A comparative study on different market samples and standard samples of Shalaparni through physicochemical methods and near infrared spectroscopy

Parth Raval¹, B.R.Patel², V.J.Shukla³, Bhavesh Patil⁴, Nehaparmar⁴, Preeti Pandya⁵, Shivangi Bhardwaj⁶

¹ M.Pharm(Ayu) Scholar, IPGT&RA, Gujarat Ayurved University, Jamnagar, India
² Assistant professor, Department of Dravyaguna, IPGT&RA, Gujarat Ayurved University, Jamnagar, India
³ Head, Pharmaceutical chemistry laboratory, IPGT&RA, Gujarat Ayurved University, Jamnagar, India
⁴ Ph.D. Scholar, Department of Dravyaguna, IPGT&RA, Gujarat Ayurved University, Jamnagar, India
⁵ Laboratory Assistant, Department of Dravyaguna, IPGT&RA, Gujarat Ayurved University, Jamnagar, India
⁶ Ph.D. Scholar, Pharmaceutical chemistry laboratory, IPGT&RA, Gujarat Ayurved University, Jamnagar, India

ABSTRACT
Dashmoolais one of the most important groups explained in Mishrakagana. One of which, Shalaparni is a potent drug used single as well as in various formulations mentioned in classics. Adulteration in Dashmoolaplants is a very big issue now days and this is because of the lack of availability of the original drugs. In this study market samples of Shalaparni (Desmodium gangeticum DC.) collected from the different part of India; compared with the standard Shalaparni authenticated sample which was collected from the natural source; by using physicochemical parameter and near infrared spectroscopy. Results were statistically processed by PCA. The results show that there is no similarity found outbetween the standard drug and market samples of Shalaparni which were collected from different regions of India. The market samples were observed for different adulterated material having poor quality.

Introduction
In Ayurvedic literature, more than 700 plant drugs have been mentioned. The scientific descriptions about the plant drugs were found in Brihattrayi and Laghu trayi. Dashmoolais one of the most important groups, explained in Mishrakagana[1]. The drugs of Dashmoolae are Bilva, Agnimantha, Shyonak, Patala, Gambhari, Shalaparni, Prishniparni, Brihati, Kantakariand Gokshura. One of which, Shalaparnnis a potent drug used single as well as in various formulations mentioned in classics. Unlike in the olden days when physicians themselves used to collect the herbs, prepare and administer the medicine, but now a days the newer generation of Ayurvedic physicians are using prepared drugs available in the market. As a result, professional plant collectors have taken over the floor and the industry is forced to accept the herbs they bring on their terms without questioning. Herb collectors, who are unable to meet increasing demand on their part, adulterate the drugs with other plants & spurioussubstances[1]. Physicochemical analysis provides the objective parameters to set the standards for quality of raw drugs as well as finished products. With the help of analytical studies, it is possible to standardize the drug and differentiate the adulterants.

NIR spectroscopy is a well-established nondestructive technique in the food, chemical, agrochemical and petrochemical industry, for qualification of incoming raw material and has now also been used for many years in the pharmaceutical industry[1]. The technique appears to be useful for the identification and assay of pharmaceutical substances, the identification and assay of such substances in the finished products, as well as for in-process control and for monitoring purposes. Near infrared spectroscopy is a nondestructive type of analysis method which can be useful to illustrate the concept of drug as whole[1]. PCA (principal component analysis) is probably the oldest and best known of the techniques used for multivariate analysis.
The overall goal of PCA is to reduce the dimensionality of a data set, with simultaneously retaining the information present in the data. Dimensionality reduction or data comparison is possible with PCA because chemical data sets are often redundant.

The present work had been carried out to compare the market samples of \textit{Shalaparni} which were collected from different regional markets of all over India with standard collected samples of \textit{Shalaparni} (\textit{Desmodium gangeticum} DC. – official botanical source as per API) by using physicochemical parameter and near infra-red spectroscopy. Root and whole plant of \textit{Shalaparni} were taken separately as standards for this study[2].

