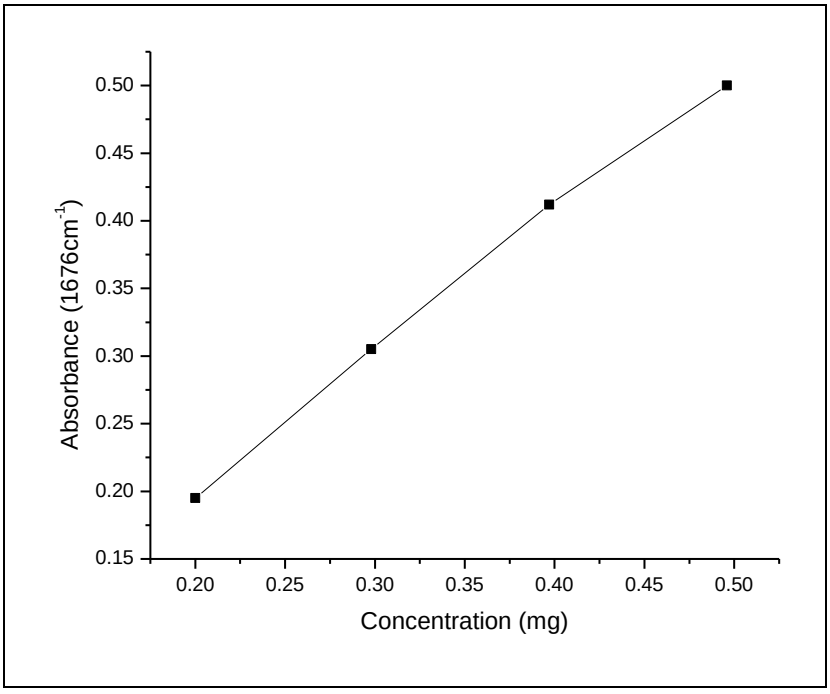

Figure 4: Calibration plot of Amlodipine Besylate

The method was applied to single dosage formulations and the spectra were recorded for Amlong (2.5mg) and Amlong (5.0mg) tablets. The peak absorbance at 1676 cm^{-1} was measured and calculated the concentration from the calibration graph. The accuracy studies in terms of % recovery and % RSD (η=4) for commercial tablets Amlong (2.5mg) and Amlong (5.0mg) were tabulated in Table 2. The % recovery was found to be >98 with a % RSD less than 2. The values of % recovery and % RSD show a high precision of the method. Table 3 shows the assay and % RSD (η=4) for commercial tablets. The % RSD was found to be 0.67 and 0.55 respectively. The estimation of AMB in tablet formulations by the proposed method yielded precise results indicating the reliability of the method.

CONCLUSIONS

In the present investigation we have studied the possibility of quantification of Amlodipine in single dosage formulation using FT-IR. From the data it is clear that FT-IR is capable of direct determination of AMB in the above formulations. The proposed FT-IR method was found to be simple, rapid, reproducible and less time consuming compared to other analytical methods, which exists in literature.

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