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Validated estimation of Gingerol and Curcumin in multicomponent herbal tablet by UV spectroscopy

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Abstract

Ginger, *Zingiber officinale* Roscoe Family- Zingiberaceae, has been used in Chinese and Indian folk medicine for centuries. Turmeric, *Curcuma longa* Linn is from the family of Zingiberaceae. There are no reported UV methods for simultaneous estimation of both the extracts, which is necessary in the development of Herbal formulation for these drugs. Hence, a simple UV spectroscopic method was developed and validated for direct estimation of gingerols and curcuminoids assay in pharmaceutical tablet formulations. Methanolic extracts of rhizome extracts were prepared and analyzed at λ max 280 and 421 nm respectively. The developed method was validated for intraday and interday variations as per ICH Q2A guideline and was found to be a stable method. Novel Pharmaceuticals, Nutraceuticals as well as cosmeceutical products can be conveniently analyzed using this method.

Keywords: Ginger; Turmeric; Multicomponent; UV spectroscopy; Validation

1. Introduction

In the past, the collection, identification, and preparation of Ayurvedic medicines were done by the Acharyas themselves; so drugs made by them were more efficacious, authentic and genuine. In the present age the suppliers make the collection. There are so many drugs; which lose their effectiveness with the passage of time. This lowers the genuine character of the drug and makes them less efficacious. Hence a need to develop the method for analysis of the extracts (1). Curcumin is chemically, (1E, 6E)-1, 7-bis (4-hydroxy-3- methoxy phenyl) -1, 6-heptadiene-3,5-dione. It is the principal curcuminoid of the popular Indian spice turmeric, which is a member of the ginger family (Zingiberaceae). The other two curcuminoids are desmethoxycurcumin and bis-desmethoxycurcumin. The curcuminoids are natural phenols and are responsible for the yellow color of turmeric. Curcumin has a long history of use for maintaining a healthy inflammatory response, via its effects on cyclooxygenase, prostaglandin and leukotriene metabolism. Ginger is native to Asia where it has been used as a cooking spice for at least 4000 year (2).The present work involves development and validation of a spectrophotometric method for analysis of multicomponent gastro-retentive herbal tablets containing Curcumin extract and Ginger extract as a drug candidate(3).

2. Material and methods

2.1. Apparatus

Instrument used was an UV/Visible double beam spectrophotometer, SHIMADZU model no.1800 (Japan) with spectral width of 2 nm, wavelength accuracy of 0.5 nm and a pair of 10 mm matched quartz cells. An electronic analytical balance was used for weighing all the samples. All the other chemicals and solvents used were of A.R. grade; Curcumin and Ginger extract was procured as a gift sample from herbal creation Bangalore India.

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2.2. Development of the method [4]

During the development of the method, the spectrum of both the extracts was measured in different solvents. This was done to enable selection of the solvent system and concentration range. Based on the spectrum, the solvent selected was methanol for both the extracts. The method was developed and validated as per the procedures mentioned.

2.3. Preparation of Standard Stock Solutions

Accurately weighed Curcumin extract (10 mg) and Ginger extract (100 mg) was transferred to a 100 mL volumetric flask, dissolved and diluted to the mark with methanol to obtain a standard solution having concentration curcumin extract (100 mg/mL) and Ginger extract (1000 mg/mL).

2.4. Calibration curve of Curcumin and Ginger

A series of calibrated 10mL volumetric flasks were taken and appropriate aliquots of the working standard solution of Curcumin were withdrawn and diluted up to 10mL with methanol (working standard solutions having concentration 100, 200, 300, 400, 500 µg/mL). The absorbance was measured at absorption maxima 421 nm, against the reagent blank prepared in similar manner without curcumin i.e Methanol. Same procedure was applied for Ginger (working standard solutions having concentration 15, 25, 35, 45, 55 µg/mL) and absorbance was measured at 280 nm, against reagent blank prepared in similar manner without ginger.

