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Research Article

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Determination of UV-Vis Spectrophotometric Method of Metal Complexes Stoichiometry between Cu (II) and Zn (II) with Vilazodone Hydrochloride

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ABSTRACT

In the presenst research work, simple, precise and accurate UV-Vis spectrophotometric method was developed for the determination of metal complexes stoichiometry of Cu(II)-Vilazodone Hydrochloride and Zn(II)-Vilazodone Hydrochloride. The separate spectrums of Vilazodone Hydrochloride and the complexes occured between Cu(II) and Zn(II). Metal ions were taken in ultra-violet area, and the stoichiometric ratios of the complexes occured between Vilazodone Hydrochloride with Cu(II) and Zn(II) metal ions. The stoichiometry of these complexes were also determined by using mole ratio method of Cu(II)-Vilazodone Hydrochloride and Zn(II)-Vilazodone. Hydrochloride were calculated as 1:1. Vilazodone Hydrochloride's recovery was 97.1%. Lambert-Beer's obeyed the concentration range of 2.40-12.0 µg/mL for Vilazodone Hydrochloride. The LOD and LOQ were found to be 2.93 and 13.67 µg/mL for Vilazodone Hydrochloride; respectively. The interaction between the Vilazodone Hydrochloride-Cu (II) and Vilazodone Hydrochloride-Zn (II) was investigated by applying this spectrophotometric method, and satisfactory results were obtained.

Keywords: *Vilazodone Hydrochloride, Metal complex, Mole ratio method, Stoichiometry*

INTRODUCTION

Vilazodone Hydrochloride's chemical name is 2-benzofurancarboxamide, 5- [4-[4-(5-cyano-1H-indol-3-yl) butyl]-1-piperazinyl]-hydrochloride (1:1) with molecular formula of $C_{26}H_{27}N_5O_2HCI$ and it has the molecular weight of 477.993 g/mole. The pKa of Vilazodone Hydrochloride is 7.1 and it is freely soluble in methanol [1,3]. It is a serotonergic antidepressant drug and used in the treatment of major depressive disorders in adults [4,6,8]. Vilazodone Hydrochloride acts as a potent serotonin reuptake inhibitor of (SSRI) and 5-hidroksitriptamin (5-HT1a) receptor partial antagonist which contains indole piperazine [2,5,7]. Literature review has described some analytical method for Vilazodone Hydrochloride but there has been no study on the metal complex stoichiometry formed for this drug with Cu(II) and Zn(II) metal ions. Therefore, the aim of this work was to develop a simple, fast, accurate and precise UV-Vis spectrophotometric method to determine the stoichiometric ratios of complexes formed between Vilazodone Hydrochloride with Cu(II) and Zn(II) metal ions.

EXPERIMENTAL

Reagents and chemicals

All the materials used were analytical grade. Pure standard of Vilazodone Hydrochloride was purchased from Sigma-Aldrich Chemical Co. (USA). Copper(II)nitrate trihydrate, zinc(II)nitrate hexahydrate and methanol reagent were obtained from Merck Chemical (Germany).

Apparatus

In this study, UV-2550 double-beam UV/Vis spectrophotometer and 1.0 cm quartz cuvvets (Germany) were used for the absorbance measurements.

Preparation of standard stock solutions

Vilazodone Hydrochloride ($8.40x10^{-4}$ M): 0.0100 g of Vilazodone Hydrochloride was taken in to 50 mL volumetric flask and dissolved in 25 mL methanol, and the volume was made up to 50 mL distilled water.

$Cu(NO_3)_2.3H_2O$ ($5.0x10^{-3}$ M): Copper (II) nitrate trihydrate was prepared by dissolving 0.0640 g of copper(II)nitrate trihydrate in 50 mL mixture of methanol-water (50:50, v/v).

$Zn(NO_3)_2.6H_2O$ ($5.0x10^{-3}$ M): Zinc(II)nitrate hexahydrate was prepared by dissolving 0.0744 g of zinc(II) nitrate hexahydrate in 50 mL mixture of methanol-water (50:50, v/v).

