

Formulation and Standardization of *Shatsakar Churna*: Quality Control Studies for Polyherbomineral Ayurvedic Formulation

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ABSTRACT

Ayurvedic formulations are highly effective and known to have minimal or no side effect, but due to lack of validation parameters for identification and quality control, there is a lacuna in demand of Ayurvedic formulations at international level. *Shatsakar Churna* is a widely used polyherbal Ayurvedic formulation for constipation and gastric troubles. Standardization and quality control parameters of *Shatsakar Churna*, are not well defined. The objective of the work is to formulate and standardize the *Satsakar Churna* according to World Health Organization (WHO), GMP guidelines which are the first available report so far. The formulation was prepared in the Institute's pharmacy and evaluated for organoleptic characters, powder drug studies, physicochemical parameters (total ash, acid insoluble ash, water soluble ash, water soluble extractive, ethanol soluble extractive), micrometric evaluation (density and flow properties) and phytochemical evaluations. The results of different standardization parameters revealed satisfactory and sufficient data to evaluate the in-house formulation and can be utilized as reference standards in various quality control aspects of the formulation, powder drug analysis revealed specific identities for crude raw drug which are useful as marker in the preparation and identification of components of the formulation.

Keywords: Herbomineral, Hyperchlorhydria, Kapha, *Shatsakar Churna*, Standardization

INTRODUCTION

Shatsakar Churna, an Ayurvedic polyherbomineral formulation, consists of different parts of different species viz. leaf of *Cassia senna* L. (Syn.-*Cassia angustifolia* Vahl.), fruits of *Foeniculum vulgare* Mill., fruits of *Terminalia chebula* Retz., rhizomes of *Zingiber officinale* Rosc. A unique feature of this *Churna* is the presence of minerals *Saindhava lavana* (rock salt) and *Sauvarchal lavana* (black salt). The *churna* is helpful in managing diseases due to the vitiation of *Kapha Dosha*, which is among one of the Tridasha described in Ayurveda. The *churna* improves digestion and ensures timely evacuation, improves functioning of liver, cures hyperacidity, heart burn and acidic belching. Majority of Ayurvedic formulations use whole plants either alone or in combinations therefore the efficacy of the Ayurvedic formulation may vary with the use of the adulterants in the formulations. It is therefore important to establish characteristics of the raw material and finished Ayurvedic products with the help of physical and chemical methods. Now a days, majority of the world population is turning toward the alternative system of medicine because of complexity and associated adverse effects with the usage of allopathic medicines. According to *Bhaishajaya Ratnavali*^{1, 2}, *Shatsakar churna* is composed of rock salt, black salt and four herbs, but there is not even a single standard mentioned for ensuring the identity, potency, purity, safety and efficacy of the *Shatsakar churna*.

The paper deals with the formulation and quality control evaluation of the important Ayurvedic formulation. The study is an attempt to evaluate the organoleptic characters, powder drug analysis, physicochemical parameters, micrometric evaluation and phytochemical evaluation as per the Ayurvedic Pharmacopoeia of India and WHO guidelines for ensuring the identity, potency, purity, safety and efficacy of the *Shatsakar churna*³. After an extensive literature search it was found to the best of our knowledge that this is the first report revealing the formulation and evaluation of this important Ayurvedic preparation.

METHODOLOGY

Procurement of plant material

Cassia angustifolia Vahl (Leaf), *Foeniculum vulgare* Mill. (Fruit), *Terminalia chebula* Retz. (Fruit), *Zingiber officinale* Rosc. (Rhizome), *Saindhava lavana* and *Sauvarchal lavana* used in *Shatsakar churna* were collected from local market of Patiala (Punjab). All the ingredients were macroscopically identified by Pharmacognosy section of the Institute and organoleptic evaluation was made for identification of sensory characteristics like colour, odour, taste, size, texture and fracture^{4-5, 7-17}. The plant material was cleaned by sorting out using a cloth duster to remove dust and air blowing to remove minute sand particles. The ingredient of the formulation was individually treated with water containing the antimicrobial agents (0.1% Potassium meta-bisulphite

Table 1: Formula of ayurvedic formulation *Shatsakar churna* (Bhaishajya Ratnavali)

Sr. No.	Botanical Name	Name of the <i>rasayana</i>	Part used	Quantity /10 g
1	<i>Cassia senna</i> Linn.	<i>Svarna Patri</i> (Sanaya)	Leaf	4.0g
2	<i>Zingiber officinale</i> Rosc.	<i>Shunthi</i>	Rhizome	1.0g
3	<i>Foeniculum vulgare</i> Mill.	<i>Saunf</i> (Misreya)	Fruit	1.0g
4	Rock salt	<i>Saindhava lavana</i>	-	1.0g
5	Black Salt	<i>Sauvarchal lavana</i>	-	1.0g
6	<i>Terminalia chebula</i> Retz.	<i>Shiva-Haritaki</i>	Fruit	2.0 g