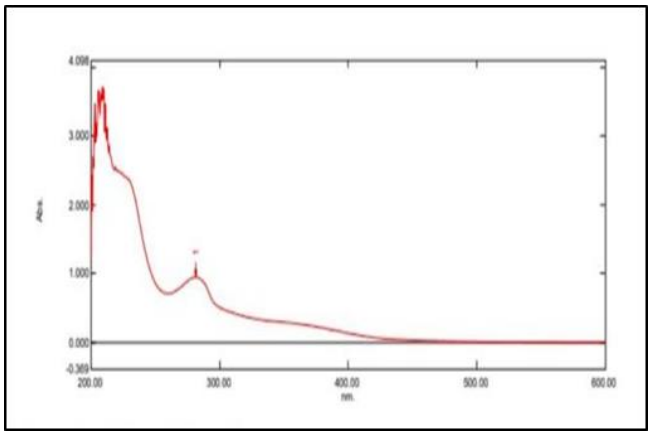

Figure 1 UV Spectrum of Ginger at 280 nm

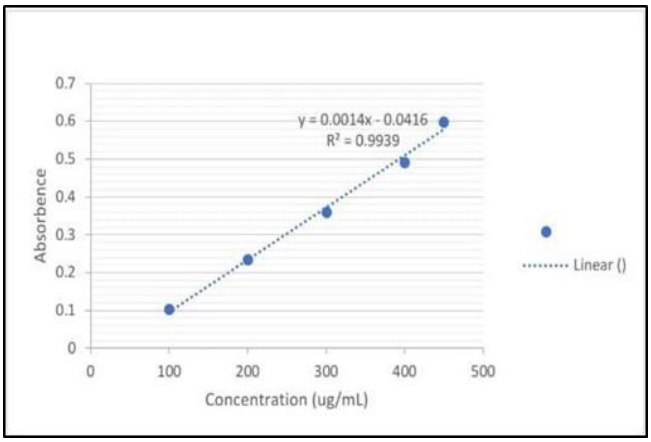

Figure 2 Calibration curve of Ginger

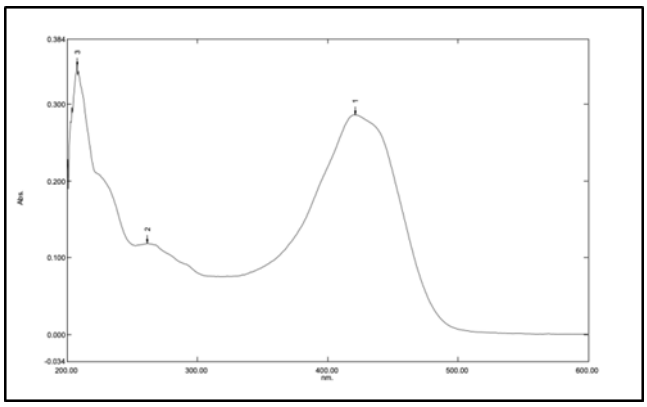

Figure 3 UV Spectrum of Curcumin at 421 nm

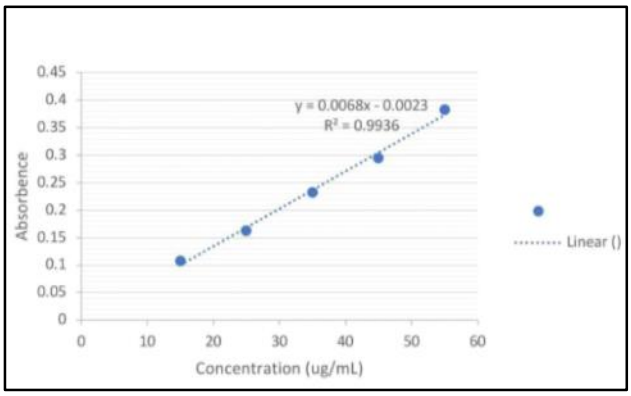

Figure 4 Calibration Curve of Curcumin

2.5. Method Validation

2.5.1. Linearity

Calibration curves were plotted over a concentration range of 100 – 450 µg/mL for Ginger extract and concentration range of 15 - 55 µg/mL for curcumin extract. The absorbance was measured at 280 nm and at 421 nm for both the drugs.

The calibration curves were constructed in triplicate by plotting absorbance vs. concentration and the regression equations were calculated.

2.5.2. Precision

Intraday Precision

Solutions containing 100-450 µg/mL of ginger extract and 15-55 µg/mL of curcumin at low, middle and high concentration were analyzed 3 times on the same day and % RSD was calculated.

Interday Precision

Solutions containing 100-450 µg/mL of ginger extract and 15-55 µg/mL of curcumin at low, middle and high concentration were analyzed on 3 subsequent days and % RSD was calculated.

2.5.3. Accuracy

The accuracy of the method was determined by calculating recoveries of Ginger extract and Curcumin extract by the standard addition method. Known amounts of standard amount of Ginger extract was added at 80, 100 and 120 % levels to pre-quantified sample solutions of 1000 µg/mL ginger 100 µg/mL Curcumin extract. The absorbance of Ginger extract and Curcumin extract were recorded at 280 nm & 420 nm. The percentage recovery was calculated by measuring the absorbance of both drug at their absorbance maxima. Each response was average of three determinations.