**Material and method**

**Collection of Sample**

Crude market samples were collected from various markets sold in the name of \textit{Shalaparni} and standard authenticate samples of root and whole plant of \textit{Shalaparni} were collected from its natural source at the time of \textit{Sharadritu} on 5\textsuperscript{th} October 2013. Standard and market samples were as follows[2].

- Standard sample 1 (S1- \textit{Shalaparni} root)
- Standard sample 2 (S2- \textit{Shalaparni} whole plant)
- Market sample 1 (M1-North India)
- Market sample 2 (M2-East India)
- Market sample 3 (M3-South India)
- Market sample 4 (M4-West India)[2-3].

**Physico chemical analysis**

In physical evaluation, moisture content, total ash, acid insoluble ash and extractive values viz., alcohol and watersoluble extractive values were determined[2].

**Near Infra-Red Spectroscopy**

NIR spectroscopy was done at SICART laboratory, Anand, Gujarat.

**Instrument model**

<table>
<thead>
<tr>
<th>No</th>
<th>Physico-chemical parameter</th>
<th>S\textsubscript{1}</th>
<th>S\textsubscript{2}</th>
<th>M\textsubscript{1}</th>
<th>M\textsubscript{2}</th>
<th>M\textsubscript{3}</th>
<th>M\textsubscript{4}</th>
<th>API\textsuperscript{a}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1.</td>
<td>Loss on drying (% w/w)</td>
<td>5.59</td>
<td>6.94</td>
<td>4.29</td>
<td>6.24</td>
<td>3.46</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2.</td>
<td>Ash value (% w/w)</td>
<td>6.6</td>
<td>7.4</td>
<td>15.5</td>
<td>21.8</td>
<td>19.8</td>
<td>16.4</td>
<td>&lt;7</td>
</tr>
<tr>
<td>3.</td>
<td>Acid in soluble ash (% w/w)</td>
<td>1.7</td>
<td>2.5</td>
<td>7.8</td>
<td>6.5</td>
<td>4.3</td>
<td>8.4</td>
<td>&lt;2</td>
</tr>
<tr>
<td>4.</td>
<td>Water soluble extractive (% w/w)</td>
<td>6.8</td>
<td>8.56</td>
<td>5.6</td>
<td>7.2</td>
<td>2</td>
<td>3.6</td>
<td>&gt;6</td>
</tr>
<tr>
<td>5.</td>
<td>Alcohol soluble extractive value (% w/w)</td>
<td>3.92</td>
<td>4.64</td>
<td>2.4</td>
<td>2.4</td>
<td>9.6</td>
<td>1.6</td>
<td>&gt;1</td>
</tr>
<tr>
<td>6.</td>
<td>pH</td>
<td>6</td>
<td>6</td>
<td>5.5</td>
<td>6.5</td>
<td>6.5</td>
<td>7</td>
<td>-</td>
</tr>
</tbody>
</table>

**Methods**

**Powder pretreatment:** The powder of the drug samples was dried to make it moisture free and then sieved to make it micro fine. Then it was spread in Barium chloride coated slit and the sample was run to obtain the reflectance.

**Data treatment (Principal component analysis)**

PCA was done on results of physicochemical parameters by using Portable Unscrambler-X software. The NIR spectra of the samples were recorded and the data was obtained in the form of % Reflectance at the interval of every 1 units of wavelength in nm. By using these data points, \(\lambda\) (Wavelength) vs. % reflectance graph was plotted by using Portable Unscrambler-X software and PCA was applied as a data projection method and the samples were compared with this parameters[2].

**Results and discussion**

**A. Physicochemical analysis:** Results of Physicochemical parameters are given below

- Perkin Elmer Lambda 19 UV/Vis/NIR spectrophotometer[2].