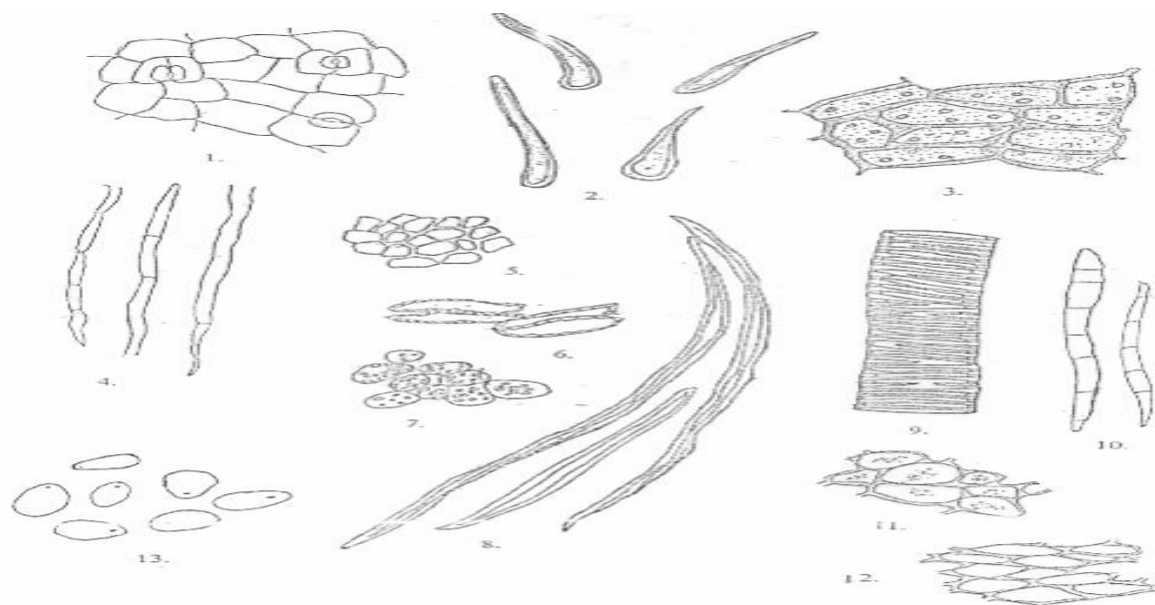


Figure 1: Powder Drugs analysis of Shatsakar Churna: 1. Paracytic stomata X 440; 2. Covering Trichomes X 440; (*Cassia senna* Linn.); 3. Endosperm cells X 864; 4. Vittae X 864 (*Foeniculum vulgare* Mill.); 5. Epidemis cells X 440; 6. Stone cells X 440; 7. Parenchyma cells with starch grains X 440; 8. Fibres X 440; (*Terminalia chebula* Retz.); 9. Reticulated vessels X 440; 10. Fibres X 440; 11. Cork cells X 216; 12. Cork cells with starch grains X 216; 13. Starch grains X 440 (*Zingiber officinale* Rosc.).

Table 2: Results of Physicochemical Parameters

S. No.	Name of Parameters	Results (Mean± SD)
1.	Loss on drying at 105 ⁰ C (% w/w)	9.27±0.54
2.	Total ash content (% w/w)	28.25±0.89
3.	Acid insoluble ash (% w/w)	3.54±0.93
4.	Water soluble ash (% w/w)	8.64±0.86
5.	Water soluble extractive value (% w/w)	51.45±1.01
6.	Ethanol soluble extractive value (% w/w)	29.74±0.34

Values are expressed as mean ± SD (standard deviation)

and 70% isopropyl alcohol).

Quantitative analysis and storage

Quantitative analysis of the raw material was done for standardization parameters including foreign organic matter, water soluble extractive, methanol soluble extractive total ash and acid insoluble ash. The approved raw material was packed in sterilized airtight polybags with proper labelling and stored in cool place¹⁸⁻²¹.

Formulation of the Shatsakar churna

The ingredients were individually pulverized and sieved (80 mesh) to obtain respective fine powders. Powder of each ingredient was weighed separately and thoroughly mixed together as per the quantity mentioned in Table 1.

The composite mixture was again sieved (80 mesh) to obtain a fine powder of the finished product i.e. *Shatsakar churna* for its better therapeutic value^{1,6,21}. The finished product thus obtained, was subjected to chemical treatment similar to that given to the raw material to inhibit microbial growth and dried at 60°C. *Shatsakar churna* obtained in powder form was packed in sterilized polythene pouches, labelled, coded and stored inside cool and dry place.

Powder drug analysis of Shatsakar churna²²⁻²⁶

The *Shatsakar Churna* was taken in three small quantities for powder drug analysis. The first part was warmed in chloral hydrate solution and washed. The second part of formulation powder was washed thoroughly with water.

