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New validated method for analysis of salymarin in polyherbal formulation (aqueous extract, oral liquid and solid dosage form)

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A B S T R A C T

A UV/visible Spectrophotometric method for the determination of Silymarin in poly herbal formulation have been developed. Developed method is based on the simple concept of solubility of chemical i.e. Silymarin in methanol. This method is simple, rapid, accurate, sensitive and precise for the determination of Silymarin in polyherbal formulation like aqueous extract, Oral liquid (i.e. Syrup) and solid (i.e. Capsules) dosage form containing crude herb *Silybum marianum*. The λ_{max}. of Silymarin measured at 287nm. The molecule (Silymarin) obeys Beer's law in between range of 0.3 – 1.6 mg/ml. The method is reproducible, accurate and linear for routine quality control testing of phyto-pharmaceutical dosage forms.

Capsule Summary: Proposed method is potential method for the utilization in analysis of commercial batches of poly herbal formulation (Aqueous extract, oral liquid and solid dosage form).

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INTRODUCTION

Silymarin is hepatoprotective flavonoid drug available as bio marker in Silybun marianum (common name Milk thistle). In British pharmacopoeia individual monograph for Milk Thistle Fruit is available with claim of silymarin, expressed as silibinin ($C_{25}H_{22}O_{10}$; MW 482.4) (British Pharmacopoeia, 2013)

Silymarin is being used in treatment of various liver disease of different etiology because of its hepatoprotective action (Cavalieri, 1974; Salmi et al., 1982; Bosisio, 1992; Ferenci, 1992).

There are different analytical test method are available in pharmacopoeia or other source of information. HPLC method is available in British Pharmacopoeia 2013 on gradient system and other HPLC methods also available (Radijabian et al., 2008; Kvasnicka et al., 2003; Lee et al., 2007; Hadad et al., 2009).
Method on other analytical techniques are also available like thin layer chromatography and UV spectrophotometer (Abdel-Salam et al., 1982; Vanhaelen and Vanhaelen-Fastre, 1983; Dube and Vyas, 2009).

Still little work has been done specially on simple analytical technique like UV spectrophotometry and no method available for poly herbal extract and phyto-pharmaceuticals.

EXPERIMENTAL

Reagent

Silymarin standard

Table 1: Calibration data and linearity

Concentration (mg/mL)	Absorbance ± SD
0.3	0.2075 ± 0.0001
0.4	0.2937 ± 0.0005
0.5	0.3642 ± 0.0003
0.6	0.4614 ± 0.0003
0.7	0.5277 ± 0.0003
1	0.7108 ± 0.0001

Table 2: LOD and LOQ results

Concentration Range	LOD (mg/mL)	LOQ (mg/mL)
0.3 – 1 g	0.015	0.06

Table 3: Inter day precision results of day 1

Concentration (mg/mL)	Absorbance ± SD
0.3	0.2075 ± 0.0001
0.4	0.2937 ± 0.0005
0.5	0.3642 ± 0.0003
0.6	0.4614 ± 0.0003
0.7	0.5277 ± 0.0003
1	0.7108 ± 0.0001

Table 4: Inter day precision results of day 2

Concentration (mg/mL)	Absorbance ± SD
0.3	0.1975 ± 0.0001
0.4	0.2836 ± 0.0003
0.5	0.3541 ± 0.0002
0.6	0.4513 ± 0.0001
0.7	0.5175 ± 0.0001
1	0.7008 ± 0.0001

Table 5: Results of commercial products

Products	Results
Extract	99.80%
Bonjigar Capsule	101.00%
Bonjigar Syrup	100.70%

Methanol
Chloroform
Polyherbal Aqueous Extract B # E14 001
Oral Liquid: Bonjigar Syrup B # 1314 001
Oral Solid: Bonjigar Capsule B # 1414 001

Instrumentation

UV Spectrophotometer: Perkin Elmer Lamba 25

Experiment conditions

Wavelength: 287 nm

Preparation of standard and sample solution

A 100 mg of standard were accurately weight and transfer into 100 mL volumetric flask, dissolve in methanol and make up the volume with methanol and mix well.

Total 6 volumetric flasks of 100 mL were taken and marked with 1, 2, …..,6, transfer 0.3, 0.4, 0.5, 0.6 and 0.7 mL of standard to volumetric flasks through pipette and make up the volume with methanol and mix well.

Preparation of sample solution

Extract

Accurately weight and transfer 200mg of extract in separating funnel, mix with 20ml water, extract the content with using 50ml chloroform. Collect the aliquot in round bottom flask through filter paper containing sodium phosphate. Repeat the same procedure 3 times. Evaporate the combine filtrate on water bath. Dissolve the residue in 10ml methanol and transfer to 50ml volumetric flask quantatively. Make –up the volume with methanol and mix well.