METHOD

Selection of analytical wavelength of Vilazodone Hydrochloride

For selection of analytical wavelength, working solution of Vilazodone Hydrochloride ($2.5x10^{-5}$ M) was scanned between 400 to 190 nm. The overlay spectrum of Vilazodone Hydrochloride was recorded.

Selection of analytical wavelength of Cu (II)-Vilazodone Hydrochloride

A number of solutions were made by mixing different volumes of $2.5x10^{-5}$ M solution of each of the copper(II)nitrate trihydrate and $2.5x10^{-5}$ M of Vilazodone Hydrochloride. The absorbance was scanned between 400 to 190 nm to determine their maximum wavelength using UV-2550 double-beam UV-visible spectrophotometer. The wavelength of the maximum absorbance was recorded.

Selection of analytical wavelength of Zn (II)-Vilazodone Hydrochloride

Similarly, the absorbance of working solutions of Zn(II)-Vilazodone Hydrochloride complex was scanned between 400 to 190 nm to determine their maximum wavelength. The maximum wavelength for Zn (II)-Vilazodone Hydrochloride complex was recorded.

Determination of complex stoichiometry

The stoichiometry of Cu(II)-Vilazodone Hydrochloride and Zn(II)-Vilazodone Hydrochloride complexes was determined by using mole ratio method. In this method, a series of standard solution which were the mole ratios of Cu(II)-Vilazodone Hydrochloride and Zn(II)-Vilazodone Hydrochloride complexes were prepared between 0-2.5 in test tubes, separately. Then, each solution was mixed in vortex, and their absorbance of maximum wavelength was measured on UV spectrophotometer, and a graphic was drawn using the values between ligand-metal mole ratio with absorbance.

Validation parameters

Linearity and calibration curve

For linearity and calibration curve, working solutions were prepared at 2.4, 4.8, 7.2, 9.6 and 12.0 µg/mL different concentrations, respectively. The absorption spectrums of solutions were scanned on spectrophotometer in the UV range of 400 to 190 nm, and their absorbance was recorded. A calibration curve was plotted with the absorbance against concentration.

Precision

Precision is the measure of how close the data values are to each other for a number of measurements under the same analytical conditions. The precision of analytical method, intra-day and inter-day measurements were determined. Intra-day precision was determined by analyzing 3.5 µg/mL, 5.5 µg/mL, 8.5 µg/mL and 10.5 µg/mL of Vilazodone Hydrochloride for three times within the day. Similarly, inter-day precision was determined by analyzing above mentioned concentrations for three consecutive days. Using the obtained result, the average (x), standard deviation (s), percentage relative standard deviation (RSD %), and relative error (RE %) were calculated.

Accuracy

Accuracy is expressed as the degree of closeness of experimental value to the true value. This parameter was evaluated by percent recovery studies at concentration level of 100 % which included the addition of known amounts of Vilazodone Hydrochloride working standard to a pre-quantified sample solution. All measurements were repeated three times and the results were calculated using the appropriate regression equation.

LOD and LOQ

Limit of quantification (LOQ) and limit of detection (LOD) were calculated using the following formula: LOD=C+3s and LOQ=C+10s (C: concentration, s: standard deviation).

RESULTS AND DISCUSSION

The proposed method was based on spectrophotometric and mole ratio method determination of Vilazodone Hydrochloride with metal complexes in UV area using the mixture of methanol and water (50:50, v/v) as a solvent. The absorption spectrums of Vilazodone Hydrochloride, Cu(II)-Vilazodone Hydrochloride and Zn(II)-Vilazodone Hydrochloride were found in the range of 190-400 nm. The curves between the absorbance and the wavelength have been shown in figure 1, figure 2 and figure 3.