Table 3: Results of Flow Property Analysis

Sr. No.	Parameters	Results \pm SD
1.	Bulk density (gm/cm^3)	0.434 \pm 0.0021
2.	True density (gm/cm^3)	0.714 \pm 0.005
3.	Angle of Repose ($^\circ$)	29.56 \pm 1.89
4.	Carr's index	39 \pm 0.98
5.	Hausner's ratio	1.64 \pm 0.002

Values are expressed as mean \pm (standard deviation)

Table 4: Phytochemical evaluation of aqueous extract of Shatskar churna

S. No.	Phyto-constituents	Results
1	Alkaloids	+++
2	Glycosides	+++
3	Saponins	+++
4	Flavonoids	+++
5	Carbohydrates	+++
6	Phenolics	+++
7	Essential oil	+++

+++ indicates the presence of the phytoconstituents

The third part of formulation powder was treated with iodine solution. All these samples were mounted in 50% glycerine separately for anatomical study and seen under Trinocular Microscope (Olympus).

Physicochemical Parameters⁶⁻⁸

Estimation of total ash

Powdered formulation (3 gm) was weighed accurately in a tarred silica crucible. It was incinerated by gradually increasing the temperature until free from carbon and cooled to room temperature. It was kept in desiccators and weighed. The percentage of total ash was calculated with reference to the air dried sample in triplicate.

Acid insoluble ash

The total ash obtained with above method was boiled for 5 minutes with 25 ml of dilute hydrochloric acid. The solution so obtained was filtered and the insoluble matter collected on ash less filter paper. The filter paper was washed with hot water and ignited in tarred crucible and cooled to room temperature and kept in desiccator. The residue was weighed and acid-insoluble ash of drug was calculated with reference to the air dried drug.

Water soluble ash

The ash of 3 gm of sample was obtained following the method as described above. It was boiled for 5 minutes with 25 ml of water. The insoluble material was collected on the ash less filter paper and ignited in tarred crucible for 15 minutes at a temperature not exceeding 450 $^\circ$ C, cooled and kept in desiccators. The weight of this residue was subtracted from the weight of the total ash. The water soluble ash of drug with reference to the air dried drug was calculated. The amount of water soluble ash was 8.64% in *Shatskar churna*.

Water soluble extractive value

100 ml of distilled water was added in 5 g of the formulation (glass stopper flask) and flasks were shaken occasionally for 6 hours and then allowed to stand for 18

hours. The extract was filtered and 25 ml of the filtrate was pipette out in a pre-weighed 100 ml beaker and evaporated to dryness on a water bath. It was kept in a hot air oven for 5 hours at 105 $^\circ$ C, cooled to room temperature, kept in desiccator for 30 minutes and weighed. The procedure was repeated till constant weight obtained.

Alcohol soluble extractive Value

100 ml of rectified spirit (ethanol) was added in 5 g of the formulation in a glass stopper flask and shaken occasionally for 6 hours. Then it was allowed to stand for 18 hours. The extract was filtered and 25 ml of the filtrate was pipette out in a pre-weighed 100 ml beaker. The filtrate was evaporated to dryness on a water bath. It was kept in a hot air oven for 5 hours at 105 $^\circ$ C, cooled to room temperature, kept in desiccator for 30 min. and weighed. The procedure was repeated till constant weight obtained.

Micrometric evaluation^{6,8,26}

Estimation of the density of formulation

Bulk density and tapped density were determined using tapping cylinder method. 10 gm formulation was filled in cylinder and the volume was measured. The final volume was measured after 100 tappings. The bulk and tapped densities were calculated and mentioned in Table no. 3

Flow properties

Angle of repose

Angle of repose has been used as an indirect method quantifying powder flow ability, because of its relationship with interparticle cohesion. The fixed funnel and the free standing cone method employs a method that is secured with its tip at a given height (H), above the glass paper that is placed on a flat horizontal surface. Powder or granules were carefully poured through the funnel until the apex of the conical pile just touch the tip of funnel. Thus, with R being the radius of the conical pile. Angle of repose was calculated using the formula $\tan \theta = h/r$. Where h is the height of the heap of powder r is the radius of the base of the heap powder and θ is the angle of repose. Static angle of repose was determined by funnel method. The weighed amount (10 gm) of *Satskar churna* was filled in the funnel. Before filling the churna the orifice of the funnel was blocked by thumb and after filling the funnel thumb was removed immediately. The space between the bottom of the funnel and the top of churna pile was maintained. After emptying the powder from the funnel the height and diameter of the pile was measured and reading is mentioned in Table no. 3.