Oral liquid

Accurately transfer 10ml of syrup in separating funnel, mix with 20ml water, and extract the content with using 50ml chloroform. Collect the aliquot in round bottom flask through filter paper containing sodium phosphate. Repeat the same procedure 3 times. Evaporate the combine filtrate on water bath to dryness. Dissolve the residue in 10ml methanol and transfer to 50ml volumetric flask quantatively. Make –up the volume with methanol and mix well.

Oral solid

Accurately transfer content of capsule in separating funnel, mix with 20ml water, and extract the content with using 50ml chloroform. Collect the aliquot in round bottom flask through filter paper containing sodium phosphate. Repeat the same procedure 3 times. Evaporate the combine filtrate on water bath to dryness. Dissolve the residue in 10ml methanol and transfer to 50ml volumetric flask quantatively. Make –up the volume with methanol and mix well.

RESULT AND DISCUSSION

Method development and optimization

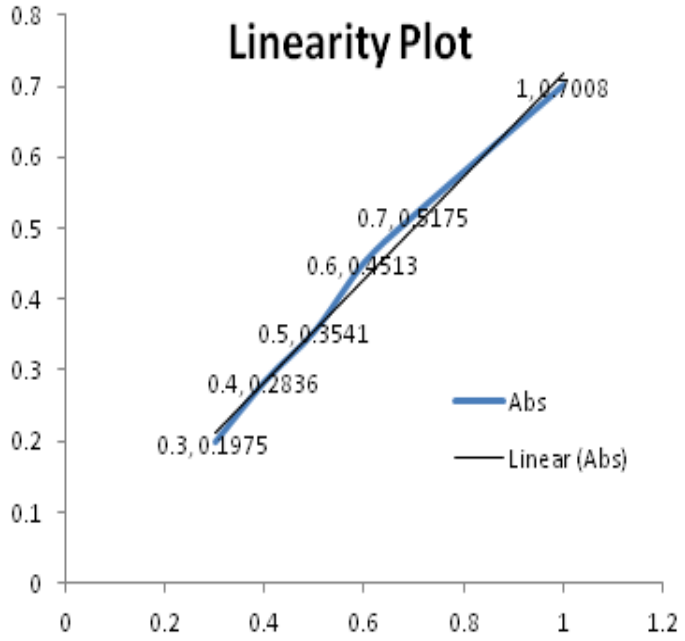

Fig. 1: Linearity plot

Standard and sample solutions were separately scan in the range of 200–400 nm to measure the absorbance maximum of standard and sample. Both samples showed absorbance maximum at 287nm. Placebo sample was also run in the same range and no peak observed at λ_{max} 287 nm.

Calibration data and linearity

Readings of 6 dilutions were taken. Results are summarized in Table 1.

Specificity/selectivity

Method specificity or selectivity was performed by running placebo samples with standard. There is no peak at 287nm.

Limit of detection (LOD) and Limit of quantification (LOQ)

The limit of detection (LOD) and limit of quantification (LOQ) of this method were determined from the known concentrations of Silymarin. The LOD and LOQ for this analysis were calculated from using three and ten times the noise level of the response, respectively. Results are given in Table 2.

Precision (Inter-day)

Same samples were kept at ambient room temperature and again readings were taken and results are shown in Table 3 and 4.

Application of method on poly herbal formulation

Samples of commercial batches were tested using this method. Results are shown in Table 5.

The method was developed and optimized by scanning on UV spectrophotometer in range of 200 – 400nm. Data showed that method is reproducible and precise.

The method selectivity/specificity was performed by scanning standard, sample and placebo and no peak found in placebo at λ_{max} i.e. 287nm.

Test for Linearity was performed in the range 0.3 – 1 mg mL -1 of Silymarin with a correlation coefficient ± 0.99. Results reveal that method is linear for the range of assay i.e. 80 – 120% (Fig. 1).

Method precision (inter-day) was checked by performing test on same samples and system on next day and results RSD found less than 2%.

A commercial batch of poly herbal extract, product Bonjigar Syrup and Bonjigar capsules was randomly selected for analysis and method was applied to quantify the content of Silymarin in product.

CONCLUSION

A simple, rapid, sensitive and reliable UV Spectrophotometric method for analysis of the Silymarin has been developed and method validity verified by performing linearity, and inter-day precision. Proposed method is potential method for the utilization in analysis of commercial batches of poly herbal formulation (Aqueous extract, oral liquid & solid dosage form).

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