**Instrumental Specifications**

- Double beam, Double monochromator, Ratio recording
- Lamp: Deuterium (UV), Tungsten-Halogen (VIS/NIR), Lead Sulphide cell (PbS) for NIR
- Wavelength range: 185-3200 nm for Absorbance/Transmission and 200-2500 for Reflectance
- Scan speed: 0.3 to 1200 nm/min
- Wavelength accuracy: ±0.15nm for UV/Vis and ± 0.6 nm for NIR
- Baseline flatness: ± 0.001 A0, 4nm slit
- Ordinate mode: Scan, Time drive, Wavelength programming, concentration[2].
Loss on drying

Values mentioned in Table no. 1 indicate that sample $M_4$ has less moisture content than standards; other market samples were nearly similar to standard one. LOD value shows percentage of moisture which directly affects the preservation of the material. Higher percentage of the moisture leads to degradation of metabolites, leading deterioration of the quality of drug. So the LOD of the sample was used as one of the parameter for analysis[3-4].

Total and acid in soluble ash

Values mentioned in Table no. 1 indicate that the inorganic materials are very common in all market samples as compared to standard samples. It indicates that the silica and mud were very common in all market samples as compared to standard samples which might be due to poor collection, storage & preservation by vendor. Presence of inorganic and salt materials like sand, dust particles etc. in a sample will lead to increase in its ash content. It also depends on the nature i.e. plant may be consuming more amount of salts and mineral from the soil. Majority of medicinal plants are collected from wild sources and other post harvesting processing like drying etc. are carried out by unskilled persons. So if proper technical guidance is not given during harvesting and post harvesting process, the chances of inclusion of unwanted material like sand, dust particles etc. are quite high[5-6].

Extractive value

Values of WSE & ASE mentioned in Table no. 1 indicate that values for all market samples were not within limits while those for both standard samples were within limits of API. An extractive value of medicinal plant is related to its phytochemical constituents and sometimes it shows wide variation from plant to plant. Water soluble and alcohol soluble extractive values are indicative of the solubility of active principles of the plant in respective solvent[7-9].

Principle component analysis (PCA)

All physicochemical results were put in Principle component analysis (PCA) under consideration of all variables. PCA showed marked difference between standard samples ($S_1$, $S_2$) and market samples ($M_1$, $M_2$, $M_3$, $M_4$). In PCA, none of the market samples were matched with standard samples; whereas both standards samples showed similarity in statistical analysis (Fig no. 1)[10-11].

Near Infra-Red Spectroscopy

Here 200 nm to 2500 nm spectral scan was done and results of NIR spectroscopy were put in Portable Unscrambler-X software by data points, $\lambda$ (Wavelength) vs. % reflectance graph was taken plotted in graphical comparison of standard samples with each market samples. First comparison done on both standard sample $S_1$ and $S_2$ to check their internal similarity between root and whole plant mentioned in Fig. no.1. While other comparison showed marked difference between standard samples ($S_1$, $S_2$) and market samples ($M_1$, $M_2$, $M_3$ and $M_4$) respectively in Fig. no. 2, 3, 4, 5 and 6. A statistical analysis (PCA) was done on results of NIR spectroscopic data. Here marked difference was found between standard samples ($S_1$, $S_2$) and market samples ($M_1$, $M_2$, $M_3$ and $M_4$) respectively in Fig. no.7[12-16].

Fig no.2: Graph plotting between sample $S_1$ and $S_2$-λ (Wavelength) vs. % reflectance

Fig no.3: Graph plotting between sample $S_1$, $S_2$ and $M_1$-λ (Wavelength) vs. % reflectance

Fig no.4: Graph plotting between sample $S_1$, $S_2$ and $M_2$-λ (Wavelength) vs. % reflectance

Fig no.5: Graph plotting between sample $S_1$, $S_2$ and $M_3$-λ (Wavelength) vs. % reflectance
Conclusion

The results of physicochemical study concluded that all market samples (M₁, M₂, M₃, and M₄) were not found within the standard range and with remarkable variations when compared with standard samples S₁, S₂ and API monograph of Shalaparni. This indicates poor collection, storage, preservation and admixture of another herb. In principle component analysis of physico chemical results and NIR spectroscopic values clearly indicate that all market samples of Shalaparni did not match with standard samples while both standard samples were nearly similar. Hence it is concluded that all market samples M₁, M₂, M₃ and M₄ of Shalaparni showed different material when compared to standard collected samples S₁ and S₂; which clearly revealed that different plant materials are being sold as source plant of Shalaparni in Indian market.

Conflict of interest statement
We declare that we have no conflict of interest.

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