Hausner's Ratio

The Hausner Ratio was calculated from equation (1), where BD is the bulk density of the *Satskar churna*, and TD is tapped density of the powder¹⁷. Hausner's ratio also measured and ratio is mentioned in Table no. 3

$$HR = TD/BD \quad (1)$$

Carr's index

The Carr's index is frequently used in pharmaceuticals as an indication of the flow ability of a powder. A Carr's index greater than 25 is considered to be an indication of poor flow ability, and below 15, of good flow ability. Carr's index of *Satskar churna* was also measured and index is mentioned in Table no. 3. The Carr Index was calculated from equation (2), where BD is the powder bulk density

and TD is the powder tapped density²¹. These tests were repeated three times for each sample.

$$CI = \frac{TD - BD}{TD} \times 100 \quad (2)$$

Phytochemical Evaluation

For the phytochemical evaluation aqueous extract of *Shatskar churna* has been employed, screening process of *Shatskar churna* for phytoconstituents was done using specific test given in reference books^{6, 8, 26}.

RESULTS

Organoleptic evaluation

Fine powder of *Shatskar churna* is greenish brown in colour, having pleasant odour and salty in taste and contains a fibrous texture, which resembles the appropriate and good quality of formulated churna.

Microscopic observation

The microscopical studies in different mounts of *Shatskar churna* formulation revealed the presence of different specific cellular structures viz. the presence of paracytic stomata and covering trichomes attributed to *Cassia senna* Linn. Fig. 1 & 2; the presence of endosperm cells filled with starch grain and the presence of essential oil containing cells i.e. vittae are associated with *Foeniculum vulgare* Mill. Fig. 3 & 4; the presence of parenchyma cells with starch grain, epidermis cells, fibres and stone cells conferred the *Terminalia chebula* Retz. Fig. 5-8; reticulate vessels, fibres and cork cell with and without starch grain leads to the specific characters of *Zingiber officinale* Rosc. Fig. 9-13. The line drawing was prepared with the help of Camera Lucida (Glass type) and scale was prepared as shown in Fig. 1.

Quantitative analysis

The ash values are useful to determine the quality and purity of the crude drug. Ash contains inorganic radicals like phosphate, carbonates and silicates of sodium, potassium, magnesium, calcium etc. Extractive values are useful for evaluation of crude drugs. It gives an idea about the nature of the chemical constituents present in the crude drug³¹. Analytical results showed that total ash value of formulation was 28.25%. The amount of Acid insoluble ash in *Shatskar churna* was 3.54%. The water-soluble extractive value indicates the presence of sugar, acids and inorganic compounds. The water soluble extractive value in the *Shatskar churna* was found 51.54% indicated the higher water soluble components in the formulation. The alcohol soluble extractive value indicates the presence of polar constituents like phenols, alkaloids, steroids, glycosides, flavonoids and secondary metabolites present in the plant sample. The alcohol soluble extractive value in the *Shatskar churna* was 29.74%. The results of detail analyses are shown in table 2.

Micrometric evaluation

The results of the micrometric analysis are expressed in table 3. The static angle of repose is the angle formed between the side of a stationary pile of powder and the horizontal²⁷ it is interrelated that the more the angle of repose, the more cohesive the powder. The parameter is used by ISO Standard 3435 as a quantitative measure of cohesiveness of a granular material²⁸ According to Bell²⁹, the static angle of repose is one of the best-known

measures of flowability, but suffers from poor reproducibility. One of the main problems is that the angle formed by cohesive powders is not stable; it builds up to a steep angle then collapses to a much shallower one³⁰.

Phytochemical Evaluation

The formulation was qualitatively analyzed for the presence of different secondary metabolites important for the humans and it was found that the formulation exhibited the presence of several phytochemicals viz. alkaloids, glycosides, flavonoids and essential oil etc. The detail result of the preliminary phytochemical screening is mentioned in Table 4.

DISCUSSION

A therapeutically important Ayurvedic preparation was formulated evaluated and resembles different characteristic features. The organoleptic characters showed the good quality of the formulation with the appropriate appearance and pleasant odour. The histological evaluation clearly displayed the presence of specific cellular characters which can be served as reference identification feature of the formulation. Various physicochemical parameters were evaluated and it was found that higher ash values are present due to the presence of mineral salt in the formulation. As part of standardization procedure and guidelines of WHO, the finished product of *Shatskar churna* were tested for relevant Organoleptic evaluation, Powder drug analysis, Physicochemical Parameters (Loss on drying, total ash, acid insoluble ash, water soluble ash, water soluble extractive value, ethanol soluble extractive value), Micromeritic evaluation (density and flow properties), Phytochemical evaluation.

CONCLUSION

The results of powder drug analysis revealed specific identities for crude raw drug which will be useful in preparation and identification of the formulation. The method of preparation of *Satskar churna* and analytical data mentioned in Table No. 2-4 are important findings for evaluation of quality control parameters for Polyherbomineral Ayurvedic formulation.

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