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Research article

METHOD DEVELOPMENT OF DETERMINATION OF QUALITY AND QUANTITY IN POLY-HERBAL DOSAGE FORM OF CARYOPHYLLINE

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ABSTRACT

Caryophyllene otherwise called as (−)-β-caryophyllene, is a natural bicyclic sesquiterpene that is a constituent of many essential oils, especially clove oil, the oil from the stems and flowers of *Syzygium aromaticum* (cloves), the essential oil of *Cannabis sativa*, rosemary, and hops. It is usually found as a mixture with isocaryophyllene (the *cis* double bond isomer) and α-humulene (obsolete name: α-caryophyllene), a ring-opened isomer. Caryophyllene is notable for having a cyclobutane ring, as well as a *trans*-double bond in a 9-membered ring, both rarities in nature. It has ability to bind with CB2 receptors in the endocannabinoidsystem, beta-caryophyllene has potent anti-inflammatory and antioxidant effects. It could help to relieve pain and anxiety, treat seizures, and reduce cholesterol. The conclusion shows the proposed method is simple, accurate, rapid, precise, economic and reproducible for UV spectrophotometric of caryophyllene estimation from pharmaceutical formulation. This method is applied for routine estimation of caryophylline in bulk & pharmaceutical dosage form.

Keywords: Caryophylline, Anti-inflammatory, UV method.

INTRODUCTION

Caryophyllene otherwise called as (−)-β-caryophyllene, is a natural bicyclic sesquiterpene that is a constituent of many essential oils, especially clove oil, the oil from the stems and flowers of *Syzygium aromaticum* (cloves), the essential oil of *Cannabis sativa*, rosemary, and hops. It is usually found as a mixture with isocaryophyllene (the *cis* double bond isomer) and α-humulene (obsolete name: α-caryophyllene), a ring-opened isomer. Caryophyllene is notable for having a cyclobutane ring, as well as a *trans*-double bond in a 9-membered ring, both rarities in endocannabinoid system, beta-caryophyllene has potent anti-inflammatory and antioxidant effects. It could help to relieve pain and anxiety, treat seizures, and reduce cholesterol. In order to determine the drug in biological fluid or in pharmaceutical preparations, there are no. of methods available, that is HPTLC, HPLC, and spectrophotometery. The new, simple, reliable, rapid, precise ultraviolet spectrophotometric method has to validate and been developed to analyses caryophylline in bulk & poly-herbal formulation. Statistical tests are conducted on validation data [1-5].

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MATERIALS AND METHODS

Instrument Used

UV-Vis spectrophotometer 1700, Make: Shimadzu, Kyoto, Japan, Scan speed: 40nm/min, Bath Sonicator

Reagents and Solutions

All the reagents used in this assay were of analytical grade. Poly herbal tablet of caryophyllene were powdered and weighed.

EXPERIMENTAL

Determination of λ_{max}

Weighed amount of caryophylline was mixed with 0.1N NaOH to obtain a solution. This solution subjects to scan between 200 – 400nm & determines maximum absorption. Study the dilution effect of on absorption maximum was by diluting the above stock solution to 20µg/ml and scanned from 200-400nm.

Preparation of Standard Stock Solution

Standard drug solution of caryophyllene was prepared by dissolving 10 mg caryophyllene in 100 ml 0.1N NaOH to obtain stock solution of 100 µg/ml concentration.

Preparation of Calibration Curve

Calibration curve was prepared in 0.1N NaOH at λ_{max} 276nm using UV-Vis spectrophotometer Model 1700. For this stock solution of 100 µg/ml was prepared. Serial dilution of 10, 15, 20, 25, 30µg/ml were prepared and absorbance was taken at λ_{max} 276nm. Averages of such 6 sets of values were taken for calibration curve, and solution were scanned in the range of 200-400 nm against blank [6-8].

Assay

500mg **o**f tablet containing 5 mg caryophyllene was weighed. Tablet equivalent to 100 mg of caryophyllene was transferred into 100 ml volumetric flask dissolved in 0.1N NaOH. The solution was then filtered through Whatmann filter paper No 40 (0.45 micron). Aliquots of the sample were removed and diluted to 10 ml of 0.1N NaOH to obtain strengths of 20µg/ml determined at the respective absorbance of 276nm against 0.1N NaOH as a blank.

Limit of Detection (LOD) and Limit of Quantification (LOQ)

The LOD and LOQ of GLP were determined by using standard deviation of the response and slope approach as defined in International Conference on Harmonization guidelines. The LOD and LOQ were seen in table 1.. Limit of detection and limit of quantification were calculated by Equation (1) LOD= $3.3\delta/s$ and (2) LOQ = $10\ \delta/s$ respectively, where δ is the standard deviation of blank and s is slope.

Recovery studies

Recovery studies were conducted to judge the accuracy of the method. Recovery studies were performed by addition of a known quantity of pure drug to the pre-analyzed formulation and the proposed method was followed. % recovery were calculated from the amount of drug. Recovery study was performed at 3 different concentration levels by adding standard drug to the sample.

RESULTS AND DISCUSSION

The UV scan of standard solution between 200 – 400 nm showed the absorption maxima at 276nm. The overlay spectra of different concentration range of standard caryophyllene was recorded fig.1. The Beer's law was verified from the calibration curve by plotting a graph of concentration vs. absorbance fig.2. The linearity range was observed between 12-35µg/ml. The plot clearly showed a straight line passing through origin with equation Y= 0.0421X - 0.0497 with correlation coefficient of 0.997. The coefficient of correlation was highly significant. The optical characteristics and other validation parameters are thus summarized in Table 1**.** The assay method was validated by low values of standard deviation and standard error, indicating accuracy and precision in table2 of the methods. Excellent recovery studies further prove the accuracy of the method in table 3. The assay result was repeated for three times which was found to be 101.27-102.84% of labelled claim in table 4 [9-13].

Table 1.Optical Parameters for Caryophylline

S. No.	Parameters	values
1	max(nm)	276
2	linearity range	12-35µg/ml
3	regression equation	Y=0.0421X-0.0497
4	correlation coefficient	0.997
5	slope	0.0453
6	intercept	0.0312
7	Limit of detection(µg/ml)	0.7538
8	Limit of quantification(µg/ml)	2.9046

Table 2. Precision Data ForCaryophylline

S. No.	Conc. ug/ml	intraday	cv	Interday	cv
1	15	0.54228±0.00672	0.9123	0.5178±0.0064	3.265
2	20	0.7465±0.0243	2.9801	0.7946±0.0092	4.190
3	25	0.8712±0.0059	0.5846	0.804±0.0053	0.798

Table 3.Recovery Study Data for Caryophylline

S. No.	Amount of sample (ug/ml)	Added drug (ug/ml)	Drug recovered (ug/ml) ±sd	%recovery
1	20	0	21.6834±0.4782	100.7931
2	20	10	31.7863±0.3096	100.2847
3	20	20	41.4781±0.5468	100.6079
4	20	30	51.3926±0.6053	100.5694

Table 4. Assay Results for Caryophylline

S. No.	Actual conc. (µg/ml)	Amount obtained (µg/ml)	%drug
1	20	20.19	101.27
2	20	21.32	102.84
3	20	21.56	101.69

CONCLUSION

The conclusion shows the proposed method is simple, accurate, rapid, precise, economic and reproducible for UV spectrophotometric of caryophyllene estimation from pharmaceutical formulation. This method is applied for routine estimation of caryophylline in bulk & pharmaceutical dosage form.

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