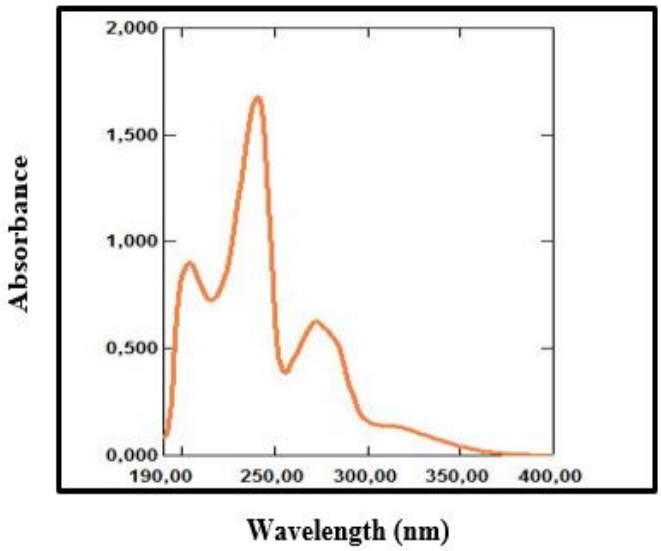

Fig. 1. The spectrum of Vilazodone Hydrochloride

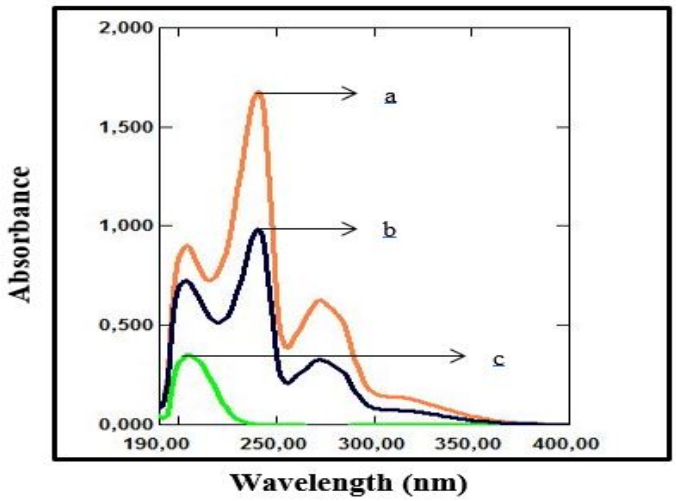

Fig. 2. a) Vilazodone Hydrochloride (2,5x10^{-5} M), b) Mixture of Vilazodone Hydrochloride-Cu (II) (2,5x10^{-5} M), c) Cu (II) (2.5x10^{-5} M) spectrums.

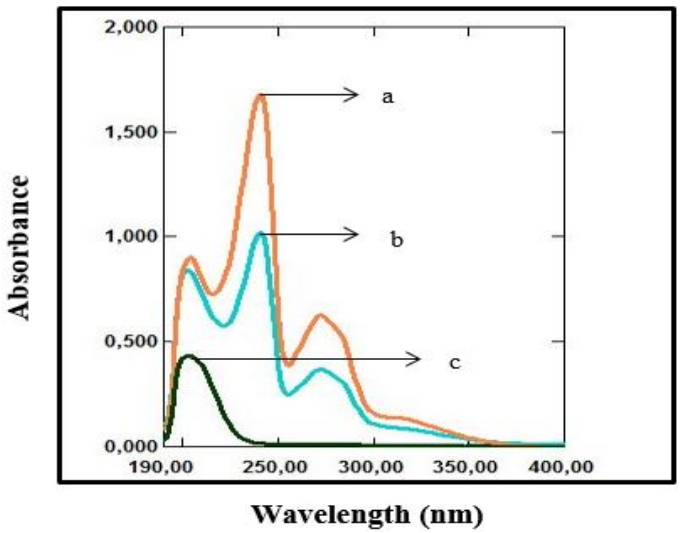

Fig. 3. a) Vilazodone Hydrochloride (2,5x10^{-5} M), b) Mixture of Vilazodone Hydrochloride-Zn (II) (2,5x10^{-5} M), c) Zn (II) (2.5x10^{-5}M) spectrums.