2.5.4. Limit of Detection and Limit of Quantitation

The limit of detection (LOD) and the limit of quantitation (LOQ) of the drug were derived by calculating the signal-to noise ratio (S/N, i.e., 3.3 for LOD and 10 for LOQ) using the following equations designated by International Conference on Harmonization (ICH) guidelines.

$$LOD = 3.3 \times \sigma/S$$

$$LOQ = 10 \times \sigma/S$$

Where;
σ = the standard deviation of the response,
S = Slope of calibration curve

2.5.5. Analysis of Ginger extract and Curcumin extract in Herbal Tablet

Tablet equivalent to 0.1 gm of Ginger and 0.01 gm of curcumin were weighed and transferred into a 10 mL volumetric flask. Using this solution, dilution equivalent to 100µg/mL was prepared. Absorbance of the resulting solution was measured at 280 nm and 421 nm against methanol, relative concentration of two drugs in the sample was calculated.

3. Results

The two wavelengths used for the analysis of the drugs in the combination dosage form were 421 nm (λ max of curcumin) and 280 nm (λ max of Ginger extract) at which the calibration curves were prepared for both the drugs.

3.1. Validation

3.1.1. Calibration curve

Linear correlation was obtained between absorbance versus concentrations of Ginger extract in the ranges of 100 – 450 µg/mL and curcumin extract in the range of 15 - 55 µg/mL . Regression parameters are mentioned in table 1 and the calibration curves of these two drugs at 280 nm and 421 nm were validated by the high value of correlation coefficients of regression (Table 1).

Table 1 Regression analysis data and summary of validation parameter for the proposed method

Parameter	Ginger extract	Curcumin extract
Wavelength range (nm)	280	421
Linearity & Range (µg/mL)	100-500	15-55
Regression equation (y = mx + c)	y=0.0014x-0.0416	y=0.0068x-0.0023
Slope	0.0014	0.0068
Intercept	0.0461	0.0023
Correlation coefficient (r)	0.9939	0.9936
Precision (%RSD)		
1a. Intraday Precision (n=3)	0.14 - 0.32 %	0.1 - 0.4%
1b. Interday Precision (n=3)	0.11 - 0.93 %	0.15 - 0.52%
2.System Precision	0.42 %	0.55%
3.Method Precision	1.3%	1.5%
Accuracy (% Recovery) (n=3)	98.9-99.5%	98.7-99.6%
LOD	28.1 µg/mL	2.8 µg/mL
LOQ	85.39 µg/mL	8.6 µg/mL

% RSD = Percent relative standard deviation; LOD = Limit of detection ; LOQ = Limit of quantitation ; SD = Standard deviation; n = number of replicates

Accuracy was determined by calculating the recovery and the mean was determined (Table 2).

Table 2 Recovery data for proposed method

Drugs	Amount of sample taken (µg/mL)	Amount added (µg/mL)	Amount added in %	% Mean Recovery ± SD*(n=3)
Ginger extract	300	240	80	98.9±0.17
	300	300	100	99.02±0.5
	300	360	120	99.58±0.08
Curcumin extract	35	28	80	98.7±0.88
	35	35	100	99.41±0.3
	35	42	120	99.68±0.3

3.2. Application of validated Method to the in-house prepared Tablet

Tablet equivalent to 0.1 gm of Ginger and 0.01 gm of curcumin were weighed and transferred into 10 mL volumetric flask. Using this solution, dilution equivalent to 100µg/mL was prepared. Absorbance of the resulting solution was measured at 280 nm (y=0.0014x-0.0416) and 421 nm (y=0.0068x-0.0023) against methanol, relative concentration of two drugs in the sample was calculated using above equations. The assay found to be 95% and 98% respectively.

4. Discussion

The proposed method was found to be simple, sensitive, accurate, precise, economical and rapid for the routine estimation of Ginger and Curcumin in bulk and pharmaceutical dosage forms.

5. Conclusion

The developed method is found to be simple, sensitive, accurate and precise and can be used for routine analysis of Ginger extract and Curcumin extract as well as combination products containing both the extracts. The developed method was validated as per ICH guidelines. Statistical analysis proved that the method is repeatable and selective for the analysis of Ginger extract and curcumin extract in their combined pharmaceutical formulations.

Compliance with ethical standards

Acknowledgments

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Disclosure of conflict of interest

The authors declare that they have no conflict of interesting.

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