Lambert-Beer obeyed in the concentration range of 2.93-13.67 µg/mL for VLZ Hydrochloride (Fig. 4 and 5). The correlation coefficient (r^2) value was found 0.9994 which showed that the absorbance of the drug was linear with the concentration. The visual characteristics such as linearity range, standard deviation on intercept and slope, correlation coefficient and regression linear equation were calculated, and have been summarized in Table 1.

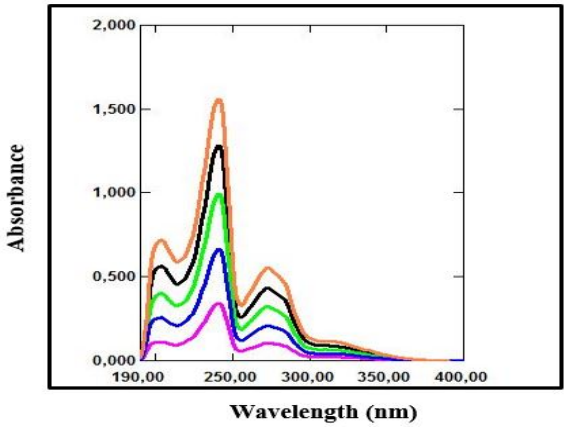

Fig. 4. Absorption spectras of five different concentrations (2.4-12.0 µg/mL) of Vilazodone Hydrochloride

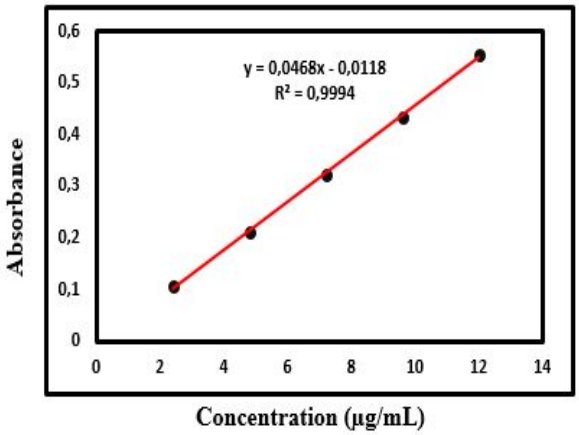

Fig. 5. Linearity plot of Vilazodone Hydrochloride

Table 1. Statistical results of Vilazodone Hydrochloride's calibration curve

Statistical parameters	Result
Wavelength	204 nm 240 nm 272 nm
Linearity range	2.4-12.0 µg/mL
Regression linear equation	y=0.0468x-0.0118
r^2: Correlation coefficient	0.0994
S_a: Standard deviation on intercept	0.41
S_b: Standard deviation on slope	0.027

The LOD and LOQ were found to be 2.21 µg/mL and 10.9 µg/mL for Vilazodone Hydrochloride; respectively. The mean ($\bar{x}$), standard deviation (s), percentage relative error (RE %) and relative standard deviation (RSD %) were calculated according to the result of intra-day and inter-day precision measurements, and have been summarized in Table 2. The RSD % values for intra-day analysis were found 0.8, 0.19, 0.23 and 0.52, but the RSD % values for inter-day analysis were found 0.23, 0.27, 0.53, and 0.55 for Vilazodone Hydrochloride, respectively (Table 2). As a result, the precision of the analytical method was further substantiating.

Table 2. Intra-day and inter-day precision measurements results.

Intra-day measurement values					**Inter-day measurement values**			
Taken µg/mL	**Found µg/mL**	**$\bar{x}$±s µg/mL**	**RE %**	**RSD %**	**Found µg/mL**	**$\bar{x}$±s µg/mL**	**RE %**	**RSD %**
3.5	3.82 3.80 3.77	3.80±0.03	8.3	0.8	3.97 3.78 3.82	3.8±0.02	8.6	0.53
5.5	5.38 5.38 5.40	5.40±0.01	-1.82	0.19	5.42 5.42 5.44	5.41±0.03	-1.64	0.55
8.5	8.65 8.63 8.67	8.65±0.02	1.76	0.23	8.75 8.71 8.74	8.73±0.03	2.71	0.23
10.5	10.81 10.79 10.83	10.81±0.02	2.95	0.52	10.91 10.94 10.89	10.91±0.03	3.9	0.27
$\bar{x}$: Mean, s: Standard deviation, RE: Relative error, RSD: Relative standard deviation								

The recovery was found 97.1 % for VLZ Hydrochloride. The result of recovery study has been shown in table 3.

Table 3. Recovery study of Vilazodone Hydrochloride

100%	**Known amounts of Vilazodone Hydrochloride (mg)**	**Found amounts of Vilazodone Hydrochloride (mg)**	**Recovery %**
1	10	9.80	98.0
2	10	9.59	95.9
3	10	9.73	97.3
The mean recovery %	10	9.71	97.1

The wavelengths of maximum absorption spectrum of Vilazodone Hydrochloride and metal complexes with Cu(II) and Zn(II) were determined. The stoichiometry's of Cu (II)-Vilazodone Hydrochloride and Zn(II)-Vilazodone Hydrochloride complexes were calculated as 1:1 (Fig.6 and 7).

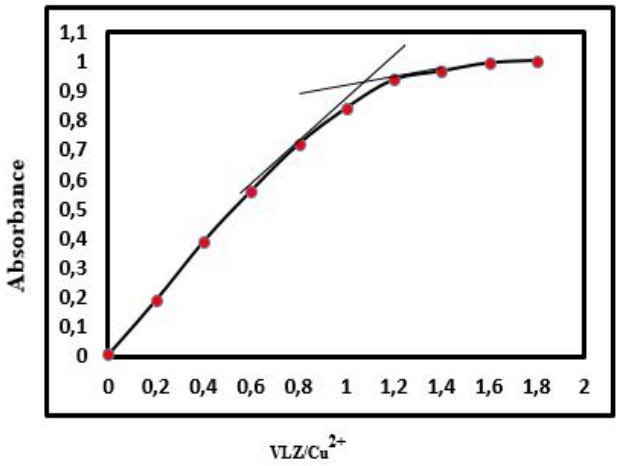

Fig 6. Determination of composition of Cu(II)-Vilazodone Hydrochloride complex by mole ratio method

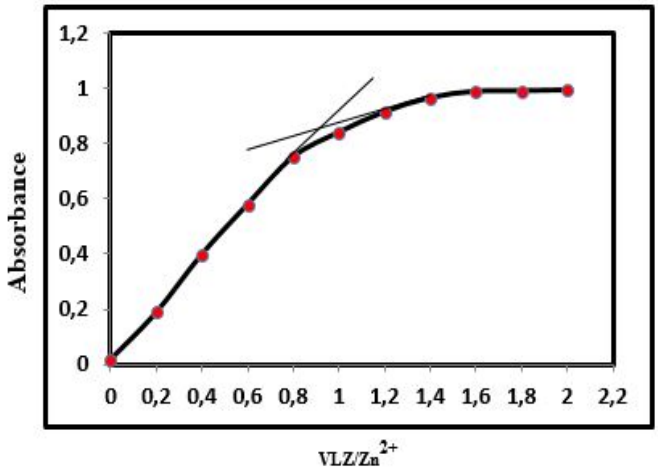

Fig 7. Determination of composition of Zn(II)-Vilazodone Hydrochloride complex by mole ratio method

CONCLUSIONS

For all the values, low relative errors (RE< ±15 %), high correlation coefficient (r^2=0,0994) and high percentage recovery (97.1%) showed the high linear relationship between the predicted and actual concentrations. The precision (intra-day and inter-day) of analytical method was found within limits (RSD < 2 %). The proposed spectrophotometric method was found to be simple, accurate and precise.

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